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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JULY 1959

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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Oak Ridge, Tennessee
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U.S. ATOMIC ENERGY COMMISSION

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OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT

July 1959

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

PROGRAM 2000 — SPECIAL NUCLEAR MATERIALS

Power Reactor Fuel Reprocessing Pilot Plant. — The Pilot Plant completed another 30 days of continuous operation (bringing the total to 81 days), and 12 tons of BNL irradiated uranium fuel, containing 6 kg of plutonium, was processed. The uranium and plutonium products met all specifications. Measured uranium and plutonium losses totaled 1.4 and 0.6%, respectively; approximately 60% of the uranium loss occurred during development of a satisfactory bottom interface control procedure for the second uranium cycle extraction column. During the last 38 days of operation the co-decontamination flowsheet was used, and second uranium cycle extraction columns were operated with the organic phase continuous; over-all gross decontamination factors were consistently 10^5. (AEC Activity 2790)

PROGRAM 4000 — REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. — The HRT operated continuously during July at power levels as high as 2.8 Mw. So far, a total of 720 Mwhr has been accumulated in run 20, in which fuel-stability behavior has been studied at a system temperature of 260°C and system pressures of 1400 and 1750 psig.

While operating at 1400 psig, there were no indications of fuel instability at power levels as high as 2.0 Mw. There were power excursions at 2.4 and 2.8 Mw. However, the excursion frequency was low; the average was five and less per day. Only three of the excursions were greater than 10% of the operating power; the largest was 22%. No more than 300 g of uranium was missing from the circulating inventory at 2.8 Mw.

When the pressure was increased to 1750 psig, instability was immediately demonstrated at 2.8 Mw by a rapid decline in the system temperature, which indicated rapid uranium loss from the circulating inventory. This same phenomenon was found to occur at 2.0 and 1.5 Mw. However, no power excursions were observed at any of these levels.

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The catalytic-recombiner difficulty mentioned last month was corrected by reactivating the catalyst above 400°C. However, after removing the source of high-temperature (500°C) steam, the efficiency of the catalyst became less than 100% but remained high enough for power operation to continue. (AEC Activity 4151.3)

Mathematics and Computation: Homogeneous Reactor Development. — The standard two-group two-region criticality code for the Oracle was reprogramed for the IBM-704. Provisions have been made for including fast fissions and for obtaining temperature coefficients of reactivity, adjoint fluxes, and the multiplication constant for reactors with specified fuel concentrations. (AEC Activity 4151.1)

Reactor Analysis. — Calculations concerning the breeding ratio and reactivity changes due to thoria settling were performed for a number of cylindrical, two-region thorium breeder reactors employing a fluidized thoria blanket. The core was a 4-ft-dia cylinder 12 ft long; the power level was 400 Mw (thermal). Power densities remained at high levels all around the core wall, so that cooling must be provided at the top and bottom core-tank surfaces as well as at the side. Dilution of the thoria bed with equimolal amounts of alumina was found to reduce the breeding ratio by 0.02. Excessive reactivity changes accompanied thoria settling when the blanket region was initially only as high as the core; by increasing the initial bed height, the reactivity change associated with thoria settling was decreased to manageable proportions. (AEC Activity 4151.1)

Homogeneous Reactor Instrumentation. — A new electric system for process instrumentation was tested and then installed as a pressure-control loop in a high-pressure pump test stand for investigation of long-term stability and reliability. The system is unique in that static saturable reactors and rectifiers (magnetic amplifiers) are used to amplify low-level signals and to derive the control functions. Previously available commercial equipment for general process instrumentation has been pneumatically powered or has utilized vacuum tubes, which do not have the inherent reliability of electric static-component systems. (AEC Activity 4151.1)

HRP Design. — Designs were completed for the following remote-maintenance tools for use at the HRT: "sector-type" hole saws, a corrosion-specimen retrieving tool, an arc-type screen cutter, and a tool to machine the inside diameter at the weld of the core access flange to the pipe. A miniature television camera was adapted for viewing operations inside the HRT core.

Calculations indicate that no inhalation hazard will exist in the event of rupture of a heat exchanger tube at the HRT if the combined leak rate of the steam-system "stop" valves, when closed, does not exceed 4 lb/min. (AEC Activity 4151.1)

Dynamic Solution Corrosion Tests. — Heat transfer studies were made by using a loop equipped with a window to allow observation of an electrically heated wire. Preliminary observations indicate a difference between boiling at a hot surface in water and in uranyl sulfate solution.

At the same temperature (200°C) and over a range of pressures (steam plus oxygen) at heat fluxes of 10,000 to 100,000 $Btu \cdot hr^{-1} \cdot ft^{-2}$, the vapor bubbles in uranyl sulfate tend to cling to the hot wire surface, whereas the vapor bubbles in water are detached more easily and form bubble columns typical of nucleate boiling. The heat transfer coefficient is correspondingly lower in uranyl sulfate solution than in water. (AEC Activity 4151.1)

In-Pile Autoclave Solution Corrosion Studies. — Reported work of other investigators has shown that alloys of zirconium and 1% and less of either iron, chromium, or nickel which are heat treated at 800°C exhibit a corrosion resistance in 500°C steam which is better than that of Zircaloy-2. In order to determine if this improved corrosion resistance carries over to in-pile conditions in uranyl sulfate solutions at lower temperatures, an in-pile autoclave test was made to compare the radiation corrosion of a Zr—1% Cr alloy with that of Zircaloy-2. A D_2O solution of 1.3 m UO_2SO_4, 0.04 m $CuSO_4$, and 0.04 m D_2SO_4 was employed. The exposure temperature was 275°C, and the power density in solution was 36 w/ml during most of the radiation exposure. This solution composition was selected in order to provide conditions under which the relative contribution of adsorbed uranium to the effective power density would be small, and to enable the attainment of the high solution fission power density for the comparative test.

Weight losses of three specimens each of the two materials indicated that the Zr—1% Cr alloy corroded at 12.4 mils/year (mpy) and the Zircaloy-2 at 12.0 mpy at the fission power density of 36 w/ml. Analyses of samples removed from the Zircaloy-2 and Zr—1% Cr specimen surfaces by acid pickling after removal of the loose scale by drying and by brushing showed about 8 $\mu g/cm^2$ of uranium on the Zircaloy-2 and 4 $\mu g/cm^2$ on the Zr—1% Cr alloy specimens. These amounts, if present during exposure, would have a negligible effect on the irradiation intensity at the surfaces, compared with that arising in the solution adjacent to the surfaces.

The Zircaloy-2 corrosion rate in this test is about 8% higher than that estimated from the equation

$$\frac{1}{R} = \frac{2.23}{P} + 0.025,$$

which expresses the relationship between corrosion rate, R, and solution fission power density, P, in the two previous 280°C autoclave experiments which employed 1.3 m UO_2SO_4 solution in H_2O, and in the 280°C loop experiments in which uranium sorption is believed to have been negligible. The comparison with the equation was made after making a small correction corresponding to the 275°C (rather than 280°C) operation of the present test and a small correction to account for the fact that the experiment was exposed to less than maximum (36 w/ml) power density for several hours. The difference between the calculated and experimental values is probably within experimental error.

It is concluded that the radiation-induced corrosion rates of the Zr—1% Cr alloy and the Zircaloy-2 were about equal in this experiment. (AEC Activity 4151.1)

Dynamic Slurry Materials Tests. — Mean 500-hr attack rates of 0.3 and 0.7 mil/year (mpy), respectively, were observed for the austenitic stainless steel piping (at 5 fps) of two 100A test loops at 200°C when atmospheres of oxygen (500 cc per liter of slurry) and of deuterium (250 cc per liter of slurry) were used with slurries (300 g of Th per kg of D_2O) of an experimental Th—15% U oxide preparation of 2.3-μ average particle size. Attack rates for pin specimens exposed at 20 fps, with atmospheres of O_2 and D_2, respectively, were (in mpy): type 347 stainless steel, 0.1 and 0.3; Inconel, 0.2 and <0.1; titanium RC-55, 0.1 and 0; Zircaloy-2, 0 and 0; and Incoloy, 0.1 and 0.1. Attack rates for specimens exposed at 40 fps in the respective atmospheres were (in mpy): type 347 stainless steel, 0.4 and 16; Inconel, 1 and 2; titanium RC-55, 0.1 and 0; Zircaloy-2, 0.1 and 0; and Incoloy, 0.8 and 6.4.

The ratio U^{4+}/total U, which was initially 0.37, did not change during circulation with the oxygen atmosphere but increased to 0.67 with the deuterium atmosphere. Soluble uranium was not detected (<5 ppm) during either test. Handling qualities of the Th—15% U oxide slurry under the above conditions were normal and satisfactory. (AEC Activity 4151.1)

HRP Service Corrosion. — The thermal-neutron fluxes during exposure in the HRE-2 were computed for samples cut from the core screens and for corrosion specimens contained in the blanket region of the reactor. The fluxes were determined by comparisons between the Zr^{95}-Nb^{95} activities in the reactor samples and in Zircaloy-2 control specimens exposed in the LITR. The screen specimens were exposed during HRE-2 runs 13, 14, 16, and 17; calculations show, however, that about 70% of the activity in these specimens at the time of counting was induced during run 17, and about 25% during run 16. The corrosion specimens were exposed during run 17 only.

Comparison between the activities of the four screen samples and the Zircaloy-2 control, uncorrected for differences in neutron activation cross sections at LITR and HRE-2 neutron temperatures, indicated thermal-neutron fluxes of 4.1, 3.4, 3.0, and 2.9 x 10^{13} neutrons·cm^{-2}·sec^{-1}, respectively, at a core power of 2 Mw. The screens from which these samples were cut have been identified as screens 9, 8 or 7, 6, and 5 and, accordingly, were located 10.9, 12.6 or 14.3, 15.7, and 16.8 in. below the reactor core center. In a semilogarithmic plot of relative induced activity against position, the measured activity values fall very near a straight line when it is assumed that the sample of uncertain identification is from screen 7.

Comparisons between the induced activities of the specimens from the blanket and the control, again uncorrected for differences in activation cross sections due to neutron temperature differences, showed thermal fluxes of from 1.1 to 1.2 x 10^{13} neutrons·cm^{-2}·sec^{-1} at a reactor core power of 2 Mw. Two of the specimens were located about 6.5 in. above the reactor core center line, and two about the same distance below. All four specimens were located 4.5 in. from the core wall and 20.5 in. from the core center. (AEC Activity 4151.1)

MARITIME SHIP REACTOR PROGRAM

Maritime Ship Reactor. — The NS "Savannah" was launched on July 21, 1959, thus ending another phase of the Nuclear Merchant Ship Program. The launching occurred approximately 14 months after the keel laying ceremony.

The review and evaluation of the design for the Nuclear Merchant Ship Reactor (NMSR) were continued; however, the emphasis on review work is now shifting from the reactor design to the reactor operating procedures. ORNL field inspectors continued to act as inspection agents for the AEC, witnessing inspections and tests on various reactor system components during fabrication.

All major components for the ORR pressurized-water loop have been procured. Installation of the loop equipment continued approximately as scheduled.

The first draft of the "Health Physics Recommendations for the Operation of the N. S. Savannah" has been prepared and submitted to the AEC for review. Preparation of this document in final form is awaiting AEC review and comments. (AEC Activity 4510)

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR-1). — The APPR-1 at Fort Belvoir, Virginia, has been operating satisfactorily on an extended power run since early June 1959. This power run will be terminated late this month to allow core physics measurements. The core has operated for 12.2 Mwyr, the expected life being 15 Mwyr.

The examination of two boron-iron control rod absorbers in the Solid State Division hot cell facility is essentially complete. These rods were received at ORNL last month, after having operated in the APPR-1 for approximately 10 Mwyr. The examination of these rods, one of which was a central control rod and the other an eccentric control rod, showed clad-frame and clad-core separation of the plates on the high-burnup ends. Also noted was a 45° crack in the type 304L stainless steel cladding on the outside face of one plate in each rod. This cracking was noted on the high-burnup tip of the rods over the iron-boron core, and it extended to the edge of the plate frame.

The specifications for core II APPR-1 fuel elements and europium oxide control rod elements have been completed and will be released this month. (AEC Activity 4630)

GENERAL REACTOR RESEARCH

Reactor Evaluation Studies. — A study is being made of maintenance problems associated with use of unclad fuel elements in gas-cooled reactors. Efforts are directed toward estimating the costs associated with remote maintenance, decontamination and direct maintenance, or with unique designs that facilitate maintenance. The costs attributable to the contaminated coolant (over and above those associated with operation of the same reactor with no contamination in the primary coolant) were estimated to be 0.7 mill/kwhr for a 3100-Mw (thermal) reactor, and 1.8 mills/kwhr for a 350-Mw reactor.

The reactivity-lifetime program for evaluation of fuel value was modified to include spectral-hardening effects that accompany changes of fuel enrichment in initial fuel loadings. (AEC Activity 4160)

Reactor Safety Studies. — The components required for the preliminary tests of the new BSF stainless steel—UO_2 core (the BSR-II) were fabricated. The first test loading resulted in a critical mass of 5877.12 g with an excess reactivity of less than 1 dollar. This was within 3% of the predicted critical mass. Several additional critical arrays have been assembled, and the control plates have been calibrated to the limit of the core excess. The reactor, control system, and all special components required to conduct BSR-II tests at the SPERT-1 facility are expected to be ready for shipment by August 14. The analog computer (reactor simulator) is being programed to simulate the expected transient behavior of the reactor, and computer test results are expected to be available prior to the SPERT-1 tests.

In order to obtain data to aid in the evaluation of the SPERT-1 tests, the BSR-II (loaded in the Pool Critical Assembly) was coupled with the BSR-1 by means of a long, small-diameter tube. A chopper was installed near the center of the tube. With this apparatus the BSR-II was pulsed with neutrons from the BSR-I, and a study of the neutron lifetime in the BSR-II was made. The results indicate that $\ell/\beta_{eff} = 3.2$ msec ± 5%, where ℓ is the prompt neutron generation time and β_{eff} is the delayed neutron fraction. (AEC Activity 4171.1)

Long-Range Planning Studies. — A comparative study of thorium breeder reactors has been initiated. It will be concerned with the relations between breeding ratio, doubling time, and fuel cost for a wide variety of reactor types. (AEC Activity 4410)

Fluoride Volatility Processing. — The liquid medium for the dissolution of zirconium-containing fuel elements by HF was modified to contain ZrF_4 initially, thus lowering the temperature of the melt and, it is hoped, decreasing corrosion of the reaction vessel. In one 8-hr test at 650°C the dissolution rate of zirconium in 25-37.5-37.5 mole % ZrF_4-NaF-LiF was 1.62 $mg \cdot min^{-1} \cdot cm^{-2}$. This rate is sufficiently high for process use and warrants further investigation with ZrF_4 in the initial melt.

The dry hydrofluorination reactor was modified to allow operation at 950 to 1000°C and to provide means of subliming ZrF_4 from the heated reaction chamber to a cold collection chamber. In three experiments to determine the effect of varying amounts of noncondensibles on the desublimation of ZrF_4, nitrogen was used to transfer to the condenser the ZrF_4 vapor from the ZrF_4 solid introduced to the reactor. Complete desublimation resulted in all three tests. A fourth experiment was the actual gas-phase hydrofluorination of zirconium metal, with hydrogen and unreacted hydrogen fluoride as the sweep gas. With nitrogen sweep gas rates varying from 2 to 30 scfh, the ZrF_4 transfer rate varied from 150 to 410 g/hr; with H_2 + HF sweep gas at a rate of 19 scfh, the ZrF_4 was transferred at a rate of 978 g/hr. (AEC Activity 4442)

Power Reactor Fuel Reprocessing: Sulfex Process. — In comprehensive tests on unirradiated specimens to determine the mechanism of stainless steel passivation and means of breaking it, passive specimens of type 304 stainless steel were consistently produced by boiling specimens for 1 hr in 15.8 M HNO_3—0.025 M Cr^{+++}. Depassivation time decreased from several days to 30 min with increasing H_2SO_4 concentration from 6 to 12 M in the presence of 0.01 M HNO_3; and at constant H_2SO_4 concentration, it increased

from 2 hr to several days with HNO_3 concentration from 0.001 to 0.01 M. The effect of temperature was more important than that of the H_2SO_4 concentration. The addition of 0.1 M formic acid to refluxing 6 M H_2SO_4 effectively destroyed nitrate ion up to 0.01 M HNO_3. Stainless steel surfaces brazed together with Nicrobraz 50 were not passivated in 6 M H_2SO_4.

Passive films on stainless steel in contact with platinum were not depassivated after several hours in boiling 6 M H_2SO_4. In decladding tests on an irradiated (~1000 Mwd/ton) Yankee Atomic fuel pin (in a platinum basket), formic acid did not break passivation in 1 hr. However, the pin was depassivated in less than 5 min in 12 M H_2SO_4; the dissolvent was then diluted to 6 M H_2SO_4 and decladding completed in 1.25 hr. Uranium and plutonium losses after a total reaction time of 2 hr were 0.07 and 0.18%, respectively. These tests will be repeated in a Ni-o-nel basket using boiling 0.1 M formic acid-6 M H_2SO_4 to determine whether passivation of irradiated samples can be broken by formic acid in the absence of platinum.

Decladding of simulated Consolidated Edison fuel pins containing hydrogen-fired high-density (~95% of theoretical) 96% ThO_2–UO_2 pellets with boiling 6 M H_2SO_4 resulted in uranium losses of 0.009 and 0.04% in decladding times of 1 and 5 hr, respectively.

After 25 Sulfex-Thorex cyclic dissolutions, the Ni-o-nel dissolver showed an over-all corrosion rate of 2.9 mils/month. Local attack around poor welds was as high as 50 mils/month. (AEC Activity 4442)

Power Reactor Fuel Reprocessing: Darex Process. – Heat transfer coefficients of 310 to 430 $Btu \cdot hr^{-1} \cdot ft^{-2} \cdot (°F)^{-1}$ were measured between boiling Darex solution and condensing steam through a 0.109-in. titanium wall for temperature differences of 30 to 68°F.

Darex feed clarification tests on APPR fuel solutions were continued for both chloride-containing and chloride-free solutions. For raw dissolver solution, the hydration of SiO_2 at 100°C was about six times more rapid than at 80°C; the rate of growth of SiO_2 particles was doubled between 80 and 100°C. Fluoride ion increased both the rate of hydration and the silica removal; the increase was more pronounced at 80 than at 100°C. After filtration of SiO_2 from chloride-free Darex solution, boiling 30% NaOH removed the silica from the porous metal filters; 2 M HNO_3–0.04 M F–0.2 M Al^{+++} was less effective but still satisfactory. Corrosion of 300 series stainless steels by the latter reagent after a 168-hr exposure at 25°C was negligible.

In Darex decladding tests with unirradiated H_2-fired 96% ThO_2–UO_2 stainless-steel-clad Consolidated Edison fuel pins, the uranium loss in a 1-hr decladding in boiling 5 M HNO_3–2 M HCl was 0.03%; the loss in a 4-hr test was 0.05%.

Darex-Thorex corrosion tests on titanium were continued. In middle and final Thorex dissolver solutions containing no Al^{+++}, titanium showed maximum rates of 0.22 mil/month over 504 hr. In Thorex solutions containing more than 0.04 M Al^{+++}, pitting produced local attack rates on titanium as high as 1.4 mils/month. Initial Thorex solutions contaminated with chloride (<100 ppm) produced pitting on titanium corresponding to maximum local

attack rates of ~7 mils/month. Pitting attack was not shown by the Darex-Thorex titanium dissolver after 25 cyclic dissolutions, but there was a slight etch near weldments in the vapor phase. (AEC Activity 4442)

Power Reactor Fuel Reprocessing: Processing of UC_2-Graphite Fuel. — More than 99% of the uranium was recovered from simulated UC_2-graphite fuels containing 5% or more uranium by grinding and leaching the fuel in boiling 15.8 M HNO_3; fuel fabrication history did not affect recovery. With fuels containing less than 5% uranium, however, losses generally increased with the degree of graphitization and the density of the uranium. In a test with a high-density 0.7% U—graphite fuel ground to -200 mesh, only 87.3% of the uranium was recovered in two successive leachings with boiling 15.8 M HNO_3. In a poorly graphitized sample of TREAT prototype fuel containing 0.2% uranium, leaching of -4 +8 mesh material resulted in recovery of 99.5% of the uranium.

A 20-g piece of high-density 9.7% U—graphite fuel was disintegrated in liquid bromine in 1 hr at 25°C. About 70% of the powder passed through a 20-mesh sieve. (AEC Activity 4442)

Power Reactor Fuel Reprocessing: BORAX-IV Flowsheet. — A three-step flowsheet was developed for the dissolution of BORAX-IV fuel. The aluminum cladding is dissolved in boiling 2 M NaOH—1.78 M $NaNO_3$ with a uranium loss of less than 0.03%. Then the lead bond is dissolved in boiling 1.5 M HNO_3, with an additional uranium loss of about 0.1%. The 93% ThO_2-$UO_{2.3}$ core pellets are then dissolved in boiling 13 M HNO_3—0.04 M NaF—0.1 M $Al(NO_3)_3$. (AEC Activity 4442)

Power Reactor Fuel Reprocessing: Solvent Extraction Studies. — A new acid Thorex flowsheet was developed which eliminates the need for aluminum as a salting agent in the aqueous phase. This process uses 42.5% tributyl phosphate (TBP) in Decalin as the extractant rather than 42.5% TBP—Amsco, which is currently used in the acid-deficient Thorex process. Decontamination factors of 600, 90, 300, and 60,000 for gross gamma, ruthenium gamma, zirconium-niobium gamma, and rare earths, respectively, were achieved with the TBP-Decalin solvent. These factors are from 1/10 to 1/3 as high as those in the acid-deficient Thorex system. The higher solubility of thorium in Decalin prevents third-phase formation, which occurs in acid TBP-Amsco systems.

TBP labeled with P^{32} was used to study the fate of TBP in the Thorex process. The solubility of TBP in the aqueous phase [containing 40 g of Th per liter and 0.008 M in $Al(NO_3)_3$] in contact with 45% TBP—Amsco was 1.08×10^{-3} M; the solubility of gross TBP degradation products was 7×10^{-5} M. About 93% of the TBP was removed from the aqueous phase by two 1/10-volume Amsco washes, and about 88% by steam distillation. Chemical washing studies showed that 80 to 90% of the fission product activity and ~1% of the TBP degradation products are removed from degraded 42.5% TBP—Amsco solvent by washing with 0.01 M HNO_3 and that, after subsequent washing with alkaline solution, filtration is required to remove the precipitated metallo-organic compounds. About 95% of the TBP degradation products were removed from TBP-Amsco by washing with Na_2CO_3 followed by filtration of the solvent.

In studies on solvent recovery, treatment with talc, magnesite, $BaCO_3$, HCl, H_2SO_4, H_3PO_4, HNO_3, H_2O_2, or methanol did not give improvement over standard lime slurry methods. (AEC Activity 4442)

Power Reactor Fuel Reprocessing: Mechanical Processing. — A device for handling deformed SRE fuel tubes, for which the standard SRE decladder is not adapted, was designed, fabricated, and successfully tested. This machine requires that the fuel tube must first be cut into 6-in. lengths. The NaK bonding material from SRE fuel was safely removed by reaction with steam to produce innocuous caustic. The 98 cc from a single tube reacted completely in 20 min at a steam rate of 3 g/min; a reaction temperature of 290 to 320°C was measured.

Fuel bundle shearing was shown to be feasible in tests at ORNL, and under subcontracts at Clearing Division of U.S. Industries and Birdsboro Steel Foundry and Machine Company. These tests indicated that shearing effectiveness was independent of the shape of the shearing blade; in all cases closure of the tube ends was 80% or less of the original cross section, and in some cases tube expansion occurred. Ferrules joined to the tubes with Nicrobraz 50 were readily broken loose in the shearing operation. In general, cut sections were separated by tearing of the tubes rather than by true shearing. The best results were obtained when the blade clearance was equal to or greater than the wall thickness of the tubing. In tests with 35-mil-wall tubing, wipedown was excessive at and below 22-mil blade clearances, resulting in excessive blade wear. High blade speed also caused excessive blade wear as a result of galling of the blades by small metal particles which became welded to the top blade. Tests are in progress to evaluate blade life for optimum blade speed and separation. (AEC Activity 4442)

Power Reactor Fuel Reprocessing: Zirflex and Perflex Processes. — Additional data were obtained in corrosion tests. In Zirflex cyclic dissolutions, welded type 309Cb stainless steel corroded at over-all rates of less than 3 mils/month but showed cracking and pitting. In Perflex process corrosion tests in 1 M HF—0.05 M H_2O_2, Ni-o-nel, Haynes 25, Carpenter 20, pretreated Monel, and 90 Ag—10 Cu alloy all showed rates greater than 14 mils/month in a 48-hr exposure. In 1 M HF—0.02 M $K_2Cr_2O_7$, Monel showed rates greater than 800 mils/month. (AEC Activity 4442)

Waste Treatment and Disposal. — Evaporation to dryness and calcination of residual solids is being studied as a means of preparing high-activity wastes for permanent disposal. Batch evaporation and calcination at 400°C of synthetic Purex, Thorex, TBP-25, and Darex wastes showed that the solid residues contain 0.1, 4, 17, and 1%, respectively, of the nitrate originally present. From 1 to 4% of the nitrate was found in the off-gas downstream from the condenser, and the remainder was recovered in the condensate. In the case of Darex waste containing 175 to 1750 ppm HCl, 10 to 15% of the chloride remained with the solids after calcination at 600 to 800°C.

Concentrated Darex waste was converted to solids in an 8-in.-dia by 48-in.-long stainless steel calcination pot maintained at 1000°C. At a feed rate of 2.75 liters/hr, the pot was filled to within about 6 in. of the top with a solid of density 1.14 g/cc containing about 0.2

wt % nitrate (0.4% of original). The flat bottom of the pot bulged 1 in. due to an internal pressure, which was postulated to have been produced by gas from decomposition of metal nitrates under a semisolid seal of wet paste in the vessel.

The thermal conductivity of calcined solids from TBP-25 waste varied linearly with temperature from 0.065 $Btu \cdot hr^{-1} \cdot ft^{-1} \cdot (°F)^{-1}$ at 235°F to 0.40 at 1575°F. The measured values of thermal conductivity were fitted empirically to a theoretical equation, but no means has yet been found for predicting the conductivity from theoretical considerations.

In the batch evaporation to dryness of synthetic Purex and Darex wastes with tracer levels of ruthenium activity, the ruthenium retention in the residual solids varied from 28 to 54%; the condensate contained 23 to 41% of the ruthenium. When TBP-Amsco was added to Purex waste, the percentage of total ruthenium in the condensate decreased from 28 to 4. Changing from glass equipment to stainless steel reduced the ruthenium in the condensate from 28 to 20%. In stainless steel, evaporation under a vacuum of 23 in. Hg increased the ruthenium in the condensate from 20 to 44%. Neutralization of the Purex waste before evaporation decreased the ruthenium in the condensate to 0.2%. (AEC Activity 4452)

MOLTEN-SALT REACTOR (MSR) PROGRAM

Chemistry. — A preliminary phase diagram for the system $NaF-ThF_4-UF_4$ was prepared. As a consequence of the close similarity between Th^{4+} (radius 1.05 Å) and U^{4+} (radius 1.10 Å), there are extensive regions of solid solution in this system.

Cryoscopic measurements show that, in dilute solutions with NaF as solvent, Ni^{++} gives slightly greater negative deviations from ideality than Mg^{++}, presumably as a result of the greater polarizability of Ni^{++}.

The melting point of SnF_2 was found to be 218°C (compared with the literature value of 210°) and the boiling point 825°C.

The solubility of CO_2 in $NaF-BeF_2$ (57-43 mole %) decreases up to 600° and then increases with increasing temperature.

Vapor pressure studies on the system $LiF-UF_4$ indicate an azeotrope at 80% UF_4.

An evaluation of coprecipitation procedures for removing nuclear poisons from molten fluoride fuels is continuing. To provide information on rates, LaF_3 was added to a simulated breeder fuel at 600°C in an amount calculated to precipitate 50% of an initial 1000 ppm of radioactively labeled CeF_3. The expected reaction was 60% complete in 5 min, 80% in 20 min, and 90% in 1 hr.

Fuel melts of a type having low oxide solubility penetrated graphite in a normal manner in response to pressure if the graphite was given a pretreatment with hydrogen to remove oxide. This further confirms the suggestion that the previously reported anomalous behavior was due to plugging by UO_2.

A typical breeder fuel penetrates ordinary graphites of the permeable type to give weight gains of about 20% at 75 psia and 1300°F.

Periodic sampling of pumped corrosion test loops showed that a satisfactorily low steady-state level of 500 ppm of chromium, a corrosion index, was still maintained after nine months of circulating $LiF-BeF_2-$

ThF_4-UF_4 (62.0-36.5-1.0-0.5 mole %) in INOR-8. In order to conserve the liquid level in this loop, no further samples will be taken until termination.

A similar Inconel loop, which is apparently exhibiting oxidation from an unidentified extraneous source, is holding steady at 3000 ppm of chromium after eight months.

An INOR-8 loop carrying LiF-BeF_2-UF_4 (70-10-20 mole %) contains only 230 ppm of chromium after 2000 hr of operation.

The compound Na_3InF_6 was prepared by the reaction between indium metal and a melt containing BiF_3. (AEC Activity 4152.1)

Dynamic Corrosion Studies. — An INOR-8 pump loop, operated to determine the compatibility of INOR-8 and graphite in a fluoride fuel medium, was examined after one year of successful operation. Metallographic examination of a section, removed from the loop wall, that had been at the maximum temperature revealed light surface roughening and pitting to less than 1/4 mil. A layer approximately 1/3 mil thick was also evident in this sample. No evidence of carburization was found in any of the sections examined. Chemical analysis of the salt circulating in the loop showed little change in the concentration of impurities from the analysis of the salt before the test. (AEC Activity 4152.1)

Mechanical Properties Studies. — A series of tensile tests at different temperatures was completed on INOR-8, testing the effects of carburization, notches, and aging treatment.

Notches increase the tensile strength of noncarburized specimens at all temperatures. The degree of strengthening is very slight for carburized specimens, however, and is not significant at the higher temperatures.

Carburization decreases the tensile strength of both smooth and notched specimens at room temperature, 1000°F, and 1200°F. The 0.2% yield strengths of the carburized smooth specimens are, in general, higher than the corresponding noncarburized values at 1000, 1200, and 1500°F.

The ductilities of both smooth and notched specimens are decreased by carburization at room temperature, 1000°F, and 1200°F. The data for noncarburized specimens at 1500°F are too scattered for comparison.

Aging treatments have no clearly defined effect on the strength properties of the notched specimens. (AEC Activity 4152.1)

Fuel-Graphite Carburization Test on INOR-8 and Inconel. — A fuel-graphite carburization test containing INOR-8 and Inconel was terminated after 6000 hr of operation at 1300°F. Room-temperature tensile-test results show practically no difference between the control and test specimens. High-temperature (1250°F) tensile strength and elongation are 72,800 psi and 16.5% (in 2 in.) for the control INOR-8, and 74,500 psi and 16.0% for the "carburized" INOR-8 (specimen exposed to fuel plus graphite). Chemical analyses on the control and test specimens show no significant differences in carbon content. (AEC Activity 4152.1)

Sodium-Graphite Carburization Test on INOR-8 and Inconel. — The last of the sodium-graphite carburization tests was terminated after 4000 hr of operation at 1400°F. Tensile tests at room temperature indicate

that the elongation of the carburized specimens was severely reduced (5 to 6% in 2 in.). No reduction of elongation was observed at 1250°F. (AEC Activity 4152.1)

Pump Development. — The third test of a helically grooved bearing in the salt-lubricated journal-bearing tester was completed. The total operating time was 215 hr at 1200°F, 1200 rpm, and loads up to 500 lb. During the latter phase of the test, journal rotation was started and stopped 260 times. Visual examination of the bearing and journal indicated that slight rubbing had occurred between the test surfaces.

The MF-F centrifugal pump has operated continuously for more than 18,100 hr circulating molten salt 30, $NaF-ZrF_4-UF_4$ (50-46-4 mole %), at 1200°F. The PK-P pump equipped with a bellows-mounted seal has operated continuously for more than 15,500 hr circulating NaK at 1200°F. (AEC Activity 4152.1)

Frozen-Lead-Seal Pumps. — A small frozen-lead-seal pump assembly consisting of a centrifugal pump mounted vertically over a fractional horsepower motor has operated continuously for 10,000 hr. The pump circulates molten salt isothermally at 1200°F. (AEC Activity 4152.1)

Remote Maintenance Demonstration Facility. — Construction was completed on July 1, 1959, as scheduled. Installation of remotely operated television equipment and remote tooling is in progress, as are the startup operations. (AEC Activity 4152.1)

Design Studies. — A design study was made to determine the feasibility and cost of installing an experimental reactor in the existing ARE building. It is estimated that a 5- to 10-Mw (thermal) reactor with the heat dumped to air could be installed for about $6,800,000. The design would be a modification of that previously reported for a 10-Mw (electrical) reactor. (AEC Activity 4152.1)

THORIUM BREEDER REACTOR STUDY

A comparative study of the capabilities of different reactor types for breeding in the $Th-U^{233}$ system has been initiated. The relationship between conversion ratio, specific power, and fuel cycle costs will be worked out for each type of reactor, using common ground rules and computational methods. Heavy-water-moderated reactors are being considered first, since their performance is not very sensitive to the values of η of U^{233} for epithermal neutrons. Current work at the Laboratory will produce better values of epithermal η, of great importance to graphite- and BeO-moderated reactors, before the study is completed. (AEC Activity 4152.1)

PROGRAM 5000 – PHYSICAL RESEARCH

Reactor Operations. – After being reworked by the manufacturer, the ORR coolers were tested and found to be deficient in cooling capacity by about the same amount as last year. Because of this deficiency, operation at 20 Mw is impossible on days when the temperature is above 83°F. It is planned to operate at a steady power of 16 Mw, rather than raise the power at night and reduce power in the day. A new cooling system is being designed to provide cooling capacity sufficient for 30-Mw operation.

The ORR hydraulic tubes have been placed in service for irradiation of samples.

The Graphite Reactor canal walls have been raised 18 in. This increase in depth of water decreased the general radiation background in the canal work area by a factor of 10.

The Graphite Reactor annealing was reviewed by the Advisory Committee on Reactor Safeguards on July 23. As soon as approval is obtained, the preparatory work can be started. Since this will require about three months, it will not be possible to anneal before the summer of 1960.

Laboratory Facilities. – The construction phase of the Segmenting Cell facility is complete, and the installation of equipment has started. Cold runs in preparation for the SRE fuel elements are planned to start during September. A portion of the facility will be ready for hot cell work about the middle of August.

The Liquid Hydrogen Dispensing Facility is expected to be ready for use during the month of August.

PHYSICS

Neutron Diffraction. – Single-crystal neutron diffraction experiments have been performed on anhydrous $CrCl_3$ at temperatures from 298 to 4.2°K, and the results show that this compound does not become ferromagnetic below 17°K as previously reported from magnetic susceptibility measurements (Conference on Magnetism, p 60, Am. Inst. Elec. Eng., Pittsburgh, 1955), but it becomes antiferromagnetic. The antiferromagnetic structure consists of ferromagnetic sheets of atomic magnetic moments within each hexagonal layer of metal ions, and the adjacent sheets are oriented antiparallel. The moment direction is perpendicular to the hexagonal c axis of the crystal. Observations on the antiferromagnetic reflections in an applied magnetic field show that the antiferromagnetism can be completely destroyed by fields of 5 kilooersteds applied parallel to the c axis and by smaller fields applied at an angle to this axis. Since these fields are much smaller than the exchange fields associated with the transition temperature, it is apparent that only the weak antiferromagnetic coupling between the layers is overcome by the fields and that this compound belongs to a special class of antiferromagnetic substances called "metamagnetics." The sheets of moments are merely rotated into the direction of the applied field, thereby giving large values of the magnetization, and it is this behavior which was probably misleading in the previous magnetic measurements. (AEC Activity 5220)

Fast-Chopper Time-of-Flight Spectrometer. — Measurements of the total neutron cross section were made on high-purity U^{233} metallic foils from 0.1 to 130 ev, with an energy resolution of 1%. Three different sample thicknesses were used: 4.72, 1.19, and 0.222 g/cm^2. These total cross sections are being compared with a computed curve based on the multi-level multichannel resonance scheme of Vogt. Direct measurements of the variation of η of U^{233} with neutron energy have been made from 0.04 to 10 ev with the use of Hornyak buttons to detect fission neutrons. The results are being analyzed and compared with the variation computed from total and fission cross sections. Additional measurements of the total cross sections of U^{233} and U^{235} in the thermal energy range with the high-intensity chopper have resulted in values of 586 ± 3 and 693 ± 5 barns for the 2200-m/sec total cross sections of U^{233} and U^{235}, respectively. (AEC Activity 5220)

High-Voltage Experimental Program. — Preliminary results of the reaction $C^{14}(p,n)N^{14}$, given in the April 1958 report, were obtained with a rather thick target of low C^{14} isotopic content. The recent availability of a thin target of much higher C^{14} content enabled this work to be extended. In addition to the levels previously reported, a narrow (about 6-kev) resonance at a proton energy of about 3.18 Mev has been found, and a second resonance near 3.38 Mev on the side of the large resonance at 3.42 Mev has been resolved. A careful search failed to locate a very narrow level which work on other reactions had indicated might occur at a bombarding proton energy of 3.14 Mev. The yield of neutrons in the vicinity of 2.9 Mev has been measured at three angles in the hope of determining whether this resonance is actually double, as evidence from other reactions indicates. The thresholds for neutron production leaving N^{14} in its first and second excited states have been observed. (AEC Activity 5220)

Thermal Diffusion. — Four 1-in. Calrod-heated columns, 15 ft long, are in operation concentrating the mass 36 isotope of argon. A continuous feed "infinite reservoir" brings normal argon into the bottom of the columns, and argon enriched in the 36 isotope is collected in 5-liter flasks at the top of each unit. This particular arrangement was set up to permit the computation of the thermal diffusion and remixing factors for the columns. At the present time, the collecting flasks contain about 16 liters of argon with a mass concentration of 23%. (Normal argon contains 0.34% mass 36.)

One 3/4-in. Calrod-heated column, 15 ft long, was in operation concentrating the mass 22 isotope of neon. Approximately 400 standard ml of product containing 56% Ne^{22} was collected. This column developed an internal electrical short circuit and had to be removed from service. However, it has been replaced with a 1-in. column, and the neon separation will be continued. (AEC Activity 5250)

Mass Spectrometry Research. — The Analytical Mass Spectrometry Group has five mass spectrometers in operation: two for gaseous samples and three for solid samples. Cylinders of argon, helium, and nitrogen to be used for special welding and for cover gases in experiments at elevated temperatures are certified routinely; most cylinders contain less than 10 ppm of oxygen. The separated stable isotopes of mercury, lutetium,

strontium, rubidium, and lead have been analyzed. A large number of helium and argon samples from tests of the compatibility of materials have been analyzed for the Gas-Cooled Reactor Project to determine the variation of the various contaminants with time and temperature. (AEC Activity 5250)

CHEMISTRY

Raw Materials Research and Development. — In studies on solvent extraction recovery of uranium from a carbonate leach liquor (from a western mill), transfer of organic matter from the liquor to the solvent phase caused almost quantitative precipitation of the extractant (Aliquat 336, a quaternary ammonium salt). The organic matter was, however, removed from the liquor by adsorption on activated carbon prior to the extraction cycle. In examination of the stripping cycle, more than 95% of the uranium was stripped from a pure uranium extract by a single contact with 0.5 M Na_2CO_3 — 1 M NaOH solution at an organic/aqueous ratio of 4/1. The presence of vanadium and molybdenum, particularly the latter, in the extract severely decreased the uranium stripping efficiency.

The flow capacity of settlers used for the stripping of uranium from amines by sodium carbonate is limited by entrainment of aqueous phase in the solvent, which increases with increased solvent flow until flooding occurs and the amount entrained is equal to the amount entering the settler. Nominal flow capacity based on solvent flow per unit settler area was selected as one-half of flooding velocity. For typical flowsheet conditions (0.1 M amine in kerosene vs 10% Na_2CO_3 stripping solution at an aqueous-to-solvent phase ratio of 1/6), the nominal solvent flow capacity was 1 $gal \cdot min^{-1} \cdot ft^{-2}$ for a secondary amine (Rohm and Haas Amberlite LA-1) and 4 $gal \cdot min^{-1} \cdot ft^{-2}$ for a tertiary amine (General Mills Alamine 336). The higher flow capacity for the latter amine is in agreement with general trends observed in other systems, where the rate of phase separation increased with interfacial tension. For the secondary and tertiary amines in contact with the sodium carbonate solution, the interfacial tensions were <1.0 and 6.0 dynes/cm, respectively. (AEC Activity 5310)

Solvent Extraction Research. — The scheme proposed for the recovery of uranium and plutonium from sulfuric acid solutions used for removing stainless steel cladding (ORNL-2756) was confirmed in continuous countercurrent extraction in a bank of six laboratory-scale mixer-settlers. The U(IV) and Pu(III) or Pu(IV) are extracted with 0.3 M primary amine. The recovery of both uranium and plutonium was greater than 99.5% from synthetic decladding solution containing 3 g of uranium and 0.0005 g of plutonium per liter, with a concentration factor in the extraction of more than 2. More than 99% of the extracted uranium and plutonium were stripped with 5 M HNO_3 in four stages of batchwise countercurrent stripping, giving over-all recovery of ~99% and a concentration factor of more than 20.

In continued study of the aggregation behavior of the amine extractants, light-scattering and viscosimetric measurements have been made on 1-undecyldodecylamine sulfate. This extractant is soluble in benzene and n-nonane only when relatively dry (0.5 mole of water or less per mole of sulfate). In this form the molecular weights in the two solvents were 140,000 and 800,000, respectively, corresponding to aggregation numbers of 180 and

1000; the particles were by far the largest yet observed in any of the organonitrogen extractant systems. The intrinsic viscosities show promise of providing qualitative relationships for estimating particle sizes from viscosity data alone.

Extractions of thorium sulfate from weakly acid (pH 2) aqueous sulfate solutions by di(tridecyl) P amine sulfate in benzene indicate, from the isotherm plateau, a complex containing three molecules of amine sulfate per atom of thorium, in close analogy with corresponding ratios for uranium loading by a number of the amine extractants. Di-n-decylamine and bis(1-isobutyl-3,5-dimethylhexyl)amine behaved similarly. (AEC Activity 5310)

Ion Exchange Technology. — The apparent uranium diffusion coefficient in 1200-μ beads of Dowex 21K anion exchange resin was approximately 1.3×10^{-7} cm^2/sec with a 0.0029 M uranium loading solution, the same as that with a 0.0058 M solution. However, with a 0.0058 M loading solution and 960-μ beads the apparent coefficient was 0.76×10^{-7} cm^2/sec. This discrepancy may be partly due to a difference in the physical or chemical properties of different size fractions of the resin or to experimental error.

The self-diffusion coefficient of sulfate ions in Dowex 21K was 1.2×10^{-6} cm^2/sec for 1200-μ beads. Beads labeled with S^{35} were placed in an Na_2SO_4 bath, and the bead activity was measured as a function of time. (AEC Activity 5310)

Inorganic and Physical Chemistry: Physical Chemistry of Ion Exchangers. — Measurements of the heats of exchange at 25°C for zinc with sodium ion in five differently cross-linked Dowex 50 cation exchangers were completed. Heat was absorbed in all cases; values of the integral heat ranged from ~650 cal per equivalent exchanged for a nominal 2% divinylbenzene (DVB) exchanger to ~2200 cal/equiv for a 24% DVB exchanger. The differential heat, $\Delta\bar{H}$, increased with the equivalent fraction of zinc ion, $x_{Zn^{++}}$, in the exchanger for the more highly cross-linked preparations; thus with the 24% DVB exchanger, $\Delta\bar{H}$ = 1500 cal/equiv for $x_{Zn^{++}} = 0.0$ and $\Delta\bar{H}$ = 3400 cal/equiv for $x_{Zn^{++}} = 1.0$. The endothermic nature of the exchange indicates that large positive entropy changes accompany the replacement of sodium by zinc ions. (AEC Activity 5330)

Inorganic and Physical Chemistry: Chemistry of Technetium. — The extraction of pentavalent and tetravalent technetium from aqueous acid solutions was measured for several insoluble organic liquids. Technetium(V) formed in 12 N HCl did not extract into diethyl ether; partial extraction was obtained with a 0.1 M solution of trioctylphosphine oxide (TOPO) in cyclohexane and with a solution of trioctylamine (TOA) in 1,2-dichloroethane. The thiocyanate complex of Tc(V) contained in 3 N HCl was extracted efficiently by highly dilute solutions of TOA in cyclohexane and in 1,2-dichloroethane. Extraction by 0.1 M TOPO in cyclohexane was also observed.

Technetium(IV) as $TcCl_6^{--}$ in 3 N HCl was poorly extracted by 1-pentanol; it was not extracted by diethyl ether or 2-ethyl-1-hexanol. Efficient extraction of Tc(IV) from 6 N HCl was effected by TOPO dissolved in cyclohexane and by TOA dissolved in 1,2-dichloroethane. (AEC Activity 5330)

Nuclear Methods of Chemical Analysis. — An investigation has been undertaken to determine the feasibility of using a low-intensity neutron source and gamma spectrometry for the rapid nondestructive analysis of ore samples for elements present in amounts of 1 mg or more. Radium-beryllium and americium-beryllium neutron sources and a multichannel gamma scintillation spectrometer are being used. Following irradiation in a laboratory analyzer (composed of paraffin, a neutron source well, and irradiation wells) for 1 min to 1 hr, the spectrometer is used to detect and measure specific gamma radioactivities being emitted in the decay of the radionuclide being determined. Such elements as Al, W, Ag, Mg, U, Na, Mn, Cu, Sc, Co, and Hf are now being determined in different types of ores by this method with a standard relative error of ±5% at the 95% confidence level.

A method for determining the specific surface areas of uranium oxide particles was investigated. The basis of the method is the heterogeneous exchange reaction between surface ions of a solid immersed in a solution and ions in the solution. If the solution is tagged with a radioactive isotopic tracer, the radioactivity of the solution will decrease with time because of a fairly rapid exchange of the tracer atoms with the atoms in the surface of the solid. The amount of radioactivity adsorbed in the initial surface exchange gives a measure of the surface area of the solid adsorbent. Uranium-237 (6.75d) was used as a tracer in these studies, and the surface areas of uranium oxide particles were determined as $(1.3 \pm 0.1) \times 10^3$ cm^2/g. This method is also applicable to other materials. (AEC Activity 5330)

Fundamental Study of High-Temperature Chemical Reactions: Vapor Pressure of Molten Fluorides. — Preliminary vapor pressure measurements on $LiF-UF_4$ melts containing 10 and 20 mole % LiF show that the heats of vaporization fall in the range 50 to 55 kcal/mole. The condensate from the vapor phase has been shown by x-ray examination to contain LiF. Condensate from the sample containing 10 mole % LiF was mostly UF_4, with a small quantity of $LiF \cdot 4UF_4$; condensate from the sample containing 20 mole % LiF was essentially all $LiF \cdot 4UF_4$. (AEC Activity 5330)

Fundamental Study of High-Temperature Chemical Reactions: Activities in Molten Nitrates. — The lattice model of the molten reciprocal salt system A^+, B^+, C^-, and D^- dilute in A^+ and C^- ionshas been utilized to calculate the activity coefficients of the components by two different approximations. The first is the asymmetric approximation, which includes the groupings $AC_n^{(1-n)+}$. This approximation has been discussed previously (ORNL-2636 and 2706). The second is the symmetric approximation, which includes the groupings $A_mC_n^{(m-n)+}$, where m and n are any integers. This approximation leads to the relation

$$\gamma_{AD} = \left(\frac{1-Y}{1-N_{C^-}}\right)^Z ,$$

where N_{C^-} is the anion fraction of C^- ion, and Y can be calculated from the distribution function for the symmetric approximation in terms of the coordination number, Z, the energy of formation of the ion pair A^+-C^-, ΔE, and the concentrations of the ions in solution, N_i.

The activity coefficients of $AgNO_3$, γ_{AgNO_3}, in the two molten solvents KNO_3 and $NaNO_3$ dilute in Ag^+ and Cl^- ions have been measured at 402°C in an emf cell. The values of γ_{AgNO_3} are all less than 1 and are much lower in the KNO_3 solvent than in the $NaNO_3$ solvent. For Z = 6, values of $\Delta E = 5.65$ in the KNO_3 solvent and of $\Delta E = 4.55$ in the $NaNO_3$ were obtained. The concentration dependence of γ_{AgNO_3} in $NaNO_3$ is closer to the calculations based on the symmetric approximation than to those based on the asymmetric approximation. The concentration dependence of γ_{AgNO_3} in KNO_3 is closer to the calculations based on the asymmetric approximations than to those based on the symmetric approximation.

The differences between the behavior in the two solvents can be correlated with the relative sizes of the K^+ and Na^+ ions and the relative effects of the fields of these two solvent cations on the energy of interaction of an Ag-Cl pair. (AEC Activity 5330)

High-Temperature Aqueous Solution Reactor Chemistry: Osmotic Coefficients. — Comparative studies of osmotic coefficients from 25 to 100°C indicate that the specific salt behavior as a function of temperature is consistent with those theories that explain aqueous solution behavior as a function of concentration at any fixed temperature. Since the energy differences (kT) in the former category as exemplified by osmotic coefficients are far smaller than those energies (the electrostatic energy of the salt) employed at any fixed temperature, it can be reasonably inferred that, up to 100°C at least, the present high-temperature isopiestic apparatus is producing information as accurate and precise as that found by conventional methods at 25°C. (AEC Activity 5330)

High-Temperature Aqueous Solution Reactor Chemistry: Solution Chemistry. — Calculations from the rate constants obtained for the homogeneously catalyzed reactions of hydrogen and deuterium in various light- and heavy-water systems (see p 32, ORNL-2756) show the energy of activation to be independent of the solute (hydrogen or deuterium). As the hydrogen (or deuterium) ion concentration is increased from 0.01 M to 1.0 M, the energy of activation decreases from 27.0 ± 0.5 kcal/mole to 24.3 ± 0.5 kcal/mole, but this change is independent of the solvent (D_2O or H_2O). (AEC Activity 5330)

High-Temperature Aqueous Solution Reactor Chemistry: Phase Studies. — The temperature of separation of a second liquid phase of a series of solutions, each 0.10 m in total SO_4^{--}, in which the UO_3/SO_3 molar ratio varied from 0.1 to 1.0, was found to increase from 309°C for 0.10 m UO_2SO_4 to 377.5°C for the solution 0.02 m in UO_2SO_4 and 0.08 m in H_2SO_4. The observed critical temperature for the latter solution was 379.5°C. No phase separation was observed in the solution 0.01 m in UO_2SO_4 and 0.09 m in H_2SO_4 up to the critical temperature of 380°C. This seems to indicate that with a high molar ratio of H_2SO_4 to UO_3 (9 to 1), UO_2SO_4 is soluble in the supercritical fluid.

Earlier investigations showed that $UO_2(NO_3)_2 \cdot H_2O$ solutions do not separate into two liquid layers at elevated temperature, although UO_2SO_4-H_2O solutions do exhibit this phenomenon. The effect of varying the proportion of NO_3^- and SO_4^{--} ions on the immiscibility temperature boundaries has been determined in 0.04, 0.08, and 0.16 m UO_2^{++} solutions. The initial substitution of NO_3^- for SO_4^{--} resulted in a considerable increase in the

two-liquid-phase temperatures, although at approximately 350°C the relative effect of apparent additional equivalencies of HNO_3 or H_2SO_4 was the same, probably due to actual loss of HNO_3 to the vapor phase in the sealed tube.

Investigation of the system UO_3-HNO_3-H_2O has been extended from 10^{-1} to 5×10^{-4} m in HNO_3 and from 200 to 300°C. At 10^{-3} m HNO_3 the saturation mole ratios, UO_3/HNO_3, are 0.41, 3.7, and 0.35 at 235, 270, and 300°C, respectively. (AEC Activity 5330)

METALLURGY AND SOLID STATE PHYSICS

High-Temperature Reactions. — From measurements on the absorption spectra of fused salts it was shown that the energy of the $^1A \rightarrow {}^1U$ type of $n \rightarrow \pi^*$ transition in NO_3^- ions increases linearly with the electrostatic potential (Ze/r) of neighboring cations. In the case of monovalent cations it was shown that the temperature coefficient of this energy shift increases progressively for a progressive decrease in the cationic radius. These phenomena are in agreement with the McConnel theory of the effect of solvents on $n \rightarrow \pi^*$ transitions when this theory is modified to take account of the randomizing effect of thermal motions. These results are a first check of the McConnel theory for the case of electrostatic perturbations and may be used to give a qualitative explanation of the complex effects which various aqueous and organic media have on the $n \rightarrow \pi^*$ transition of dissolved NO_3^-. (AEC Activity 5420)

Metal Surface Studies. — Previously reported x-ray measurements on very thin Cu_2O films have indicated the existence of large numbers of cation vacancies within the films. In order to check this result, a method for determining the copper content of Cu_2O was devised. The results of the analysis of a film 650 Å thick, as measured with a polarizing spectrometer, indicated that each unit cell of the oxide contained three copper atoms instead of the normal four. This finding is consistent with the previous x-ray data and indicates the presence of a much larger concentration of lattice defects in thin Cu_2O films than had previously been supposed. (AEC Activity 5420)

THERMONUCLEAR PROJECT

DCX-1 Facility. — The main activities on the DCX-1 during the past month were directed toward the establishment of a more accurate accounting for the particles known to be injected into the plasma in terms of those seen to emerge under equilibrium conditions. The discovery of a loss mechanism by charge exchange in the carbon arc has improved the situation somewhat, but measurements do not yet cover the complete surface of the volume occupied by trapped ions, and the accountability is still incomplete. (Last month's statement that all particles are accounted for did not hold after further measurements were obtained.) At present the situation is that at 10^{-6} mm Hg about 20% of the input rate has been accounted for in terms of emerging ions. The axial survey can now be done with good resolution, but the azimuthal survey of surface loss is still experimentally incomplete, and the mirror losses from the ends have not been measured. The decay time measurements with and without arc imply that at 10^{-6} mm the arc loss and the loss due to residual gas are about equal; if this is so, then the arc losses have not yet been fully assessed azimuthally, or endwise. (AEC Activity 5540)

Ion Source Development. — The von Ardenne source has been made to yield a total ion current of 93 ma on a 3-in.-dia catcher that was 8 in. away. The increase over the previous 50 ma was the result of the application of a larger power supply. The gas efficiency under these conditions was 71%, with H_2^+ ions assumed to predominate. (AEC Activity 5540)

PROGRAM 6000 — BIOLOGY AND MEDICINE

BIOLOGY

Genetic and Cytogenetic Effects of Radiation. — The mode of action of ethyl methanesulfonate in the induction of mutation in bacteriophage T4 has been investigated. Previous experiments have demonstrated the induction of r mutants among r^+ phage treated with this mutagen; induction of r^+ reversions from some r mutants treated with ethyl methanesulfonate has now been shown.

Previous observations of the induction of mutants by ethyl methanesulfonate led to two distinct hypotheses regarding its mode of action: (1) the mutagen could act only on the phage-bacterium complex and was nonspecifically associated with the phage and injected into the bacterium upon infection by the carrying phage; (2) extracellular treatment of the phage resulted in a specific association of the phage and the mutagen which eventually led to the formation of mutant clones by the treated phage. It has been possible to show that nonrevertible r phage treated with the mutagen extracellularly and simultaneously adsorbed onto bacteria with revertible untreated r phage are not capable of mediating the reversion of the mutable r phage. These results are inconsistent with the first hypothesis and suggest the second, namely, that the mutagenic action of the ethyl methanesulfonate is available only to phage that have been directly exposed to the mutagen. (AEC Activity 6130)

Insect Cytology and Genetics. — Mature eggs or sperm are employed in most studies of radiation-induced dominant lethality. It has recently been shown that dominant lethal mutations can be induced by x rays in oogonial cells of the female wasp Habrobracon. These dominant lethals do not appear to be caused by radiation-induced chromosome breakage or inhibition of nuclear division, since in appearance the developing embryo dies at a time different from that when chromosome imbalance or mitotic inhibition kills the embryo. The condition and time of death, however, are almost exactly mimicked by ultraviolet irradiation of the cytoplasm of mature eggs. It is suggested therefore that x irradiation of oogonia affects the ability of a gonial cell, while growing into a large egg, to produce the proper cytoplasm necessary for normal embryonic development. (AEC Activity 6130)

Pathology and Physiology. — Serological markers and differences in the electrophoretic mobility of hemoglobin have been used to identify erythrocytes after bone marrow transplantation in lethally irradiated mice. Genetically differing red cells that cannot be distinguished by these techniques have been identified by differences in hemoglobin solubility and crystallization, as follows. The optical densities of filtrates of carbon monoxyhemoglobin, salted out in 3.08 M phosphate

buffer at pH 6.5 and read at 575 mμ, were found to be significantly different for red cells of the 101, C57BL, and (C57BL × 101)F_1 strains, being 0.16, 0.02, and 0.36, respectively. Furthermore, on salting out, the different hemoglobins differed in the type of precipitate formed: the 101 formed an amorphous precipitate, the C57BL formed aggregated hexagonal crystals, and the (C57BL × 101)F_1 formed mixtures of the two types. Hemoglobins from mice of other strains, with electrophoretic mobilities corresponding to those of the foregoing prototypes, also showed corresponding solubilities and crystallizing properties: the C3H, RF, and DBA/2 resembled the 101; the C57L and the 11G/R1 resembled the C57BL; and the (C57BL × DBA/2)F_1 and (C57L × A)F_1 resembled the (C57BL × 101)F_1. In comparative analyses of a group of radiation chimeras with the use of both starch gel electrophoresis and hemoglobin solubility, the two techniques gave comparable results. Consequently, differences in hemoglobin solubility may be used to identify the erythrocytes in irradiated mouse chimeras if the hemoglobins of the donor and recipient can be thus distinguished from one another to begin with. (AEC Activity 6130)

Biophysics. — Measurements of electron spin resonances induced in multicrystalline glutamic acid in air at room temperatures by doses up to about 10^8 rads of Co^{60} gamma rays indicate a saturation effect of stable resonance when the number of resonances is roughly 2/1000 the number of molecules. This saturation level seems too high to represent a structural saturation of the surfaces of crystals and too low to represent a true molecular saturation. If the effect should represent a saturation of an impurity, the production of stable resonances in the impurity would have to be much more frequent than the production of ion pairs. For x-ray-irradiated DL-valine a value of 85 ev per resonance induced has been found. Signals from irradiated L(-)-cystine have also been observed, but none were observed for DL-isoleucine.

The presence of naturally occurring free radicals in a riboflavin-tryptophan complex is under investigation. It has been found that there is one unpaired spin for every 200,000 molecules of complex. This might be due to an impurity. No signal is detected from pure riboflavin or pure tryptophan, although riboflavin recrystallized from distilled water gives a signal. Experiments to determine whether oxygen or light is involved in this phenomenon are in progress. (AEC Activity 6130)

Mammalian Radiation Recovery. — Autoradiography with tritiated thymidine has indicated that intestinal crypt cell enlargement is not a result of polyploidy or polyteney. After 5000 r, neither total grain count nor the labeling rate between control and enlarged cells is different. The loss of epithelial cells by death and migration is evidently compensated for by a nearly equal displacement of existing villus stroma into the crypt region.

The rate at which Cr^{51}-labeled mouse erythrocytes disappear after intravenous injection into homologous mouse strains may be comparable to the rate for the isologous situation or much higher. Homologous erythrocyte survival in the normal mouse is strain-dependent.

Several clonal lines of tissue-cultured cells have been isolated which yield high plating efficiencies. These lines are being used for

quantitative measurements of the effect of cell number in the initial inoculum on the shape of x-ray survival curves. In the range from 0 to 800 r, a hypotetraploid line derived from normal human heart so far shows a logarithmic survival curve, with a mean lethal dose near 100 r and a very low "hit number" when plated in constant low inocula for each dose.

In experiments on bone marrow preservation by slow freezing and subsequent storage at -196°C, many organic compounds have been found that protect bone marrow cells as well as the usual glycerol (15%). The maximum protective action is given at these concentrations: glycerol, 5%; mannitol, 0.5 and 0.7 M; sorbitol, 0.2, 0.4, and 0.8 M, and 0.2, 0.4, and 0.8 M in 3.5% polyvinylpyrrolidone; inositol, 0.1 M; choline, 0.05 and 0.17 M in 3.5% polyvinylpyrrolidone; and sucrose, 0.2 M.

Bone marrow cells and hemic cells from fetal liver show evidence of limited multiplication and maturation when cultured in spinner cells in a medium containing mixture 199, horse serum, and ultrafiltrate of beef embryo extract.

Bone marrow cells irradiated in vitro are protected if they are exposed in the frozen state (-196°C). (AEC Activity 6230)

PROGRAM 8000 – WORK FOR OTHERS

International Reactor Courses. – Twenty-six students currently enrolled in the foreign programs have completed 24 weeks of school since the beginning of classes in February. The ten Operations Supervision students complete their classwork at ORSORT in August and begin full-time participation in the Laboratory's reactor operations for three months. The 16 students enrolled in Hazards Evaluation will continue classes until the beginning of problems in November.

The AEC Division of International Affairs has received 22 applications from 11 countries for the session starting in November. A review of applications is now in progress. (AEC Activity 8717)

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STATUS AND PROGRESS REPORT

July 1959

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