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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JANUARY 1960

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JANUARY 1960

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

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

January 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 4000 – REACTOR DEVELOPMENT

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. – The HRT has operated continuously for more than three months at power levels up to 5 Mw. Approximately 2000 Mwhr of heat was generated in January to bring the total for run 21 to about 5500 Mwhr. The reactor operated at a sustained power of 5 Mw for over seven days during one period in this month with none of the usual indications of fuel instability (i.e., loss of temperature or power disturbances). The principal purpose of the present operation continues to be the study of fuel stability.

As reported previously, the maximum stable power is related to the system pressure and to the concentration of excess acid in the fuel solution. Additional data were obtained to better correlate these factors. At an acid level (H_2SO_4/SO_4) of about 0.30 to 0.32, while operating at 5 Mw, small power disturbances (< 300 kw) were observed starting at a pressure as low as 1350 psig. At 1450 psig, losses in reactivity began to occur. When the acid level was increased to 0.35, the maximum pressure for operation without power pips or reactivity loss appeared to be increased by approximately 100 psig. An experiment was conducted in which the oxygen used by the reactor was increased more than twofold above the normal. There was no noticeable effect on the pressure threshold for instability at 5 Mw. (AEC Activity 4151.3)

Reactor Analysis. – An IBM-704 code was constructed for the solution of two-group, two-dimensional neutron-diffusion equations in cylindrical geometry. In its present form the code can treat problems involving a maximum of 625 mesh points. Machine running times and the required number of iterations have been equal to or less than those required by similar programs. This code facilitates the writing of programs which utilize two-dimensional calculations as subroutines. (AEC Activity 4151.1)

Homogeneous Reactor Instrumentation. – A program was initiated to develop a reactor-grade flowmeter suitable for use in UO_2SO_4 solution at 100°C and in ThO_2 slurry at 300°C. Magnetic-type flowmeters appear to offer the most promise in these applications, and an effort is being made to obtain

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an aluminum oxide–lined flowmeter. Foxboro Instrument Company is investigating the feasibility of lining their magnetic flowmeters with aluminum oxide sleeves. Sample coupons of Coors aluminum oxide are being tested at ORNL to determine the suitability of aluminum oxide for this service.

A 1-in. hydraulically operated ball valve was designed and is being fabricated in the ORNL shops. This valve, designed for ThO_2 slurry service, has a type 17-4 PH stainless steel ball and a replaceable Zircaloy-2 seat. (AEC Activity 4151.1)

Design. – The design was completed for a new line to be installed in the HRT to simplify the return of condensate from the fuel side to the blanket dump tank.

Drawings were prepared of a shielding assembly to permit certain maintenance operations to be performed at the HRT without flooding the reactor cell with water. (AEC Activity 4151.1)

Dynamic Solution Corrosion Tests. – A dissolved-oxygen analyzer, developed at the Bettis Plant of the Westinghouse Electric Corporation, was constructed, calibrated, tested in 100A-loop D, and installed at the HRT. The analyzer was sensitive to less than 10 parts per billion (ppb) of oxygen dissolved in water circulating at 200°C in 100A-loop D, gave readings in the 0.1- to 30-ppm range in agreement with chemical analysis of the water, and appeared to be reliable, easy to use, and adaptable to automatic recording. A value of 22 ppb of oxygen in the condensate from the HRT steam system was obtained with the reactor at 4 Mw, in agreement with the Winkler method of analysis. (AEC Activity 4151.1)

Dynamic Slurry Materials Tests. – In toroid screening tests to evaluate the relative corrosion-erosion properties of D_2O slurries of experimental flame-calcined ThO_2, thorium-uranium oxides, and thorium-uranium-aluminum oxides, attack rates of type 347 stainless steel, titanium, Inconel, and Zircaloy-2 were comparable with rates normally observed with slurries of ThO_2 prepared by conventional methods. In oxygenated tests, slurries of 6% U/Th oxide (6% uranium with respect to thorium) were generally more aggressive than those of pure ThO_2 or of 6% U/Th–2% Al/Th preparations. In tests with deuterium atmosphere, attack by slurries of thorium-uranium-aluminum oxides was greater by an average factor of 2 than with slurries of thorium-uranium oxides.

In a dynamic loop test at 250°C with an oxygenated slurry of flame-calcined mixed oxide (3.2% U/Th–1.6% Al/Th) at a concentration of 450 g of Th per kg of H_2O, attack rates of stainless steels, titanium and its alloys, Zircaloy-2, and noble metals were 0.1 mil/year (mpy) or less at a velocity of 20 fps. At 40 fps, attack rates of the materials ranged from 0.2 to 1.3 mpy. Rates of carbon steel, 15-7 Mo PH stainless steel, Inconel, INOR-8, and alloy steels containing 2 and 5% chromium ranged from 1 to 6 mpy at 20 fps and from 1 to 11 mpy at 40 fps and were least affected by changes in slurry flow velocity. The aggressiveness of the flame-calcined material compared favorably with that of regular-production thoria.

In two loop tests made at comparable test conditions (250°C, ~ 700 g of Th per kg of D_2O, with oxygenated slurries of 2.8% U/Th oxide and of pure ThO_2 – average particle diameters of 2.5 and 2.4 μ respectively), attack by the slurry of mixed oxide was approximately a factor of 4 greater than that

of pure thoria. The 1075°C-calcined thorium-uranium oxide was degraded (average diameter reduced from 2.5 μ to 1.8 μ) during 340 hr of circulation. The pure thoria did not degrade. With the pure ThO_2 slurry at a flow velocity of 20 fps, attack of stainless steels 329, 347, 17-7 PH, 15-7 Mo PH, and USS Corex, and of Croloy 5, Zircaloy-2, titanium, and the alloy Ti–13% V–11% Cr–3% Al ranged from < 0.1 to 1 mpy. At 40 fps, rates of the materials were 0.2 to 1.4 mpy. (AEC Activity 4151.1)

In-Pile Slurry Corrosion Loop. – A 5-gpm pump loop designed to circulate thorium oxide slurries while exposed to reactor radiation is under test. This loop is suitable for operation in beam hole HB-2 of the LITR and is similar to the in-pile loops used in the radiation-corrosion studies of uranyl sulfate solutions during the past five years.

Satisfactory operation of this loop with slurries is dependent on maintaining the pressurizer free of thoria. This has been accomplished by means of a sintered-metal filter installed in the loop main stream, with the thoria-free filtrate used as the pressurizer feed stream and as a pump-bearing purge.

Some 2500 hr of out-of-pile operation at 280°C have been accumulated in a prototype loop circulating a thorium oxide slurry containing 1/2 wt % uranium dispersed in D_2O. Circulating-slurry concentration ranged from 200 to 800 g of Th per kg of D_2O. Some loss of thoria was observed after ~ 700 hr of operation by deposition in the loop core section, where the slurry flow velocity was relatively low (2 to 4 fps). This loss may be associated with the particle-size degradation (1.9 to 0.7 μ average particle diameter) observed during circulation. Improvement in particle integrity has been indicated in toroid tests when the firing temperature of the thoria was increased from 1225 to 1500°C, and a quantity of 1500°C-calcined material has been prepared for testing in the loop. Also to be tested is a redesigned core section fabricated of bent piping to improve the core flow geometry and to increase the flow velocity.

The kinetics of recombination of deuterium and oxygen were studied in the loop while it was circulating slurry containing a palladium catalyst at an operating temperature of 280°C. At a palladium concentration of 180 mg per liter of circulating slurry, catalytic activities ranged from 0.45 (initially) to 0.25 mole of deuterium per liter per hour at a reference partial pressure of 100 psia deuterium during an operating period of 200 hr. Such recombination rates are adequate for the first in-pile slurry loop experiment. (AEC Activity 4151.1)

In-Pile Autoclave Slurry Corrosion Studies. – Irradiation of a larger volume Zircaloy-2 autoclave, experiment L5Z-152S, in the HB-5 facility of the LITR was completed. The autoclave, operated at 280°C under an excess-oxygen overpressure, contained slurry at a net concentration of 1080 g of Th per kg of D_2O, with 8 wt % enriched uranium based on thorium and 0.0006 m palladium suspension added as a recombination catalyst. The autoclave contained corrosion coupon specimens of Zircaloy-2, Ti-75A, and type 347 stainless steel. The fission-power density generated in the autoclave in the fully inserted position at 280°C was 20 w/ml at an assumed neutron flux of 1×10^{13} neutrons·cm^{-2}·sec^{-1}.

The experiment was operated in-pile at 280°C for 64 hr following out-of-pile pretreatment and experimentation for a period of 676 hr at temperature. Insertion of the experiment resulted in a pressure rise of 125 psi

due to radiolytic gas, indicating a catalyst performance index of 24 w/ml at 100 psi deuterium. The increase in temperature differentials between internal thermocouples and thermocouples on the autoclave exterior was more than that which could be attributed to usual gamma heating effects. The experiment was terminated because the 20-mil-ID capillary line leading to the pressure cell became plugged. Partial relief of the plug permitted pressure measurements, which indicated no corrosion during the irradiation period. (AEC Activity 4151.1)

Supporting Radiation Corrosion Studies. – Batch-type experiments to measure the adsorption, by hydrous zirconium oxide, of uranium from UO_2SO_4 solutions containing 0.02 m H_2SO_4 continued in both Pyrex and titanium autoclaves at 250°C. This method has proved very satisfactory and yields quite reproducible results. The adsorption capacity of the 100°C-air-fired oxide was found to range from 35 to 115 mg/g in 4-hr exposures over the concentration range of 1.5 to 28.6 g of uranium per liter. The ratio of adsorbed sulfate to adsorbed uranium was found to be practically constant at 1.25 on a molar basis. These data are best represented by a general expression which is in the form of the Freundlich isotherm equation:

$$\frac{\text{mg of U}}{\text{g of oxide}} = kC^n \quad ,$$

where C is the uranium concentration in g/liter at 25°C, and k and n are constants with values of 30.6 and 0.397 respectively. This equation should provide a base line for use in studying the effects of other variables of the uranium adsorption. (AEC Activity 4151.1)

Molten-Salt Reactor Program

Chemistry. – The solidification behavior of representative breeder fuels is being studied in greater detail. For $LiF-BeF_2-UF_4-ThF_4$ (71-16-1-12 mole %) freezing begins at 513 ± 2°C, rather than at the previously estimated temperature of 525°C, and proceeds by the precipitation of Li_3ThF_7 containing both UF_4 and BeF_2 in solid solution. The composition of this fuel is equivalent to that of a mixture of Li_3ThF_7 (solid solution) and Li_2BeF_4, the only phases present after freezing is complete at 439°C. Due to the relatively wide range of compositions manifested by solid solution in Li_3ThF_7, the same is true, at slightly different temperatures, for most of the blanket compositions and high-thorium fuels of current interest.

Preliminary indications are favorable for the substitution of ZrF_4 for ThF_4 in core fuels to achieve a smaller uranium inventory and yet retain the advantages of improved chemical behavior associated with less basic melts.

Experiments related to the use of solid BeO to remove UF_4 from breeder melts have been extended to lower ranges of uranium concentrations. In two trials the addition of 30 g of BeO per kilogram of blanket mixture ($LiF-BeF_2-ThF_4$, 67-18-15 mole %), containing 1800 ppm of uranium as UF_4, reduced the uranium concentration in the melt to less than 1 ppm. Experiments have also been initiated to determine the efficacy of this procedure for removing protactinium.

Weight gains representative of the permeation of extruded graphite tubing (RLM-24-CEY) by breeder fuels at 60 psi are now recognized to be even lower than the previously reported 0.2 wt %, since it has been confirmed by analysis that most of the penetration occurred at the exposed ends of 3-in. lengths of tubing samples. (AEC Activity 4152.1)

Metallurgy. — A screening study was completed to determine the flowability and wetting characteristics of 36 brazing alloys on graphite. Only an alloy consisting of 48% Ti–48% Zr–4% Be was rated excellent. Three other alloys were considered moderately good.

A study is being conducted of the Au-Ni-Ta system for use as braze material to attach graphite to metal for molten-salt application. Many of the alloys with melting points below 1300°C were found to have good wetting and flow characteristics on graphite-to-molybdenum joints when brazed in a vacuum muffle furnace. The high-tantalum alloys were found to exhibit good bonding and minimum interfacial cracking. AGOT graphite was brazed more readily than fine-grain low-permeability graphite.

A five-tube, tube-to-tube-sheet assembly was successfully fabricated by using brazing techniques; the assembly contains graphite tubes, molybdenum transition sections, and an INOR-8 tube sheet. This arrangement was established to minimize stresses caused by differential expansion of the materials. The assembly appeared to be structurally sound. (AEC Activity 4152.1)

Graphite Systems. — A successful graphite-to-metal static seal was established in a loop circulating molten salt at 1100°F. The seal, approximately 5 in. in diameter, was formed in an annulus of frozen salt between the graphite and metal surfaces. Such a seal is applicable to sealing the fuel from the blanket fluid in a molten-salt thermal breeder reactor. (AEC Activity 4152.1)

In-Pile Tests. — Assembly of the first MSR in-pile graphite-fuel capsule was completed. The experiment was inserted in the MTR during the shutdown on January 25. Parts for the second experiment were fabricated, and assembly was begun. In this experiment, scheduled for insertion on March 7, four specimens of new low-permeability grades of graphite will be irradiated. A capsule bellows of INOR-8, instead of Inconel, will be used. (AEC Activity 4152.1)

Pump Development. — Test operation of the molten-salt pump containing a salt-lubricated journal bearing continued. The operating conditions are 1200°F, 1400 rpm, and 100 gpm with salt 130. To date, the total operating time is 840 hr. One stop-start operation was performed.

One test was performed on the molten-salt journal-bearing tester. It was a repeat of a previous test at 1500°F with an INOR-8 carburized journal and a three-axial-groove bearing in which start-stop ability was to be investigated. Seizure was impending soon after startup, and consequently start-stop operations were not initiated. Examination revealed evidence of considerable rubbing and galling in the bearing.

The MF-F pump endurance test has operated continuously for more than 21,790 hr circulating molten salt 30 at 1200°F. The PKP pump rotary assembly was removed from the test stand and was disassembled after circulating NaK at 1200°F for 18,800 hr. The rotor and stator faces of the lower seal had

worn considerably and will require replacement prior to further testing. The impeller was found to be in good condition, with no more than a clean etched appearance in the flow passages, which is typical of long service. (AEC Activity 4152.1)

Remote Maintenance Demonstration. — Practice operations were performed on the remote removal and installation of the reactor. Several minor mechanical modifications to the facility have been completed. Remote maintenance operations perfected to date are the removal and replacement of the pump, of the heat exchangers, of the reactor core vessel, and of the fill and drain tank.

The loop is being assembled as preparations are being made to fill it with molten salt for the first time. (AEC Activity 4152.1)

HIGH FLUX ISOTOPE REACTOR

Reactor Physics Calculations. — Calculations have been made to determine the approximate reactivity worth of a proposed set of control plates in the HFIR and to determine the effect the position of the plates has on the power distribution during a typical fuel cycle. The control region, adjacent to the outer surface of the fuel region, consisted of two 1/4-in.-thick concentric cylinders that would move vertically but in opposite directions, maintaining symmetry about the vertical axis and horizontal midplane. Each cylinder consisted of a black, a gray, and an essentially nonabsorbing vertical section, in that order. Each section was one core length long. The upper cylinder was divided into four 90° circular arcs so as to increase the probability of poison insertion in the event of a scram. Because of design considerations it was assumed that the lower cylinder was divided in a like manner but rotated 45° with respect to the upper cylinder. It is not anticipated that the lower plates would be used during a scram.

A calculation showed that enough reactivity control was available in the shim plates to keep a ten-day core subcritical even if the burnable poison were inadvertently omitted, a most unlikely accident. The same shim plates would control the same core in the unlikely event that an accident occurred whereby the plates normally inserted from the bottom were inadvertently removed concurrently with a failure of one of the top plates whereby it did not release and drop.

Results from one-dimensional fuel-cycle-time calculations were superimposed on results from two-dimensional calculations, in which the actual geometry of the control plates during normal operation was considered, in order to determine the ratio of maximum to average power density during the fuel cycle. After one day of full-power operation, a neutron window appears between the gray regions of the control plates. The results of the calculations when carried to the end of the cycle indicate that peaking of the power density in the vicinity of the window does not exceed the maximum power density that occurs at the beginning of the cycle or at any time during the cycle. At the beginning of the cycle the ratio of maximum to average power density is 1.45.

A study has recently been made to determine the effects of the (n,α) reaction in the beryllium. Changes detrimental to reactor operation are produced in beryllium when it is subjected to a high fast-neutron flux. These

fast-neutron reactions [(n,α) and (n,2n)] lead to the formation of gases (He^4, He^3, and H^3 primarily) which can cause dimensional changes. Another possible detrimental effect is the loss of reactivity due to the formation of Li^6 and He^3, which have high thermal absorption cross sections. Tending to offset this poisoning effect is the (n,2n) reaction in beryllium. The net effect of the fast-neutron reactions is a function of the neutron energy spectrum in the beryllium and must be evaluated for each particular reactor. Estimates made for the beryllium reflector of the HFIR indicate that the net reactivity decrease due to Li^6 and He^3 formation is small and will not exceed 2% for the beryllium lifetime.

Gas formation in the beryllium appears to limit the lifetime of the beryllium reflector. Comparison with experimental data from the MTR indicates that the innermost ring of the reflector may have to be replaced in as little as six months. Even if the estimate is too conservative, prudence dictates a design that permits the beryllium reflector to be replaced, especially the inner portion.

Studies were made on the effect of nonthermal neutron capture on the production of Cf^{252}. The optimum ratio of nonthermal to thermal flux was found to be very dependent on the Cm^{248} thermal cross section, a value which is very uncertain. Considering the limits of uncertainty of this cross section, it was concluded that the present concept of the HFIR is close to or can be easily made optimum with respect to the nonthermal flux. It was also estimated that the Cf^{252} production rate will be enhanced by 50 to 100%, depending on the Cm^{248} cross section, as the result of nonthermal capture. (AEC Activity 4410)

Critical Experiments. — The simulated reactor vessel has been installed in the critical experiment facility and has been made critical. The observed critical mass checked the calculation within 8%. Detailed neutron flux traverses are being made with miniature fission chambers 3/16 in. in diameter and 1 in. long. (AEC Activity 4410)

Heat Transfer. — A considerable number of local and average heat-transfer coefficients and average friction factors, derived from data obtained with aluminum test sections under conditions which bracket the present HFIR design, correlate better with the Sieder-Tate equation than with the GE-ETR data, which are 1/3 lower. A smaller number of burnout tests indicate that burnout is not inherently penalized by the narrowness of the proposed 50-mil coolant gap, as was thought possible earlier. One more test with the present aluminum plate design is planned.

Experimental and analytical studies of natural-circulation burnout are nearing completion. Calculations of the burnout safety factor as a function of time after pump failure will be made when prediction of the flow coastdown curve is completed.

Test sections for investigation of the effect of plate spacers on temperature distribution and burnout heat flux are being designed. (AEC Activity 4410)

Corrosion. — The first test designed to determine the effect of heat flux on the corrosion of type 1100 aluminum has been completed. In this test high-purity water flowed through a channel (0.058 ± 0.001 by 0.500 in.) in

the aluminum test specimen at a rate of 33 fps. An alternating current of approximately 10,000 amp was passed through the specimen, which had a non-uniform metal cross section, and produced calculated heat fluxes of 1.6×10^6 Btu·hr^{-1}·ft^{-2} across the 55% of the surface that was under the thicker section of the aluminum and 4.9×10^5 Btu·hr^{-1}·ft^{-2} across the remainder of the surface. The 3 gpm of water that flowed through the test piece entered at 66°C and was heated to 89°C upon passing through the heated section. The system was pressurized to 300 psia.

Several thermocouples were located on the outside of the test section. During the ten-day run the temperature on the outside of the aluminum increased, presumably because of the formation of a corrosion product layer on the water-cooled surface. The measured temperature on the outer surface of the aluminum near the outlet end increased from 191°C at the beginning of the test to 294°C at the end of the ten-day test. The corresponding increase close to the entrance of the test piece was from 169°C to 197°C.

Visual examination of the water-cooled surface showed that some film stripping occurred near the outlet of the piece on the surface with the higher heat flux; some random pitting was observed on the remainder of the surface. A determination of the extent of corrosion has not been completed, but corrosion was not catastrophic, although the heat flux was about 16% greater than at the hottest spot in the reactor and the velocity was 82% of the design value, which, however, is about equal to that in the worst channel assumed in the hot-spot-hot-channel analysis. The system pressure was 300 psi instead of 600 to 1000 psi as presently contemplated in the design. The next test will attempt to duplicate all conditions, except that it will operate at 900 psi for the purpose of observing the effect of raising the boiling point of the water. (AEC Activity 4410)

Metallurgy. — Forming studies have continued in an attempt to develop optimum conditions for fabricating involute fuel plates with reproducible dimensions for the mark II element. Varying the pressures between 22 and 102 tons resulted in no significant change in the ability to duplicate the dimensions of solid aluminum (6061-T0) plates when formed with rubber-metal dies. Repressing with metal dies at 75 tons and intermediate anneals at 500°C did not improve the reproducibility and in the latter case resulted in doubling the dimensional variation. Measurements at similar locations of 14 solid aluminum plates formed with metal-rubber dies under similar conditions indicated a maximum spread of 0.008 in. and an average spread of 0.0025 in.

Measuring gages are being developed for determining both the spacing between plates and the plate contour in the mark II element. The channel-spacing gage consists of a modified Laninagage (eddy-current cladding thickness gage) and a special electromagnetic transducer that fits into the channel. The channel spacing influences the electrical properties of a miniature coil in the transducer, and by appropriate calibration the spacing dimension is indicated by a meter on the instrument. The plate-contour gage contains four dial indicators which span the width of the plate and may be moved along the plate length to make measurements. A check of 180 duplicate measurements on four plates resulted in a maximum deviation of 0.003 in. (AEC Activity 4410)

Design. — The first drafts of performance specifications for the HFIR building and reactor cooling system were written. These are to be used by the architect-engineer in preparing drawings and specifications for the HFIR.

Calculations of the activity in the HFIR cooling system are being carried out. The N^{16} and N^{17} activities in the primary coolant discharging from the pressure vessel were calculated to be 2.5×10^6 and 5.5×10^2 disintegrations$\cdot cm^{-3} \cdot sec^{-1}$, respectively.

The proposed Pu^{242} target array consists of nineteen 3/8-in. rods suspended vertically in a cylinder within the HFIR island. The target material is cooled by water flowing through the island.

Heat transfer studies associated with the mechanical design assume that 200 g of Pu^{242} as the oxide is uniformly distributed throughout the rods, that the ratio of the maximum to average heat generation rates is 1.268, and that the total heat generation rate is about 500 kw. Preliminary calculations indicate that a velocity of 30 fps past the target rods is needed. This requires a mass flow rate of about 1000 gpm through the island.

The single-plate test rig is operable. An involute plate collapsed when 26 psi was applied to the convex side. A plate deflection of 1 mil/psi was observed. (AEC Activity 4410)

Core Test Loop. — All of the core test loop has been assembled except for the test specimen and its support. (AEC Activity 4410)

MARITIME SHIP REACTOR PROGRAM

Maritime Reactors. — The nine participants from the NS "Savannah" Training School have satisfactorily completed three months of on-the-job training with the Laboratory's Reactor Operations Department. The program was terminated on January 15, 1960. (AEC Activity 4510)

GENERAL REACTOR RESEARCH

Reactor Evaluation Studies. — The effects on the net fuel cost resulting from changing the over-all handling and fabrication charges and from varying the fuel inventory charge in a Dresden-type reactor were studied. The value of plutonium fuel was assumed to be $12 per gram. Decreasing the over-all uranium charge (this includes conversion, fabrication, shipping, and processing charges) from $215 to $60 per kg of U decreased the optimum initial uranium enrichment from about 3% to 2% and decreased the fuel cost by about 1.8 mills/kwhr (electrical). Increasing the fuel inventory charge from 4% to 8% increased the fuel cost by about 0.6 mill/kwhr (electrical). (AEC Activity 4160)

Reactor Safety Studies. — The BSR-II test program at the SPERT-I facility of the National Reactor Testing Station has been carried to the point of incipient core rippling at self-limiting shutdown. A re-evaluation of further test programs was undertaken at NRTS, and testing will resume about February 10, 1960, following installation of additional channels of reactor instrumentation. No further self-limiting excursions are planned. (AEC Activity 4171.1)

Fuel Element Catastrophe Studies. — A short-term program was undertaken to furnish preliminary data on the release of radioactive fission products

in a meltdown in the GE-ANP reactor. The method of optically focusing the radiant heat from a carbon arc was employed. By this method no container crucible is used, since the melted portion is contained by the remainder of the sample. An Arthur D. Little 300-amp strong arc image furnace was procured for this purpose. Results on the first of three types of samples are available. (AEC Activity 4171.1)

Radiation Detector Development. — The program directed toward development of a suitable detector for use in the model IV gamma-ray spectrometer at the Bulk Shielding Facility has been delayed because of damage to the large (9-3/8-in.-dia) NaI(Tl) crystal suffered during shipment from the supplier. Pending replacement of the large crystal, an investigation of the effects of variations in collimator design is being carried out with smaller crystals. (AEC Activity 4410)

Long-Range Planning Studies. — An IBM-704 program has been written which computes multigroup-averaged cross sections for an element having a single Breit-Wigner-type resonance; required input data are the cross section at 2200 m/sec, the resonance integral, and the energy location of the resonance. (AEC Activity 4410)

Nondestructive Test Development. — The program for the development of techniques for assaying fuel elements by gamma-ray spectroscopy has been reactivated. After the completion of current determinations of calibration and operation procedures for the application to fully enriched uranium-aluminum alloy core blanks, the technique will be extended to other fuel systems and to the scanning of fabricated fuel plates.

A program has been initiated to study methods of producing artificial defects in tubing. The use of these artificial defects will facilitate the calibration of electromagnetic inspection equipment and the specification of inspection procedures. A variety of machining and etching techniques have been investigated with regard to reproducibility, instrument responses, and relationship to natural defects. The most promising artificial defects investigated are narrow longitudinal notches made by an arc discharge machining technique.

Recent efforts have produced a radiographic inspection method which can be successfully applied to thin sections of beryllium and other low-absorption materials. This method requires the use of x-ray energies down to 8 kev, a helium atmosphere between the x-ray source and film to reduce scatter, and the use of a dark facility to allow radiographic exposure of bare film. With these techniques, beryllium samples in sections down to 0.040 in. in thickness have been successfully radiographed. (AEC Activity 4420)

Mechanical Properties Research. — Equipment was constructed for conducting complex mechanical tests simulating various service conditions within a reactor. Several machines were designed for relaxation tests. Data have been obtained from these machines for Inconel, INOR-8, type 304 stainless steel, Hastelloy B, Zircaloy-2, and graphite. For some of these materials, the relaxation data were used to predict the constants of the creep equation.

Several machines of the type used in relaxation testing were modified for conducting fatigue tests of various types, including (1) creep tests in which the stress is cycled between tension and compression at any selected

frequency, (2) creep tests in which the stress is cycled between tension and compression but the creep is limited to a selected strain amplitude, and (3) relaxation tests in which the specimen is instantaneously cycled between fixed strains in tension and compression and held at these strains during each half cycle. The machines show satisfactory performance at room temperature.

Another machine was modified for conducting torsion fatigue tests on tubular specimens. Special attachments were included to permit the superimposition of pressure and axial stress on the tube. The performance of the machine at room temperature was satisfactory. (AEC Activity 4420)

Fuel Cycle Development. — Fabrication of dense powder beds from loose particles by vibratory compaction is facilitated by using a material of high density (within the particle boundaries) and with a size distribution favorable for close packing of the smaller particles into the voids between larger particles in contact.

The densities of 57 different preparations of thoria and uranium-bearing thoria were determined pycnometrically by means of isopropyl alcohol and toluene. The densities of 16 of these preparations were determined more precisely by mercury porosimetry. Densities greater than 9.9 g/cc, which is considered suitable for vibratory compaction, were found for the following preparations: ground fused thoria (Norton Company), 9.98 g/cc; DT-7-1600°C thoria (Pilot Plant), 9.94 g/cc; 3- to 10-μ thoria spheres (Houdry Process Corporation), 9.90 g/cc. (AEC Activity 4420)

Power Reactor Fuel Processing: Darex Process. — In engineering studies on chemical decladding of Consolidated Edison (stainless-steel-clad ThO_2-UO_2) fuel in a new 4-in.-ID dissolver, stainless steel dissolved in 5 M HNO_3—2 M HCl at a rate of 80 g/min. In laboratory tests on unirradiated ThO_2—5% UO_2 pellets, initial rates of dissolution in 200% excess boiling 13 M HNO_3—0.04 M NaF—0.1 M $Al(NO_3)_3$ decreased from 16 to 2 $mg \cdot min^{-1} \cdot cm^{-2}$ as the pellet density increased from 60 to 94% of theoretical. The rates appeared to be independent of the O/U ratio in the pellet. In continuing engineering-scale studies on dissolution of high-density ThO_2-UO_2 pellets, dissolution rates in chloride-freed Darex decladding solution (9.84 M HNO_3, 0.04 M fluoride, 60 ppm chloride, 53 g stainless steel per liter) were somewhat lower than those with Thorex dissolvent (13.3 M HNO_3, 0.07 M Al^{+++}, 0.04 M fluoride) under similar conditions.

Boildown curves for thorium nitrate—nitric acid solutions show that regardless of original composition the acid is not completely removed until the thorium is concentrated to about 1300 g/liter.

In flowsheet studies for NS "Savannah" fuel processing, stainless-steel-clad UO_2 pellets, irradiated to 7490 and 12,400 Mwd/ton, were dissolved in hot-cell tests in 5 M HNO_3—2 M HCl; the chloride was removed by the Darex reference flowsheet method to final concentrations of 490 and 160 ppm, respectively.

Corrosion rates of welded A55 titanium were < 0.1 mil/month in the vapor phase after nine cycles of Darex decladding and seven cycles of UO_2-ThO_2 dissolution. In 1008-hr exposures of titanium to Thorex dissolver solutions contaminated with chloride and Fe^{+++}, maximum rates were 7, 0.23, and 0.16 mils/month in beginning, middle, and final solutions, respectively. No aggressive localized attack was observed. In Thorex feed adjustment tests,

titanium corrosion was maximum at the bottom of the total reflux condenser, amounting to 1.4 mils/month in 336 hr. In a complete Thorex feed adjustment boildown, titanium specimens did not lose weight; the maximum fluoride concentration in the distillate was 0.0012 M. In Darex-Thorex tests, maximum over-all rates were 0.02 mil/month after 32 cycles. Type 304L stainless steel was corroded at maximum rates of 0.061 and 0.34 mil/month at 80°C in Darex-Thorex and Darex-Purex waste solutions, respectively. (AEC Activity 4441)

Power Reactor Fuel Processing: Sulfex Process. — The results of two runs in which prototype Nuclear Merchant Ship Reactor fuel elements (stainless-steel-clad UO_2 pellets) were declad with 4 M H_2SO_4 indicated that decladding rates for tubular cladding are approximately half the rate for plate of the same type of stainless steel under comparable conditions. In cyclic Sulfex depassivation tests, stainless steel corrosion rates were 10.7 and 4.4 mils/month for three cycles when 10 M and 8 M H_2SO_4, respectively, were substituted for 12 M H_2SO_4 in the initial 5 min of the cycle. The corresponding rate for 12 M H_2SO_4 is at least 80 mils/month (27 mils per 100 cycles).

Waste solids from Sulfex decladding liquor were shown, by Cintel flying spot microscopy, to have a mean particle diameter of 3 μ. This value is in fair agreement with results obtained earlier by centrifugal classification.

In tests in a 12-in.-dia bowl centrifuge, a wet cake of submicron UO_2 powder, approximately 0.5 in. thick, was successfully dislodged by 3 liters of water discharged from a spray nozzle at 100 psi; cyclic acceleration to 2180 rpm and braking to rest did not dislodge the cake. (AEC Activity 4441)

Power Reactor Fuel Processing: Zircex Process. — In a Zircex run in which all the hydrochlorination residue was converted to volatile chlorides by reaction with carbon tetrachloride, less than 0.01% of the uranium from a 7% U-Zr alloy sample was lost. The uranium desublimer was packed with copper turnings. (AEC Activity 4441)

Power Reactor Fuel Processing: Zirflex Process. — In hot-cell Zirflex decladding experiments with unirradiated samples and with PWR fuel specimens irradiated to 1950 Mwd per ton of uranium, soluble uranium and plutonium losses were 0.01 to 0.11 and 0.05 to 0.08%, respectively. The gross gamma and gross beta activities in the cladding solution were 2.2 to 8.3 and 0.1 to 0.6% of those in the fuel, respectively. The soluble uranium losses compare favorably with experimental values on unirradiated fuel samples previously reported by other groups. The main difficulty in the flowsheet appears to be precipitation in the diluted cladding waste solutions.

The solubility of zirconium fluoride in nitric acid-ammonium fluoride solutions was determined at 25°C. In solutions where the F/Zr mole ratio was greater than about 8, the solubility decreased with increasing nitric acid concentration. Since the solubility was greater in the presence of nitric acid when the F/Zr ratio was between 6 and 8, no deleterious effect is expected if a Zirflex decladding solution should be contaminated with a trace of nitric acid.

In the Modified Zirflex flowsheet developed for 7% uranium-zirconium alloy, the NH_3 off-gas evolved during dissolution contained 1% H_2, 0.3% O_2, and 0.2% N_2.

In cyclic Zirflex corrosion tests, Ni-o-nel was corroded at maximum rates of 4.6 to 5.1 mils/month over 23 cycles, whereas Hastelloy F gave

maximum rates of 3.5 to 5.0 over 5 cycles. In boiling 6 M NH_4F–0.3 M NH_4NO_3, Hastelloy F, Ni-o-nel, and type 309 SCb stainless steel were corroded at rates of 5.0 to 7.6, 4.6 to 8.7, and 4.8 to 13.4, respectively, in the absence of additives. Rates for all three alloys were decreased by factors varying from 3 to 10 (for a 3-hr exposure) by the addition of 0.03 M H_2O_2 and/or by contamination with small amounts of copper from simultaneous corrosion of Monel or copper wire. The lowest solution-phase rates observed for a 3-hr exposure were 2.5 for Hastelloy F and 4.0 for Ni-o-nel. (AEC Activity 4441)

Power Reactor Fuel Processing: Head-End Processes for Uranium-Graphite Fuels. — Graphitized fuel specimens containing 0.7 and 5% uranium were disintegrated (to -20 mesh) and then leached by boiling 90% HNO_3 for 4 hr. After washing and a second HNO_3 leach, the uranium recovery was 99.85% from the 5% fuel and 97% from the 0.7% fuel. With mechanical grinding to -16 +30 mesh and leaching twice with boiling 15.8 M HNO_3, uranium recoveries were 99.85 and 83% of the uranium, respectively. Leaching of the 0.7% fuel with 90% HNO_3 for 2 hr at 25°C recovered 50% of the uranium without any washing of the residue.

Pebble Bed Reactor prototype 1.5-in.-dia balls containing 10% high-fired UO_2 mixed with graphite were attacked by the 90% acid at a much lower rate. A 2-hr contact at 25°C, followed by 4 hr in boiling 90% HNO_3, washing, and a second 4-hr reflux in 90% HNO_3 disintegrated 80% of a graphitized specimen for a uranium recovery of 87%. Only 41% of an ungraphitized sample was disintegrated; the amount of uranium recovered was 62%. Increasing the contact time to 32 hr resulted in disintegration of 93% of the ungraphitized material; the metal recovery was 95%. (AEC Activity 4441)

Power Reactor Fuel Processing: Head-End Processes for Uranium-Molybdenum Fuels. — It is planned that the stainless-steel-clad NaK-bonded uranium-molybdenum PRDC blanket and CPPD fuel will be mechanically declad and the NaK destroyed with steam prior to dissolution of the uranium-molybdenum alloy. In uranium-molybdenum dissolution and feed preparation studies, the solubility of $MoO_3 \cdot 2H_2O$ in 2 to 5 M HNO_3 at 26°C decreased with increasing uranyl nitrate concentration. For example, in solutions containing 3 M HNO_3, the molybdenum concentration decreased from 0.12 to 0.08 M as the uranium concentration increased from 0 to 0.95 M. These data indicate that molybdenum will not precipitate during solvent extraction of nitric acid solutions of uranium and molybdenum from which $MoO_3 \cdot 2H_2O$ has been removed by prior centrifugation. (AEC Activity 4441)

Power Reactor Fuel Processing: Mechanical Processing. — A 36-tube ferrule-connected stainless-steel-clad mark I prototype fuel assembly filled with porcelain was sheared at 1-in. intervals at an average blade velocity of 2 in./sec. Two similarly shaped "concave upward" plane-of-contact blades operating with a V-bottom anvil in a vertical 150-ton-thrust hydraulic press were used. About 1.5% of the total metal of the prototype appeared as metallic fines passing a diamond-shaped screen with a mesh size of 0.86 by 0.25 in., which represented essentially all the highly worked pieces. A total of 2.6% of the 86.5 g of Nicrobraz per ferrule section in the mark I prototype was in these fines. About 0.4% of the Nicrobraz was less than 3 mils in diameter; this size range of Nicrobraz particles (less than the blade separation) would be expected to contribute significantly to blade wear by galling between the blades.

Eighty-eight tons of thrust was required to shear a UO_2-filled mark I assembly with straight plane-of-contact blades in the Manco 125-ton shear, approximately the same force as for a porcelain-filled mark I prototype. (AEC Activity 4441)

Power Reactor Fuel Processing: Solvent Extraction Studies. — Fuel solutions prepared from two irradiated NS "Savannah" stainless-steel-clad UO_2 fuel pins by the Darex process were blended and processed through two cycles of solvent extraction according to a modified first-cycle and a standard second-cycle Purex flowsheet. Uranium losses were within specifications of 0.02% per cycle; plutonium losses were also 0.02% in the first cycle but increased to 0.9% in the second cycle. The over-all two-cycle gross beta and gross gamma decontamination factors were 7.38×10^5 and 1.23×10^5, respectively.

In solvent extraction tests on recovering uranium from enriched uranium fuels, uranyl nitrate was extracted from originally acid-deficient solution by adding acid to a late extraction stage. Decontamination factors from ruthenium and zirconium-niobium were 1.6×10^4 and 2.2×10^4, respectively. The plutonium goes into the aqueous waste in this system.

The acid in the Acid Thorex uranium-thorium product may be decreased without decreasing the decontamination factors by using a divided nitric acid-water scrub. (AEC Activity 4441)

Fluoride Volatility Processing. — In further studies of the effect of corrosion products on dissolution of zirconium in fused salt, a test element was hydrofluorinated to about 50% weight loss at an average rate of 1.5 $mg \cdot cm^{-2} \cdot min^{-1}$, and the fused salt was discharged and cooled. Examination of the fuel element showed the expected heavy layer of metallic contaminants, and the salt phase contained visible amounts of dark-colored contaminants. The same element and the salt were recharged to the dissolver and the reaction was carried to completion. The second half of the reaction proceeded at a normal rate, 0.7 $mg \cdot cm^{-2} \cdot min^{-1}$. A sharp end point was indicated by a greatly decreased hydrogen evolution. Both salt and vessel were relatively clean and free of contaminants, with no test element remaining. Previous reports of heavy corrosion-product accumulations have been based on runs that were stopped short of completion to allow examination of conditions during dissolution. It appears that at the completion of zirconium dissolution the corrosion products are also dissolved rapidly and cleanly; analysis of the salt to verify this conclusion is not complete. (AEC Activity 4442)

PROGRAM 5000 – PHYSICAL RESEARCH

Reactor Operations. – The Graphite Reactor has been operating routinely since December 22, and all the laboratories inside the building have been decontaminated. Some decontamination is still being done on equipment which was removed from the building. Air conditioners, especially, became contaminated during the incident of November 20 and require extensive decontamination.

The ORR operated very well during the cycle which ended January 24. There were no spurious shutdowns.

Laboratory Facilities. – Design is proceeding on a permanent monitoring system for both the process waste and the gaseous waste systems. Since some time will elapse before the permanent system can be installed, temporary monitors are being installed wherever possible. Design is also proceeding on filters for the cell-ventilation system, on a soil column to replace the waste pits, and on a pipeline between the tank farm and the waste pit area which would permit the process waste to be pumped to a special pit in the event of an incident in the process waste system.

Waste pit No. 4 began leaking fission product ruthenium, and it was necessary to intercept the drainage area below this pit and pump the drainage containing the leakage back to the pit. By this means the radioactivity at White Oak Dam has been reduced nearly to normal levels. The total radioactive effluent to the Clinch River in the last quarter of 1959 was quite low. The levels of radionuclides in the river were about 11% of the maximum permissible concentrations, which was lower than in any other quarter of 1959.

PHYSICS

High-Voltage Experimental Program. – The pulsed 3-Mv Van de Graaff is undergoing the first stages of modification to permit installation of the duo-plasmatron ion source. This new source is expected to raise the available peak current by a factor between 5 and 10 over that presently available. Theoretical studies have indicated that beams of this order of magnitude cannot conveniently be chopped shorter than about 4 mμsec at the terminal. To make available shorter pulses, a system of postacceleration auxiliary pulsing is being engineered simultaneously with the terminal modifications for the duo-plasmatron. Since ion burst production requires introducing into the beam an energy spread roughly inversely proportional to the burst time, the immediate application of the 1-mμsec bursts to the kev neutron studies awaits more precise evaluation of the energy spread introduced. (AEC Activity 5220)

Spectroscopy Research Laboratory. – The infrared and Raman spectra of the selenocyanate ion, $NCSe^-$, have been observed and studied in solution and in solid KNCSe. Absorptions arising from the fundamental vibrational modes in the solid occur at the following frequencies: $\nu_{(CN)} = 2070\ cm^{-1}$, $\nu_{(CSe)} = 558\ cm^{-1}$, and $\nu_{(NCSe\ bend)} = 424,416\ cm^{-1}$. The splitting of the bending mode indicates the crystal structure to be similar to that reported for KNCS.

Absorptions arising from $NC^{13}Se$ have been identified and potential functions evaluated. (AEC Activity 5230)

CHEMISTRY

Raw Materials Research and Development. – The feasibility of stripping uranium from both Dapex and Amex solvents with ammonium carbonate solutions, while recycling the strip solution to precipitate ammonium uranyl tricarbonate (AUT), was demonstrated in continuous processing equipment. Calcination of the AUT at 500°C yielded sodium- and molybdenum-free products containing > 95% U_3O_8. (AEC Activity 5310)

Solvent Extraction Research. – In a batch countercurrent test of the recovery and separation of technetium, neptunium, and plutonium from uranium hexafluoride transfer cylinder wash by amine extraction (ORNL-2874), ~99.99% of the uranium was extracted in 11 stages from 1:1 diluted Paducah wash [~ 0.5 N HNO_3, ~ 2.5 M $Al(NO_3)_3$] with 0.3 M trilaurylamine at an aqueous to organic phase ratio of 1, and > 99.99% was stripped in eight partitioning stages with 0.5 N HNO_3 at a phase ratio of 1/2. More than 99.8% of both the technetium and the neptunium were extracted in the first five stages. Partially completed analyses indicate that neptunium partitioning by 0.1 N H_2SO_4 and technetium stripping by 1 N NH_4OH gave < 0.1% cross contamination, but that a back extraction step may be required to prevent loss of neptunium with the uranium.

In tests of reagents for the separation of americium from rare earths, extractions from 9.9 M LiCl–0.1 M HCl by 1 M tributyl phosphate were in the order Am > Eu > Ce ≅ Y, with approximate ratios 7:2:1. The behavior of yttrium indicates that the transplutonic elements are extracted in preference to the entire lanthanide series.

Continued study has confirmed previous observations that nitration products of the diluent can contribute adversely to zirconium-niobium decontamination factors in tributyl phosphate extraction processes. In recent tests Amsco 125-82 was fractionally distilled at 15 to 18 mm Hg over the range 66 to 92°C. The seven fractions, varying in volume from 5 to 35% of the initial sample, were degraded by refluxing over nitric acid at 107°C and used to prepare 1 M tributyl phosphate solutions for zirconium-niobium extraction tests. More tracer activity was extracted from nitric acid and less was stripped by nitric acid as the boiling point of the Amsco fraction increased. These results are consistent with the fact that the more complex higher-boiling components contain more sites susceptible to nitration than do the lower-boiling constituents. (AEC Activity 5310)

Ion Exchange Technology. – Work is continuing on the evaluation of phenolic resins for removing cationic activity from high-pH fission product wastes. More than 1000 volumes of ORNL low-level process water waste, containing Cs^{137} and Sr^{85} tracers and adjusted to pH 11.6, was passed through one volume of sodium-form Duolite C-3 resin at a flow rate of 0.7 ml/min per milliliter of resin before the effluent activity exceeded background, corresponding to decontamination factors for strontium and cesium of about 1000. At pH 10.6 the number of volumes of waste before breakthrough was over 600, and at pH 7.3 it was over 200.

Dowex 50W resin that had received a total dose of almost 12 whr of Pm^{147} beta radiation per gram of dry resin had a "weakly acid" capacity that was 10 to 33% of its "strongly acid" capacity, depending on the pH. Unirradiated Dowex 50W resin had negligible "weakly acid" capacity.

Apparent uranium diffusion coefficients were 1.1×10^{-7}, 1.0×10^{-7}, and 1.3×10^{-7} cm^2/sec when uranium was being loaded on 1200-μ Dowex 21K resin equilibrated with nitrate, chloride, and sulfate, respectively. The loading solution was 0.004 M UO_2SO_4 containing 0.30 M total sulfate. When resin that had been equilibrated with sulfate was eluted with neutral 1 M $NaNO_3$ and with acid 1 M NaCl–0.1 M HCl, the apparent uranium diffusion coefficients were 1.5×10^{-7} and 3.0×10^{-7} cm^2/sec, respectively. The high value during the chloride elution may have been due to the presence of acid. (AEC Activity 5310)

Extraction Reagent Performance. – Uranyl dibutylphosphate was prepared in pure form for determination of its solubility in aqueous nitric acid solutions and in 30% solutions of tributyl phosphate in Amsco 125-82 which had been previously contacted with aqueous nitric acid solutions. The solubility of uranyl dibutylphosphate in the aqueous phase increased from 0.004 to 0.816 g of uranium per liter as the nitric acid concentration increased from 0 to 3.19 M; the solubility in the organic phase in equilibrium with the aqueous phase increased, correspondingly, from 14 to 165 g of uranium per liter.

The use of Solvesso-100 as a diluent for tributyl phosphate in place of Amsco 125-82 decreased the radiolytic acid yield of 1 M tributyl phosphate solutions from 0.81 to 0.28 molecule per 100 ev, again showing the protective effect of the benzene structure. The yield of acid from 1 M di-sec-butyl phenylphosphonate, which itself contains a phenyl group, decreased only from 0.22 to 0.19 molecule per 100 ev when the diluent was changed from Amsco to Solvesso. The Amsco 125-82 initially contained 0.5% unsaturation, the Solvesso-100 about 2%. The yields of unsaturation produced by irradiating the pure diluents were 2.28 and 5.03 double bonds per 100 ev, respectively. Norit A, a charcoal, was shown to be potentially very useful in removing ruthenium, zirconium-niobium, and colored impurities from used Purex Pilot Plant solvent. (AEC Activity 5310)

Inorganic and Physical Chemistry: Thermochemistry and Calorimetry. – Measurements of the low-temperature heat capacity of K_2ReBr_6 are essentially complete. The previously observed cooperative-type transition with a maximum at 15°K was confirmed and thoroughly characterized. An evolution of heat observed at approximately 150°K was established as arising from a small cooperative-type transition with a maximum at 214°K. The transition does not appear when the sample is cooled to 200°K but is observed if the sample is cooled to 150°K and the heat evolution is allowed to take place. The energy "frozen in" is approximately 0.8 cal. (AEC Activity 5330)

Inorganic and Physical Chemistry: Physical Chemistry of Ion Exchangers.– Measurements of the selectivity coefficients for the zinc-sodium ion exchange equilibrium as a function of loading were completed on eight differently cross-linked preparations of Dowex 50 cation exchanger. These measurements

are an essential part of an experimental study to determine whether ion exchange equilibria for ions whose charges are of the same sign but unequal in magnitude can be computed from thermodynamic theory.

Measurements of the densities of the halide salts of a strong-base anion exchanger (Dowex 2) as a function of the hydration of the exchanger were completed. Apparent molal and partial molal volumes of water, $\bar{v}_w$, and of resinate were computed as a function of resinate concentration. Comparisons of the $\bar{v}_w$ values with those for aqueous solutions of ordinary electrolytes suggested that the behavior of water in an ion exchanger is very similar to its behavior in electrolytes.

According to the thermodynamic theory of ion exchange equilibria, it should be possible to calculate selectivity coefficients from ion exchanger swelling or, conversely, to calculate swelling from selectivity coefficient measurements. Previous work has demonstrated that selectivity coefficients can be calculated from swelling. A series of measurements on a weakly cross-linked anion exchanger (Dowex 2) was completed recently, and it has been confirmed that swelling can be calculated from selectivity coefficients. (AEC Activity 5330)

Inorganic and Physical Chemistry: Chemistry of Technetium. — Measurements of the absorption in the ultraviolet by dilute aqueous solutions of pertechnetate ion are useful in establishing molar concentrations, provided accurate values of the molar absorbancy indices, ϵ, are known. A collation of recent measurements of ϵ at 2440 and 2880 Å with earlier determinations at ORNL gave values of 6054 ± 31 and 2291 ± 15 cm^2/mole, respectively. The ratio of these values, 2.643 ± 0.006, has served as a useful criterion of the purity of heptavalent technetium salts.

Studies on the behavior of small amounts of technetium(VII) in alkaline solutions showed that ferrous hydroxide is an excellent reducing agent for the quantitative removal of technetium from such solutions. Further, a quantitative separation of technetium from rhenium can be effected by a ferrous hydroxide precipitate.

The solubility of $KTcO_4$ in water was measured as 0.1057 m at 25°C and 0.0344 m at 0°C. The heat of solution calculated from the temperature coefficient of the solubility is 14.4 kcal/mole. (AEC Activity 5330)

Chemistry of the Heavy Elements. — A 0.017 m UO_2SO_4 solution in 0.01 m D_2SO_4 (in D_2O, the hydrogen being 98.5 at. % D) was examined spectrophotometrically from 0.34 to 0.80 μ over a period of 43 days and over the temperature range from 25 to 251°C. The 0.42-μ band (which exhibits characteristic vibrational structure) showed a monotonic increase in absorptivity with temperature, reaching 1.74 times the room-temperature value at 251°C.

The data indicate that the spectra observed throughout were due solely to hexavalent uranium species; there was no turbidity or indication of soluble lower-valence species which absorb in the visible region. Further, while there was no evidence of solution instability at the lower temperatures, the data show a slow, steady loss of uranium totaling 9.8% in 98 hr at 251°C, the cause of which is under investigation. (AEC Activity 5330)

Nuclear Chemistry: Characterization of Short-Lived Fission Products. — The de-excitation of nuclear energy levels in Ge^{74} populated in the decay of

Ga^{74} and As^{74} was investigated by means of gamma- and beta-ray scintillation spectrometry. The single-crystal gamma-ray spectrum of Ga^{74} comprises gamma rays having the following energies (and relative intensities): 0.5 (9), 0.600 (100), 0.70 (2), 0.87 (9), 0.96 (4), 1.14 (6), 1.20 (9), 1.35 (5), 1.46 (7), 1.55 (4), 1.74 (3), 1.95 (4), 2.35 (40), 2.50 (3), 2.7 (4), 2.95 (4), and 3.2 (3) Mev. Gamma-gamma coincidence experiments were performed on the 0.50-, 0.600-, 0.87-, 1.46-, and 2.35-Mev gamma rays. The end point of the highest energy beta-ray group was observed to be at 2.7 Mev, and this same group was shown to be in coincidence with the 2.35-Mev gamma ray. These results indicate levels in Ge^{74} at 0.600, 1.20, 1.47, 2.16, 2.55, 2.95, and 3.16 Mev. The angular correlation between the two 0.60-Mev gamma rays deexciting the 1.20-Mev level was studied with an As^{74} source. The results were consistent with a 2,2,0 spin sequence and a predominantly E2 character of the upper transition.

The radiations of 22-hr I^{133} are being studied with both single- and multicrystal scintillation spectrometers. Samples of I^{133} were chemically separated from fission product Te^{133}. Analysis of a single-crystal gamma-ray spectrum showed peaks at 0.530, 0.630, 0.700, 0.779, 0.874, 1.05, 1.23, and 1.30 Mev. Gamma-ray spectra were measured in coincidence with the peaks at 0.53, 0.70, 0.87, 1.05, 1.23, and 1.30 Mev. The coincidence experiments demonstrated that some of the peaks in the single-crystal spectrum were multiple and that additional gamma rays were present below 0.53 Mev. This information, together with data obtained from single-crystal beta spectra and from beta-gamma coincidence measurements, indicates that the maximum energy beta-ray group has an energy of 1.54 Mev and decays to the 0.233-Mev isomeric level in Xe^{133}. The intense 0.530-Mev gamma ray is a ground-state transition following the decay of a 1.25-Mev beta group. (AEC Activity 5330)

Physical Chemistry of Molten Salts. — Correlations of the crystal properties of 7:6 compounds formed by the alkali fluorides with ZrF_4, HfF_4, ThF_4, and UF_4 have led to the conclusions that the ratio of the radius of the alkali cation to that of the quadrivalent cation can serve as a criterion which permits prediction as to whether formation of a 7:6 compound is possible and in which of two crystal systems the compound will occur if it forms. These correlations have led to the prediction that the compounds reported as $KF \cdot ThF_4$ and $RbF \cdot ThF_4$ should be $7KF \cdot 6ThF_4$ and $7RbF \cdot 6ThF_4$. Experimental corroboration of this prediction has been obtained. It can also be inferred from these experimental results and correlations that the 1:1 compounds of NaF, KF, and RbF with PuF_4, reported by Zachariasen, and the compound $KF \cdot AmF_4$, reported by Asprey, are actually $7NaF \cdot 6PuF_4$, $7KF \cdot 6PuF_4$, $7RbF \cdot 6PuF_4$, and $7KF \cdot 6AmF_4$. These examples and the very nearly identical unit-cell parameters of the known quadrivalent fluorides of the actinides indicate that the 7:6 compounds will be routinely observed in the fluoride chemistry of the quadrivalent actinides.

The optical properties and x-ray diffraction data for three pure compounds, $NaF \cdot FeF_2$, CrF_3, and $BeC_2O_4 \cdot 3H_2O$, have been derived. The measured optical properties of these compounds for monochromatic light of 5890 Å are as follows:

$NaF \cdot FeF_2$	biaxial, probably negative; 2V large; all indices = 1.446; polysynthetic twinning common, much right-angle twinning
CrF_3	uniaxial positive; $N_\omega = 1.568$, $N_\epsilon = 1.582$
$BeC_2O_4 \cdot 3H_2O$	biaxial; $N_\alpha = 1.428$, $N_\beta = 1.490$, $N_\gamma = 1.552$; $2V \sim 90°$

(AEC Activity 5330)

High-Temperature Aqueous Solution Chemistry (See AEC Activity 4151.1, ORNL-2895). — The temperature of separation of a second liquid phase for a series of solutions, each 0.2 m in total SO_4^{--}, in which the UO_3/SO_3 mole ratio varied from 0.1 to 1.0, was found to increase from 298°C for 0.2 m UO_2SO_4 to 380°C for the solution 0.05 m in UO_2SO_4 and 0.15 m in H_2SO_4. No phase separation was observed in solutions less than 0.05 m in UO_2SO_4 up to the critical temperature, near 380°C. Similarly, the two-liquid-phase separation temperature for a series of solutions, each 0.05 m in total SO_4^{--}, was found to increase from 327°C for a solution 0.045 m in UO_2SO_4 and 0.005 m in H_2SO_4 to 377°C for a solution 0.01 m in UO_2SO_4 and 0.04 m in H_2SO_4; the two-liquid-phase separation temperatures for another series of solutions, each 0.02 m in SO_4^{--}, increased from 351°C for a solution 0.015 m in UO_2SO_4 and 0.005 m in H_2SO_4 to 375°C for a solution 0.0064 m in UO_2SO_4 and 0.0136 m in H_2SO_4. At lower concentrations of UO_2SO_4 for each series of experiments, this component was found to be soluble in the supercritical fluid.

The range of solubility information previously reported for the system UO_3-HNO_3-H_2O from 5×10^{-5} to 5 m HNO_3 and from 135 to 300°C was extended by solubility experiments at 25, 325, and 350°C with solutions 0.05 to 1 m in HNO_3. In 1 m HNO_3 solution the solubilities of $UO_3 \cdot nH_2O$ at 25, 325, and 350°C were found to be 0.70, 0.47, and 0.45 m, whereas in 0.05 m HNO_3 the corresponding values were 0.045, 0.016, and 0.011 m. (AEC Activity 5330)

Chemical Separation of Isotopes. — The exchange of boron between boron trifluoride gas and the liquid dimethyl sulfide—boron trifluoride complex was studied from −20 to +26°C. The single-stage separation factor changes from 1.056 to 1.031 over this temperature range, with B^{10} concentrating in the liquid phase. The vapor pressures of dimethyl sulfide and of various mixtures of boron trifluoride and dimethyl sulfide were determined. The heat of the reaction $BF_3(g) + Me_2S(l) \rightarrow BF_3 \cdot Me_2S(l)$ was estimated to be $\Delta H = -10.1$ kcal/mole from −20 to +26°C. (AEC Activity 5340)

METALLURGY AND SOLID STATE PHYSICS

Metal Surface Studies. — Efforts to characterize the factors that determine the degree of protectiveness of oxide films on metals have been continued. A new apparatus for measuring oxidation rates was constructed, and the transition from the protective to the nonprotective stage of oxidation of niobium was investigated by means of extended oxidation-rate measurements with polycrystalline specimens at temperatures of 400 to 425°C. Electron microscopic studies of these heavily oxidized specimens emphasized the dependence of the oxidation rate on crystallographic orientation and revealed the development of a layerlike structure in the oxide, visible at the edges of heavily oxidized grains. This latter phenomenon was thought to be indicative of a patchlike cracking of the oxide film, consistent with the previously proposed oxidation mechanism for niobium. Preliminary oxidation-rate

studies with monocrystalline niobium specimens have been undertaken in an effort to determine the influence of crystallographic orientation on the mechanism of crack formation in niobium oxide films. (AEC Activity 5420)

THERMONUCLEAR PROJECT

DCX-1 Facility. – Preliminary observations are now at hand concerning the performance of DCX under its new configuration (field strength 10,000 gauss as compared with the former 6000 gauss; H^+ orbit radius 3.25 in. vs the former 5.3 in.; necessary changes in arc location and in the path and focus of the H_2^+ beam). They suggest that the mean containment times τ are now about 4 msec under pressure conditions which previously gave about 6 msec. The most important observation, however, is that whereas at pressures of 10^{-6} mm Hg or lower the trapped H^+ circulating beam formerly spread axially over a region about 1 ft long, now the spreading is absent. The breakup efficiency has not changed much, being at present typically 4%, so that the number of trapped H^+ particles is still somewhat in excess of 10^{12}. With no spread, however, the reduction in volume occupied implies a possible increase in H^+ density. This point is at present being investigated.

The disappearance of the spread may lead to some solution of the nature of the spreading mechanism.

An examination of curves of $1/\tau$ vs pressure for DCX has shown that they are not necessarily always linear. Some have been observed that have plateaus or actual bumps; others droop at low pressures; others flatten at low pressures. This varied behavior is thought to be probably connected with changes in the composition of the background gas in the DCX vacuum tank. It is apparent, however, that curves of this kind should not be given a naive interpretation.

New amplifiers on the detectors in DCX have given a substantial improvement in sensitivity and in the quality of the oscillograms from which τ values are deduced. (AEC Activity 5540)

PROGRAM 6000 – BIOLOGY AND MEDICINE

BIOLOGY

Drosophila Cytology and Genetics. – Algebraic models of two different sets of experiments have been formulated for maximum-likelihood estimation of the parameters. These formulations have been programed for the IBM-704 computer. The programs have met with different degrees of success. The first problem deals with tetrad analysis in a situation in which nonrandom disjunction and mortality of one of the recombinant products are occurring; the parameters of the first set of such experiments have been estimated, and slight modification of the program will allow completion of this project. The second problem involves the influence of x rays on the survival of sperm as measured by sex ratio. The algebraic formulation of this problem is considerably more complex than that of the tetrad analysis, and it is still in the process of "debugging." (AEC Activity 6130)

Pathology and Physiology. – In order to investigate the influence of partial body shielding on delayed irradiation effects, RF female mice were exposed at 12 weeks of age to 250-kvp x rays in the following groups (approximately 100 mice per group): I, 100 r, whole body; II, 300 r, whole body; III, 300 r, upper third of body; IV, 300 r, middle third of body; V, 300 r, lower third of body; and VI, 0 r (controls). The median survival times were: group I, 17.7 months; II, 15.2 months; III, 19.6 months; IV, 18.1 months; V, 18.5 months; VI, 19.4 months. The thymic lymphoma incidences in the respective groups were 25, 31, 11, 14, 18, and 18%. The myeloid leukemia incidences were 5, 19, 3, 3, 4, and 6%, respectively. The ovarian tumor incidences were 63, 56, 12, 41, 25, and 14%, respectively. These results support the concept that life shortening depends on a combination of radiation effects, only some of which are induced by exposure of part of the body. (AEC Activity 6130)

Biophysics: Electron Spin Resonance Studies. – Measurements of the decay constants or lifetimes for free radicals produced in representative proteins have been continued. In addition, studies of the dependence of radical induction on linear energy transfer are being extended to proteins. For gelatine, fast neutrons were found to be very ineffective in producing radicals, in sharp contrast to the case of the simple amino acids previously examined. Relative to cobalt gamma rays, fast neutrons (14 Mev) were 0.7 as effective in producing radicals in the amino acids and less than 0.2 as effective for gelatin. (AEC Activity 6130)

Biophysics: Radiological Physics. – A thorough compilation and statistical analysis of data on the decay of several polonium-beryllium fast-neutron sources yielded for the half lives of two Po^{210}-Be sources (with 95% confidence intervals) 140.4 ± 2.2 and 139.1 ± 0.7 days, the latter value being for the chronic radiation source, and for the half life of the reference Po^{208}-Be source 1041 ± 6 days. The half lives of the polonium isotopes are given in the literature as 138.4 and 1058 days. As a cross check on these measurements the ratios of 14-Mev neutron doses since July 1956 indicated by the long- and alpha-counter monitoring systems were analyzed; the regression formula, with 95% confidence intervals for the coefficients, was found to be

$$\frac{L}{\alpha} = 1.015 \pm 0.018 - (4.1 \pm 3.8)10^{-5}\ t \quad .$$

A device for extending the range of standard 100-r Victoreen ion chambers to 2000 r, intended primarily to make possible the necessary precise measurements of animal exposures in studies of protective compounds at the 1500-r level, has been built and is giving promising initial results.

The use of a rotating-target assembly, which provides for very close positioning of small materials, has made possible the achievement of 14-Mev neutron doses of about 80,000 rads in one day. (AEC Activity 6130)

Biophysics: Ultraviolet Studies. – A large photoreactivation (inverse dose-reduction factor of 3.2) has been found in *Pseudomonas aeruginosa*. This is the first demonstration of photoreactivation in the order Pseudomonadales, a result of some significance since many species of bacteria are not photoreactivable. This organism also shows a moderate photoprotection (inverse dose-reduction factor of 1.7). Since previously photoprotection had been

reported among bacteria only in Escherichia coli, this result suggests that it may be a more common effect than it has hitherto been suspected to be. (AEC Activity 6130)

Radiation Protection: Living Cells. – Bacteria probably have the widest range of radiosensitivities of all types of living cells known. Any attempt to explain the great differences in sensitivity between the many genera must take into account their differences in metabolism or physiology. Some appreciation for the variations in radioresistance can be gotten by an investigation of conditions which influence the sensitivity of a single species. Several strains of Escherichia coli show relatively large effects of cultural conditions on x-ray sensitivity. The usual culture medium (nutrient broth or peptone) is poorly buffered, and as a result of the metabolic activity of the bacteria the pH increases during growth from 6.8 to about 8.4. If glucose is added to such a medium, the cells reduce the pH from 6.8 to 4.8. Addition of 0.2 M phosphate buffer to either culture, however, results in no pH change during the growth cycle. Cells from cultures of these three kinds are quite different in their sensitivity to x rays, the sensitivity increasing with increasing final pH of the culture. Although the most resistant cells (final pH 4.8) yield a sigmoid survival curve, the shape of the curve does not result from structural abnormalities such as multicellularity, clumping, or multinuclearity (microscopic analyses give no indication of multicellularity or clumping, and chemical analyses of resistant and sensitive cells show little, if any, difference in the amount of deoxyribonucleic acid per cell).

A change in pH during growth seems to be required for development of resistance, since neither resistant nor sensitive cells show any effect of the pH of the suspending medium either during irradiation or during postirradiation incubation on plates. The organic constituents of the plating medium following irradiation influence the surviving fraction of both resistant and sensitive cells. Higher survival is obtained on complete, natural media than on minimal, synthetic media. The resistant cells, however, show a greater effect of the postirradiation medium than do the sensitive cells. (AEC Activity 6230)

Mammalian Radiation Recovery. – Either isologous or homologous blood can reverse the ability of homologous bone marrow to promote survival in lethally irradiated mice. The injection of homologous bone marrow and 0.25 ml of isologous blood into lethally irradiated mice resulted in 100% mortality by day 15. The same mortality was seen by day 35 when homologous blood was substituted for isologous blood.

Investigation of the ability of inorganic salts to protect mouse bone marrow against freezing and thawing showed that in certain concentrations NaI, NaBr, $NaNO_3$, Na_2SO_4, and NaSCN are protective, while $NaNO_2$ is not.

Single-cell plating experiments with the hypotetraploid clonal human cell line GH7A6 have shown that while the curves of x-ray survival S vs dose D fit the expression $S = 1 - (1 - e^{kD})^n$ in a general way, neither k nor n is a constant, as is implied by the chromosome aberration theory of mammalian cell killing. Rather, they are a function of cell density and of time of irradiation. Preliminary calculations based on the experimental results indicate that at least two separate cell-killing processes are operating in this system.

The subcutaneous injection of nutritive solutions extends the survival of mice exposed to x-ray doses between 1700 and 5000 r. The treatment is more effective in mice whose food and water are withheld than in mice allowed food and water ad libitum. Autoradiographs made after repeated (at 3-hr intervals) injections of tritiated leucine into mice indicate that abnormal enlargement of intestinal crypt cells is associated with an increase in cellular protein as well as an increase in ribonucleic acid. (AEC Activity 6230)

Radiation Immunology. — A model has been devised whereby the antibody-forming capacity of a known number of cells can be determined quantitatively. In this model, the cells are transplanted intravenously into lethally x-irradiated isologous mice, serving as a tissue culture in vivo, and the test antigen is injected by the intravenous or intraperitoneal route at various times thereafter. The animals are bled within two weeks after the transplantation. Previously it took seven weeks to obtain a comparable result. Recently emphasis has been placed on determining the variables that may affect antibody response, such as the time of antigen injection relative to the time of transplantation, the time of bleeding for maximum antibody response, and the time of preimmunization and dose of antigen injection for maximum secondary antibody response.

The passive cutaneous anaphylaxis reaction (PCA test) in mice is being investigated for two reasons: as a model to study the nature of transplantation antigen, and as a model to further understand one aspect of an antigen-antibody reaction in vivo. The preliminary data clearly show the importance of the genetic relation between the source of antisera and the test animal; thus in mice, anti-BSA (bovine serum albumin) sera of comparable titers can be rated in the following order: mouse, rat, rabbit, and chicken. Radiation injury (950 r) seems to have no effect in altering the reaction, suggesting that this phenomenon is not necessarily mediated through viable lymphoidal or other radiosensitive cells.

Continuing studies on the age factor in mice relative to their immunologic capacity have shown that immunization of an animal at 12 weeks of age with an optimum dose of antigen results in a sustained production of antibodies for a period of at least 18 months after antigen injection, at which time a plateau in the level of circulating antibody is reached. That this production of antibodies is not due to nonspecific antigenic stimuli is revealed by the absence of antibodies to the test antigen in noninjected control mice. (AEC Activity 6230)

PROGRAM 8000 — WORK FOR OTHERS

International Reactor Program at ORNL. — Twenty-five students representing 15 different countries are currently enrolled in the two foreign programs, Reactor Operations Supervision and Reactor Hazards Evaluation. The students have completed 12 weeks of school since the beginning of classes in November. Thirteen Hazards Evaluation students of the 1959 session have completed hazards reports on reactors of four different types and have either returned to their homelands or accepted assignments elsewhere. Tentative plans have been made for starting a third session of the international program in the fall of 1960. (AEC Activity 8717)

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STATUS AND PROGRESS REPORT

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