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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JANUARY 1959

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

January 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 4000 — REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. — The experimental run (No. 18) which started in December and has as its purpose the investigation of the relationship between reactor power and fuel stability continued through January. The normal operating temperature and pressure were 260°C and 1500 psig. The inventory of circulating uranium was determined at reactor powers of approximately 100, 400, and 750 kw. There was no decrease in inventory with increasing power nor any other indication of power-dependent fuel instability over this range of power. During the coming month, the reactor power will be raised in increments of 300 to 500 kw until instabilities appear. (AEC Activity 4103.3)

HRT Chemical Pilot Plant. — The corrosion products (273 g) separated by the hydroclone in run 18-7 contained 22% Fe, 45% Zr, 4% Cr, 0.5% Ni, and 8% U.

The rate of separation of solids continued at just under 2 g/hr in run 18-8. In 330 hr on stream, 613 g of corrosion products containing 4.4% U, 1.5% Cu, 24% Fe, 9% Cr, 38% Zr, and 0.1% Ni were separated. (AEC Activity 4103.1)

Fuel Processing. — A second liquid or solid phase formed on Zircaloy-2 pins which were immersed in a simulated HRT solution, at 320°C, in sealed quartz tubes and which were heated by induction until boiling occurred at the surface.

Preliminary results indicate that treatment of simulated HRT fuel solutions with ozone may make possible the use of a stainless steel electrolytic cell containing a mercury pool cathode for removal of manganese from the solution. (AEC Activity 4103.1)

Gaseous Fission Product Disposal. — The effect of linear velocity of sweep gas on holdup time of gaseous fission products and on the number of theoretical plates in a bed has been studied for Columbia G charcoal in pipes with an inside diameter of 0.80 in., with Kr^{85} as a tracer and oxygen as the sweep gas. Preliminary values of the number of theoretical plates per foot of trap length decreased from 34 to 0.7 as the mean value of the superficial linear velocity increased from 0.5 to 87 fpm. (AEC Activity 4103.1)

Thorium Oxide Slurry Development. — Thorium-uranium oxides with average particle sizes around 2.5 μ, some spherical in shape, were prepared by flame denitration of alcohol solutions of the nitrates.

Boric acid is being considered as a soluble poison for criticality control in thorium oxide slurries. Under expected reactor conditions in a slurry of 1100°C-fired thorium-uranium oxide, with a B/(Th + U) ratio of 0.02, much less boron was adsorbed on the solid than the 0.029 mg/g estimated to result in a 1% neutron loss. Slurries of thorium-uranium oxide containing chromic acid and autoclaved at 300°C showed a soluble U/soluble Cr ratio of 2.6 over the Cr concentration range 125 to 1330 ppm.

Small radiolytic-gas pressures (<200 psi) were generated in slurries (500 and 250 g of Th per kg of D_2O) of 650°C-fired thorium oxide containing 0.5% U^{235}, with 145 ppm Pd as recombination catalyst, under irradiation in an O_2 atomosphere at 300°C in the LITR. Preliminary results of a study of the effect of pumping time and gas atmosphere on the catalytic activity of slurries of low-fired thorium-uranium oxide containing Pd catalyst indicated decreased activity with pumping time under an argon atmosphere but recombination rates still high enough for reactor use ($\sim$2 moles of O_2 recombined per liter per hour at P_{D_2} = 100 psi). Preliminary gas recombination experiments with a slurry of 650°C-fired thorium oxide containing 300 ppm Ru gave promising results. (AEC Activity 4103.1)

Equipment Decontamination. — Several reagents, in addition to 0.5 M $CrSO_4$—0.5 M H_2SO_4, have been found that will dissolve scale deposited on type 347 stainless steel by uranyl sulfate solutions at 250 to 300°C. Scale was completely dissolved by Deoxidine-170 (inhibited phosphoric acid with added detergent) at 85 to 123°C in 9 1/2 hr or by pyrophosphoric acid at 150°C in 1 hr. Corrosion rates during descaling were 1 to 1.5 mils per month in Deoxidine and approximately 6 mils per month in pyrophosphoric acid. Synthetic HRT fuel (0.025 M uranium) circulated through a zinc bed and over a piece of HRT pipe at 75 to 95°C completely removed the corrosion scale in 9 1/2 hr. (AEC Activity 4103.1)

HRP-PAR In-Pile Slurry Loop. — Work has been discontinued on the HRP-PAR in-pile loop pending resolution of the disposition of the PAR portion of the equipment. (AEC Activity 4103.1)

Homogeneous Reactor Instrumentation. — Ceramic-to-metal seals for mineral-insulated cable have been developed which are sturdier and more easily silver-soldered than those used on the magnesium oxide—insulated copper-sheathed cables installed in the HRT.

The differential transformers being developed for use as sensors of instrument primary-element motion at high temperature and in radiation fields were redesigned to reduce the effects of changes in insulation resistance of the winding form and the potting.

A prototype Y-body-design valve with a bellows stem seal was fabricated for use in 500-psi slurry service.

Zircaloy-2 is proving to be the most satisfactory valve trim tested to date for slurry service applications. (AEC Activity 4103.1)

HRP Metallurgy. — Analyses of Zircaloy-2 exposed in the HRT blanket region during run 17 and of Zircaloy-2 screen samples removed from the core after run 17 indicate that no appreciable quantities of hydrogen or deuterium are being picked up by Zircaloy-2 during reactor operation. This conclusion was confirmed by a gas analysis for combined hydrogen and deuterium and for deuterium. Microexamination of the limited number of Zircaloy-2 screen samples and mechanical-property specimens now available indicates that the only structural changes in Zircaloy-2 exposed in the core took place in areas in close proximity to the titanium holder and were undoubtedly caused by the high temperatures of the holder. In these areas a wide range of elevated-temperature structures was found. (AEC Activity 4103.1)

Dynamic Solution Loop Corrosion Tests. — Deposition of uranium, copper, and nickel, presumably as sulfate salts, occurred when solutions originally containing 0.025 m UO_2SO_4, 0.006 to 0.025 m D_2SO_4, 0.01 m $CuSO_4$, and 0.004 m $NiSO_4$ in D_2O were circulated at low flow rates through a horizontal section of a loop heated to cause the solution to boil. Preliminary results indicated that increasing the acid concentration tended to suppress the salt deposition, although it could still be made to occur by the application of higher heat fluxes. Deposition occurred during some tests at temperatures well below the second-liquid-phase temperature of the solution. The deposited material could be redissolved within a few minutes by turning off the heat or increasing the system pressure to prevent boiling, or by increasing the solution flow rate. Deposition did not occur in a heated section of loop mounted vertically. The vertical orientation apparently allowed the heated surface to be wet continuously. Therefore it appeared that deposition occurred by crystallization of salts from droplets of the boiling solution which splashed onto a hot, dry metal surface in the vapor phase above the solution.

This method of losing uranium from solution is one of three possible methods by which uranium and copper could be lost from the main circulating stream in the HRT, namely, crystallization of a salt as above, formation of hydrolysis products, or formation of a second liquid phase. If the components of the fuel are lost by the first means, simply increasing the pressure of the system or increasing the acid concentration should be beneficial. (AEC Activity 4103.1)

Small-Scale Dynamic Slurry Corrosion Tests. — The inclusion of substantial proportions of uranium oxide in thoria reduced appreciably the corrosion of various metals under oxygen and deuterium atmospheres in 300-hr toroid tests at 280°C and 26 fps with 500 g of Th-U per kg of D_2O and with and without 0.008 m MoO_3. The normalized reference corrosion rate of type 347 stainless steel pins by thoria containing no urania was 4 mils/year (mpy) in an oxygen atmosphere. The corrosion rates of Ti-75A and Zircaloy-2 were 2 and 0.6 mpy, respectively. In a deuterium atmosphere the corrosion of these materials increased by a factor of 2. The effect of MoO_3 was insignificant. The corrosion of these metals was reduced by increasing the urania content of the slurry.

The corrosion rate of Inconel in thoria slurries containing no urania was 12 mpy in an oxygen atmosphere and 5 mpy in a deuterium atmosphere. There was no particular effect of uranium content on the corrosion of Inconel under an oxygen atmosphere; however, in a deuterium atmosphere the effect of increasing the uranium content roughly paralleled the effect given for the metals above. The attack of Inconel under an oxygen atmosphere appears to be predominantly water corrosion rather than erosion. (AEC Activity 4103.1)

Dynamic Slurry Loop Corrosion Tests. — An argon-deuterium atmosphere was maintained in the first phase of a prolonged 100A loop test being made to evaluate a slurry of 0.8-μ, 650°C-calcined ThO_2 containing 0.5% U (added as $UO_3 \cdot H_2O$ rods) and 140 ppm palladium-metal catalyst. The slurry, which had been proposed for use in the PAR in-pile slurry loop program, was circulated for 508 hr at 280°C at an average concentration of 427 g of Th per kg of D_2O. The mean attack rate of the austenitic stainless steel components of the system was 1 mil/year (mpy).

The yield stress of the slurry did not change during circulation, and no thoria particle degradation was noted. During dropout tests, 15% of the slurry solids were lost from the flowing stream at a pipeline flow velocity of 2.5 fps; however, at 4.6 fps the solids were readily resuspended and the slurry circulated with no evidence of segregation. A small amount of the palladium catalyst was deposited on gold corrosion specimens exposed in the loop during the test.

Attack rates computed from pin weight losses at 20 to 40 fps, respectively, were (in mpy): type 347 stainless steel, 0.8 and 4; Inconel, 0 and 0.6; titanium, 0.3 and 0.3; Zircaloy-2, 0.4 and 2.5; Croloy 5, 6 and 47; and Incoloy, 0.2 and 2. The circulating-pump scroll and thermal barrier, fabricated of titanium, were not detectably attacked. (AEC Activity 4103.1)

In-Pile Solution Corrosion Loops: LITR. — Loop experiment L-2-22 was operated satisfactorily in the LITR for approximately 2630 hr. Three charges of solution, each with D_2O as solvent, were used for three separate periods of operation in which a main-stream temperature of 280°C was maintained. The first and third solutions contained 0.04 M UO_2SO_4, 0.2 M Li_2SO_4, 0.0015 to 0.0025 M $CuSO_4$, and 0.1 M H_2SO_4; the second contained 0.04 M UO_2SO_4, 0.0067 M $CuSO_4$, and 0.018 M H_2SO_4.

An over-all corrosion rate of 0.56 mil/year, based on oxygen consumption, was obtained for ~1950 hr, the first period of operation, which was conducted with the pressurizer temperature at 295°C.

Eleven "boiling" experiments were performed during operation with the second and third charges. Solution "boiling" and solids deposition were not produced under the normal flow rate of ~0.8 fps. However, they were easily produced with the second solution, at a flow rate of ~0.3 fps, when the pressurizer temperature was lowered from 295°C to a value at which the pressure was less than the estimated total pressure (steam and oxygen and radiolytic gas) at the surface of the core wall. The "boiling" was accompanied by an increase of fission power in the loop core. Raising the tem-

perature back to its previous value failed to arrest these power excursions, but a decrease in reactor power succeeded. A power increase of more than 500 w was observed during some excursions before corrective measures were employed. With the third solution, power excursions were not achieved until the total pressure on the system was about 80 psi less than that at which "boiling" occurred with the second charge. It is probable that "bulk boiling" was taking place in the core at this lower pressure. The fission power increase appeared to be at a lower rate than with the second solution. An increase in flow rate to $\sim$0.5 fps was tried with the third charge and found to be effective in arresting the power excursions.

The increase in the core nose temperature paralleled the fission power increase in the core in practically every experiment. Analytical data from solution samples taken during the power excursions indicate loss of solutes, but are inconclusive as to whether the losses were hydrolytic in nature. (AEC Activity 4103.1)

In-Pile Autoclave Slurry Corrosion Studies. — A Zircaloy-2 autoclave slurry corrosion experiment was completed in the HB-6 facility of the LITR. The autoclave was loaded with a slurry of thoria-urania (5 wt % enriched uranium based on thorium) containing 400 g of Th per kg of D_2O and 0.01 m MoO_3. An excess of oxygen was maintained. The experiment operated at 280°C out-of-pile for 174 hr and in-pile at an average fission power density of 0.5 w/ml for 948 hr. The maximum power density achieved was 1.9 w/ml. Initially the catalytic activity was only 0.034 mole per liter per hour at 100 psi D_2, but after 110 hr it had increased to 0.26 mole per liter per hour at 100 psi D_2; G_{D_2} values between 0.74 and 0.92 were estimated. During later operations the G_{D_2} values declined, then increased. The corrosion rate for the first week in-pile was 1.7 mils/year, at an average fission power density of 0.4 w/ml. The over-all corrosion rate was 0.7 mil/year. (AEC Activity 4103.1)

Supporting Radiation Corrosion Studies. — An electrochemical method has been developed to study the out-of-pile corrosion behavior of zirconium alloys in high-temperature aqueous environments. This method has been used successfully by other investigators at temperatures below 100°C.

Experiments were performed to study the rate-time corrosion behavior of Zircaloy-2 in 0.05 m H_2SO_4 containing 600 ppm O_2 at 208°C. A determination has also been made of the activation energy for the corrosion process between 248 and 207°C in the above solution on specimens which were previously exposed for 50 and 100 hr at 208°C. The measured value is 15 to 16 kcal/mole. The rate-time behavior follows the expected pattern, with rates which are initially high and which drop off with exposure time. The indicated initial rates are in the range 25 to 50 mils/year at 207°C. After an exposure period of six days, the longest period investigated, the measured rates are about 0.1 mil/year. The Tafel slope for O_2 reduction on platinum in 0.05 m H_2SO_4 at 208°C is 0.043. (AEC Activity 4103.1)

CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR

Design Review of Philadelphia Electric Proposal. — At the request of the Oak Ridge Operations Office a design review was conducted of the Philadelphia Electric proposal for a high-temperature gas-cooled reactor. A report has been issued on this work. (AEC Activity 4110)

Modified PEGCPR Cost Estimate. — A survey was made of the potential capital cost reductions which might be realized in the Kaiser-ACF prototype reactor (PEGCPR). The survey is incomplete because of the unavailability of complete cost data on the Kaiser design. The survey indicates that elimination of the bottom fuel charge-discharge machine, modification to 850°F—850 psi steam conditions, and elimination of some features included in the Kaiser building layout would permit a reduction in direct costs of the plant of almost $4 million. If the plant were built in Oak Ridge the estimated cost would be $26.5 million, as compared with $35.3 million for the Kaiser design built at Idaho. Both estimates must be regarded as preliminary at this time. (AEC Activity 4110)

ORR Loop Design. — The loop as presently conceived will be of concentric pipe construction; probably a 1-in.-dia center tube will supply cold gas to the test section, with a 1/4-in. annulus for the return flow. The test section will also be of concentric design to fit a single lattice position (B-1). Many of the auxiliaries will be located in the ORR pool. The auxiliary package as now conceived is very compact. It will be possible to vary the coolant flow over the range 0 to 500 lb/hr of helium in order to obtain surface temperatures up to 1400°F for fission power within the test section of up to 20 kw. The system is designed for operation at 400-psi helium pressure. (AEC Activity 4110)

LITR Experiments. — Fuel capsule experiments have now been installed in all six LITR-GCR facilities. All six experiments are operating satisfactorily. Central UO_2 pellet temperatures are steady and in the range from 1515 to 2078°F. In order to raise the central temperatures, future capsules will utilize higher fuel enrichments and/or increased gas gaps between the pellets and the capsule walls. (AEC Activity 4110)

ORR Experiments. — Installation work continues on the out-of-pile facility for these experiments. Instrumentation and gas control panels have been installed, and connections are in final stages of assembly. The gas supply lines have been installed and tested; assembly of the shielded valve box containing valves and tubing is more than 50% complete. The gas lines from the valve box into the pool for connections to the experiments have been fabricated, tested, and installed in the pool. Fabrication of the shutter mechanism and experiment mounting pieces has been completed. (AEC Activity 4110)

GETR Experiments. — All the major pieces of equipment, except for the three dump tanks and the in-pile facility tubes, have been installed at the G-E test reactor (GETR). The valve room and the shielding wall are now being built. The facility tubes have been fabricated and have passed an

x-ray examination. The dummy assembly for flux depression measurements is being fabricated. (AEC Activity 4110)

Radiation Effects in Structural Steel. — The first series of hardness tests on step-annealed irradiated SA-212B steel have been completed. These tests showed hardening of the irradiated specimens in the temperature range from about 400 to 500°F, followed by softening. A temperature of approximately 900°F was required to bring the hardness down to that of the unirradiated steel. The corresponding temperatures for cold-worked unirradiated material were about 600 and 1200°F. (AEC Activity 4110)

Mechanical Properties of Type 304 Stainless Steel. — Additional data which have been obtained from tube-burst tests of type 304 stainless steel are summarized below:

Temperature (°F)	External Pressure (psig)	Tangential Pressure (psi)	Rupture Life (hr)	Average Strain (%)	Max. Strain (%)
1300	600	10,650	920	1.6	2.2
1500	700	12,425	1.5		
1500	200	3,550	1010	3.1	4.2

(AEC Activity 4110)

Fuel Pellet Fabrication. — Prototype-size fuel pellets of high quality are now being produced as needed for the capsule irradiation program. Solid and cored pellets for eight ETR capsules and four ORR experiments have been qualified by physical property measurements and delivered for assembly. Additional pellets for approximately six more capsules are now being checked, including four sets for ORR experiments which were sintered to finished size.

Beryllium oxide rods of low density have been fabricated and delivered for use as core rods in six of the ETR and ORR capsule experiments. These rods are solid but were fired to a low density to provide a maximum of void volume. The average density of the first two rods used was 49% of theoretical.

Miniature pellets of 20%-enriched, 95%-dense UO_2 were delivered for use in two experiments in the LITR. (AEC Activity 4110)

Low-Pressure Thermal-Convection Loops. — Loop No. 6, which was terminated after 900 hr at 1400°F, was disassembled and examined visually. The cold leg of the loop showed deposits similar to those found in test No. 1. The metal test specimens showed some change in appearance, varying from a fairly bright crystalline surface on the low-alloy steels to a dark uniform surface on the stainless steels. A new cold leg has been cut, and the test facility is being reassembled for the next test, which is to be conducted with AGOT graphite at 1400°F.

Test No. 7, containing type AGOT graphite at 1100°F, has now completed 240 hr of the 900-hr test phase. (AEC Activity 4110)

GENERAL REACTOR RESEARCH

Reactor Evaluation Studies. — The group cross sections of the plutonium isotopes have been obtained for 34 specific energy groups (from 10^7 ev to thermal energy). These will be used in a determination of the effective resonance integrals as a function of plutonium concentration; these latter integrals are needed in the fuel-value evaluation of "recycle" plutonium. (AEC Activity 4119)

Radiation Detector Development. — All the components of the BSF model IV gamma-ray spectrometer, with the exception of the housing for the crystal, have been installed at the BSF. The lead-lithium housing has been poured and is currently at the ORGDP machine shops for final machining. The results of the latest investigations of the big NaI (Tl) crystal which will be used as a detector in the spectrometer have indicated that the observed double-peak pulse-height distribution may be due to the optics of the system. Plans are being made to rework the surface of the crystal and change the reflector in an attempt to improve the response to gamma-ray energy. (AEC Activity 4202)

Liquid Metals Research. — The solubility of nickel in liquid mercury was determined; it varies from 0.1 ppm at 129°C to 35.1 ppm at 278°C. The heat of solution of nickel in liquid mercury calculated from these data is ~11 kcal/mole.

Samples of mercury that had been in contact with nickel in a thermal-convection loop were found to have nickel compositions as high as four times that corresponding to saturation at the highest temperature in the system. This result can only be accounted for by assuming that particulate nickel is being circulated around the loop. Calculations based on Greenwood's (Acta Met. 4, 243–48, 1956) growth-rate equations for fine crystal suspensions indicate that particles of mean diameter $<50\,\mu$ may be stable in such a system.

The diffusion coefficient of silver in a liquid lead–1% silver solution was measured between 375 and 650°C and found to increase with temperature from 3.5×10^{-5} to 1.0×10^{-4} cm^2/sec over this temperature range. (AEC Activity 4203)

Reactor Safety Studies. — In the Pool Critical Assembly experiments for the study of the effect of voids in reflectors, the change in reactivity ($\Delta C/C$) caused by a void covering the entire surface of one face of the reactor was found to be 3.49%. In arriving at this number the effect of the aluminum wall of the void tank was considered, and the value of γ, which accounts for the relative effectiveness of delayed neutrons, was assumed to be 1.25. The experimental value is to be compared with a calculated value of 3.62%; however, the experimental value has an uncertainty of $\pm 3\%$ based on a detailed error analysis plus an uncertainty of several per cent associated with the method of reactivity calibration.

The grid plate and fuel elements for the BSF stainless steel—UO_2 reactor (BSR-II) are expected to be completed in February and April, respectively. Detail design of the control system is under way, and control system fabrication, which began in mid-January, is proceeding as the design permits. Tests with a prototype control-plate drive and dummy element to determine life cycles and plate insertion rates are scheduled for February. It is expected that the initial low-power critical experiments will be carried out at the BSF in May, and that the capabilities of the control system will be tested at the NRTS SPERT-I facility immediately thereafter. Calculations have definitely established the necessity of using "double-ended" fuel insertion devices in the SPERT-I tests. The present proposal is to use a device which consists of two three-plate blades which will be inserted in a special element placed near the center of the core. Each blade will have a 7-in.-long lower poison section, a 1-in.-long transition section, and a 15-in.-long upper fuel section. The difference in the extreme positions of the device is calculated to be 6.49% $\delta k/k$. The worth of the control plates in the core is calculated to be 14.13% $\delta k/k$. Since the devices will be inserted with modified control-plate drives, the results of the control-plate life tests will be used to determine the final design changes for the SPERT-I tests. (AEC Activity 4252)

Power Reactor Fuel Reprocessing: Darex Process. — Three full-size unirradiated sintered UO_2—stainless steel (APPR) elements dissolved in 2 M HCl—5 M HNO_3 in 1 hr except for the braze metal. The average rate was 28 g/min at a dissolvent rate of 4.3 liters/min.

In further batch chloride-removal tests using continuous 60% HNO_3 addition, the product contained 130 ppm to 0.07 M chloride. Chloride was removed to 220 and 50 ppm with the standard batch recycle and a new double recycle flowsheet, respectively. The use of 90 to 92.5% HNO_3 eliminated the waste acid cut; chloride was removed to 35 ppm. Updraft and downdraft condensers, with air sparging, were equally effective in mixed HCl-HNO_3 recovery, but air sparging was detrimental to chloride recovery.

In laboratory studies the silica was filtered from a 325-liter batch of chloride-free Darex dissolver solution on a 1-ft^2 sand bed in less than 1 hr by a split feed procedure, in which the clear supernatant from a sedimentation step was filtered first, and then the thickened slurry. Filtration of the same material as a well-mixed slurry required ~3 hr. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Hermex Process. — Reduction of 1700°C-fired UO_2 powder with magnesium in the presence of anhydrous magnesium chloride approximated 100%. About 93% of the uranium was recovered, as the mercuride, from the mixture of metal and slag by leaching with boiling mercury. The remaining 7% was contained in a hard crust above the mercury solution, where dissolution and extraction by the mercury were hindered.

Extraction of uranium from an alloy of approximately 4000 g of U per ton of Th was very incomplete. The UHg_4 coprecipitated with the $ThHg_3$, and the alloy on the bottom of the dissolver became covered by the mercuride that formed, preventing further mercury attack. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Mechanical Processing. — Stainless steel—UO_2 core fuel tubes in a typical fuel element array were chopped into short sections satisfactorily. Rapid cutting and flattening of the bundle decreased the degree of closure on the fuel tubes. Leaching rates of UO_2 varied from 3 to 272 $mg \cdot cm^{-2} \cdot min^{-1}$ in leaching solutions varying in composition from 3 M HNO_3 containing no uranyl nitrate to 13 M HNO_3 containing 2 M uranyl nitrate. (AEC Activity 4301)

Waste Treatment. — The temperature rise in cylinders of radioactive solid waste stored in an infinite salt medium was calculated as a function of time. The maximum rise was from 700 to 1900°F for one-year-decayed waste stored in a 9-in.-radius cylinder, 400 to 1200°F for three-year-decayed waste in a 14-in.-radius cylinder, and 400 to 1400°F for eight-year-decayed waste in a 29-in.-radius cylinder. Thermal conductivities ranging from 1.0 to 0.1 $Btu \cdot hr^{-1} \cdot ft^{-2} \cdot (^{o}F)^{-1} \cdot ft$ and convection coefficients between 0.7 and 1.3 $Btu \cdot hr^{-1} \cdot ft^{-2} \cdot (^{o}F)^{-1}$ were assumed. The waste was assumed to have resulted from the Darex process and to have heat generation rates of 100, 30, and 10 $Btu \cdot hr^{-1} \cdot gal^{-1}$ at decay times of 1, 3, and 8 years, respectively. (AEC Activity 4352)

Long-Range Planning Studies. — Samples of U^{233} irradiated in the LITR for seven months have been removed from the reactor and cooled; the fission product radioactivity has been removed from these samples by use of ion exchange methods. These samples will be used to obtain the effective value of α^{23} in the LITR neutron spectrum.

Work has been initiated concerning the factors which determine the maximum thermal flux obtainable in high-flux reactors having a total power less than 100 Mw; these factors include heat transfer limitations, geometry, and materials effects. Initial results indicate that an average flux of 3×10^{15} $neutrons \cdot cm^{-2} \cdot sec^{-1}$ can be obtained in a 100-g sample having an effective thermal cross section of 75 barns. (AEC Activity 4510)

PROGRAM 5000 — PHYSICAL RESEARCH

Reactor Operations. — The ORR has performed very well recently. In cycle 7 only one spurious scram occurred in almost three weeks of operation. Arrangements have been made with the manufacturer of the air coolers to repair them in order to permit them to meet specifications. This work is scheduled to start January 26. The turbulators will also be repaired at the same time.

A proposal is being written for annealing the stored energy in the Graphite Reactor by reversing the air flow.

PHYSICS

Neutron Velocity Selector. — The total cross section of Am^{241} has been measured for neutrons of energies from 0.2 to 45 ev. Two solid samples of AmF_3 and two liquid samples of $Am(NO_3)_3$ in D_2O, resulting in sample thicknesses from 7.87×10^{19} to 1.92×10^{21} atoms/cm^2, were used. The data up to 17 ev have been analyzed to give the resonance parameters listed below. An additional 19 resonances were observed from 17 to 32 ev.

Resonance Parameters of Am^{241}

E_0 (ev)	$2g\Gamma_n$ (ev)
	$\times 10^{-3}$
0.306	0.060 ± 0.003
0.575	0.075 ± 0.007
1.275	0.39 ± 0.02
1.93	0.125 ± 0.006
2.375	0.080 ± 0.010
2.60	0.20 ± 0.02
3.99	0.26 ± 0.02
4.40	0.027 ± 0.06
5.00	0.21 ± 0.02
5.44	1.3 ± 0.2
6.14	0.23 ± 0.03
6.78	0.059 ± 0.015
8.10	5.0 ± 2.0
9.14	0.80 ± 0.12
9.90	0.71 ± 0.11
10.4	0.7 ± 0.2
11.05	0.65 ± 0.06
12.9	0.24 ± 0.05
14.8	3.5 ± 0.4
15.8	0.40 ± 0.12
16.5	1.8 ± 0.3
17.0	0.7 ± 0.2

(AEC Activity 5220)

High-Voltage Experimental Program. — A new time-of-flight measuring apparatus is under construction and is presently being tried out with coincidences produced by an Na^{22} source. It differs from conventional time-to-pulse-amplitude converters in that it produces a d-c output large enough to be fed directly into a multichannel sorting device. By eliminating separate amplifiers and pulse stretchers between the time conversion and the voltage measurement, potential sources of scale-factor and scale-zero drifts, nonlinearities, counting rate dependence, and overload effects are eliminated. In particular, the converter has all the features of a precision pulse stretcher and is arranged to cancel the stretching period for those events whose delay time exceeds the useful time range T_{max}. Thus the total dead time for each such event is reduced to $T_{max} + T_{reset}$, with $T_{reset} \leq 0.5$ μsec (such events, which are the majority in many experiments, would overload the linear amplifier required with the conventional arrangement).

Since practically all operations are performed by d-c coupled logical circuitry, the time conversion factor and stretching period may be chosen arbitrarily in a wide range. Presently available conversion factors of 3, 6, 12, 24, and 48 mμsec/v, with the output scan set at 67 v, correspond to a selection of T_{max} from 200 mμsec to 3.2 μsec. Time resolution is 4 mμsec full width at half maximum, as tested with the annihilation coincidence by use of fast scintillators, and appears to be independent of the length of delay cable inserted in the "stop" channel, up to at least 140 mμsec. Factors governing linearity, long-term stability, maximum useful stretching time, the "walk" effect, and behavior at >10,000 counts/sec are being further investigated. (AEC Activity 5220)

Charge Spectrometry. — Following the beta decay of a tritiated hydrocarbon, the organic fragment is left with a distribution of excitation energy. To calculate this distribution, data (ORNL-2546; J. Phys. Chem. 62, 1377, 1382, 1958) from the fragment spectra resulting from the decay of CH_3T, C_2H_5T, and C_6H_5T were subjected to analyses with the use of a semiempirical unimolecular decomposition model. The calculations show that in about 70% of the decays little or no excitation is involved, but that in the remaining cases the organic fragment can be excited with energies up to 40 ev. These findings are consistent with the quantum mechanical calculations made by Migdal (J. Phys., USSR, 4, 779, 1956) of the excitation following the atomic transition $H^3 \longrightarrow He^+ + \beta^-$. (AEC Activity 5220)

Neutron Diffraction. — Neutron diffraction investigations have been performed on CrF_2 and $CrCl_2$ to determine the existence of magnetic ordering in these compounds at low temperatures. Although the chemical unit cells of these compounds are monoclinic and orthorhombic, respectively, both structures are distortions of the rutile structure and are consequently similar to the structures of MnF_2, FeF_2, CoF_2, and NiF_2, which have been investigated previously at ORNL. Antiferromagnetic reflections were observed from polycrystalline CrF_2 below about 50°K, and these reflections can be indexed by a magnetic unit cell which has the same dimensions as the chemical unit cell. The magnetic structure appears to be identical with those of the other iron-group difluorides, in which the

atomic moments at the lattice corners are antiparallel to those at the center of the unit cell. In $CrCl_2$, an antiferromagnetic transition is observed at about 20°K, and the magnetic structure is different from those of the difluorides. The antiferromagnetic reflections require a magnetic unit cell in which the b axis and c axis are twice those of the chemical cell, but the a axis is the same as that of the chemical cell. The suggested magnetic structure has ferromagnetic (011) planes with adjacent planes antiparallel. In both compounds the atomic magnetic moments have values which are close to those expected for the Cr^{++} ions if the orbital contribution is quenched. (AEC Activity 5220)

Low-Temperature, Nuclear and Solid State Physics. — A study of the broad-area germanium surface-barrier counters described previously (ORNL-2471) has been carried out on both theoretical and experimental bases. A simple theory gives satisfactory agreement with measurements at 77°K of the sizes and rise times of pulses from alpha particles. Pulses ranging from 0.05 to 1.5 mv were observed, depending on the counter area (1 to 5 cm^2) and applied bias. At 77°K, rise times were found to be less than 2×10^{-8} sec. At this temperature, the theory also successfully predicts the capacitance of the counters in terms of the purity of the germanium used and the applied bias. At liquid-helium temperatures the rise time was found to be longer; this indicates that the electrostatic potential gradient was not confined to a thin barrier region, probably because of the very large electrical resistivity of germanium at these temperatures. (AEC Activity 5220)

CHEMISTRY

Raw Materials Research and Development. — The primary amines 1-undecyldodecylamine and 1-nonyldecylamine, which can be made available commercially, showed thorium extraction equal to that of Primene JM-T, with <5 ppm steady-state loss to aqueous liquors. (AEC Activity 5310)

Extraction Reagent Performance. — Two of the newer amine extractants, N-benzylheptadecylamine and trioctyldecylamine, were irradiated with 1-Mev electrons to 200 and 400 whr/liter, respectively. In each case a polymer was the primary condensed-phase product and hydrogen the primary gaseous product. Per 100 ev of absorbed energy, respectively, 1.81 and 2.20 molecules of reactant were converted to polymer and 1.79 and 2.98 molecules of hydrogen were formed.

In one equal-volume contacting, 2 M NaOH solution removed only 56% of the acid formed during irradiation of TBP-Amsco solution to 500 whr/liter. The resulting acidity of the extractant solution was 0.186 N, indicating very inefficient purification. Preliminary data suggest that $Ca(OH)_2$ as a saturated aqueous solution, ~0.044 N, is more efficient.

Olefin formation in TBP-Amsco solutions irradiated to 400 whr/liter decreased from 3.37 to 1.00 molecules per 100 ev as the TBP in the reagent was increased from 4.5 to 100 g per 100 ml of Amsco. (AEC Activity 5310)

Solvent Extraction Research. — Tri-sec-butyl phosphate, tri(2-octyl) phosphate, di-n-butyl phenylphosphonate, and di-sec-butyl phenylphosphonate gave ten or more times greater uranium separation than tributyl phosphate from thorium, rare earths, and ruthenium but not from Zr-Nb. Tracer studies confirmed that high zirconium extraction is due chiefly to acid and ester hydrolysis products of neutral polyphosphate contaminants.

The coefficients for the extraction of Np(IV) with 0.1 M di(2-ethylhexyl) phosphoric acid (D2EHPA) in Amsco 125-82 from 0.25 and 2.5 M H_2SO_4 solutions were 500 and 0.05, respectively, decreasing by approximately the fourth power of the acid concentration. Extraction increased on partial neutralization but decreased on adding sodium sulfate at constant acid concentration.

Extraction of Fe(III) from acid perchlorate solutions by D2EHPA in octane at low loading varies with the 2.65 power of the D2EHPA concentration in the range 0.001 to 0.1 M (ORNL-2636). Extensive measurements of E_a^o vs D2EHPA and $Fe(ClO_4)_3$ molarities ruled out the formation of $Fe(OH)X_4H_2$ as the explanation of the low dependence. At high iron loading, extraction isotherms showed inflections at Fe/D2EHPA mole ratios of 1/6 and 2/5. Isopiestic measurements showed the formation of associated iron species. Appreciable perchlorate was extracted with the iron, and charge balances indicated partial iron hydrolysis. (AEC Activity 5310)

Chemistry of Corrosion. — Electrochemical kinetic studies were continued on the mechanism of reduction of uranyl ions on passive stainless steel in aqueous sulfate medium. In particular, the effect on the reaction kinetics caused by the complexing of uranyl ions with sulfate ions is under investigation. Accurate values of the dissociation quotient of the bisulfate ion, at temperatures from 20 to 85°C, are prerequisite for treating data on the systems studied. Accordingly, potentiostatic and galvanostatic techniques were developed which allowed precise determination of the bisulfate dissociation quotient for the temperatures 22 and 45°C, at an ionic strength of 0.3. Other measurements are in progress at 67 and 85°C.

By cathodic polarization of iron electrodes passivated in aerated inhibitor solutions at pH 5.4, it was shown that, in both benzoate and chromate solutions, the cathodic current induces reduction chiefly of oxygen. In an atmosphere of helium, no steady-state potential could be obtained in the chromate solution, even at a current density as low as 10^{-7} amp/cm^2. On the contrary, in an oxygenated benzoate solution, a 100-fold larger current density gave a stable potential falling on a linear Tafel line. In an oxygenated chromate solution, addition of sulfate ions lowers the current density at which activation sets in. (AEC Activity 5330)

Chemistry of Technetium. — A spectrophotometric study of the reduction of pertechnetate ion (TcO_4^-) by $SnCl_2$ in HCl solutions showed that no products of intermediate technetium valence appear between VII and IV, the final state. This holds over a range of HCl concentration from 5 M to less than 1 M. At the highest acidity (5 M), the product is $TcCl_6^{--}$. At lower acidities, two different Tc(IV) species, presumably oxygenated, appear, identified

by different absorption maxima. At HCl concentrations below 1 M, both appear; in 1 to 2 M HCl, only one; and in 3 to 4 M HCl, the same one plus $TcCl_6^{--}$. (AEC Activity 5330)

Characterization of Short-Lived Fission Products. — Information on the decay properties of I^{134} accumulated to date is as follows: Its half life was measured as 52.8 ± 0.4 min. Prominent gamma rays were found at 0.12, 0.23, 0.42, 0.62, 0.85, 0.89, 0.99, 1.08, 1.17, 1.47, 1.60, 1.79, 2.29, and 2.43 Mev, with several additional transitions of low intensity. In a gamma-gamma coincidence measurement with the single-channel window set on the low-energy side (0.82 Mev) of the double peak at 0.85 to 0.89 Mev, gamma rays were observed with energies of 0.24, 0.39, 0.50, 0.55, 0.83, 0.89, 0.98, 1.08, and 1.16 Mev. In another measurement with the window at 0.91 Mev, the coincidence spectrum appeared very similar, except that better counting statistics in this case showed additional weak transitions up to 1.8 Mev. The spectrum coincident with 1.08 Mev contained gamma rays at 0.13, 0.24, 0.44, 0.62, 0.78, 0.86, and 0.96 Mev. With the window covering the 1.80-Mev region, a pronounced peak was observed at 0.86 Mev plus several less intense transitions at lower energies.

An I^{134} beta-ray spectrum was taken on an anthracene scintillation spectrometer, and its Fermi analysis gave groups at 2.41 ± 0.05, 2.07 ± 0.06, and 1.62 ± 0.07 Mev. Beta-gamma coincidence experiments indicate that the beta ray of highest energy feeds a level in Xe^{134} at about 1.7 Mev. (AEC Activity 5330)

Nuclear Methods of Chemical Analysis. — A routine analytical method based on thermal-neutron activation and a radiochemical separation procedure has been developed to determine trace oxygen in lithium metal. The thermal- neutron activation involves the irradiation of a lithium sample in a reactor and the production of a radionuclide (in this instance, F^{18}, 1.87- hr) from the stable oxygen atoms contained in the sample by the neutron reactions $Li^6(n,\alpha)H^3$ and $O^{16}(H^3,n)F^{18}$. The radioactive F^{18} is separated from other induced radionuclides in the sample by means of a radiochemical separation procedure in which a known amount of inactive fluorine is added as an isotopic carrier to a solution of the irradiated sample. The inactive fluorine and the F^{18} are distilled from the mixture and precipitated as lead chlorofluoride, PbClF. The gamma radioactivity of the F^{18} is measured by means of a scintillation counter. The concentration of oxygen in the unknown sample is measured by comparing the F^{18} found in it with the F^{18} found in a comparison sample irradiated and radiochemically processed in the same manner. Concentrations as small as 20 μg of oxygen per gram of lithium have been determined by this method. The limits of measurement for the present irradiation conditions (3.0 hr at a flux of 6.2×10^{11} neutrons·cm^{-2}.sec^{-1}) are of the order of 2.0 μg. Increased sensitivity can be obtained by use of more intense neutron sources.

The method is applicable to the analysis of other metals and their compounds for trace oxygen. (AEC Activity 5330)

Chemical Separation of Isotopes. — A single-stage separation factor for oxygen isotopes of 1.016 at 21°C was measured between SO_2 gas and liquid H_2O; O^{18} concentrated in the gas phase. No fractionation of oxygen isotopes was observed upon equilibrating SO_2 gas with SO_2 dissolved in ethylene sulfite. For analysis, the SO_2 from the above experiments was converted to H_2O by reaction with H_2S. The H_2O was equilibrated with CO_2 in the usual manner, and the mass spectral analysis was performed on the CO_2.

The fractionation of lithium isotopes by ion exchange chromatography was studied as a function of temperature. Lithium adsorbed on Dowex 50-X16 was eluted with 0.25 N HCl. The measured separation factors were 1.0032 at 3°C, 1.0028 at 20°C, 1.0027 at 40.5°C, and 1.0025 at 59°C. (AEC Activity 5340)

METALLURGY

High-Temperature Reactions of Metals and Ceramics. — The optical absorption bands for halide ions known as charge transfer to solvent (C.T.T.S.) bands are quite sensitive to changes in the chemical environment of the halide ions. The theory governing this behavior should be applicable semiquantitatively to halides in fused salt mixtures. A recent publication (by others) reported C.T.T.S. bands for the Br^- and I^- ions in fused LiCl-KCl eutectic which are quite out of line with the theory. Consequently, measurements in the case of the I^- ion were repeated at ORNL. The results are in line with the theory and hence are very different from those previously reported. The C.T.T.S. band for I^- was measured in the LiCl-KCl eutectic at 400°C, using concentrations of 0.01 to 0.28 M at path lengths of ca. 0.2 and ca. 0.006 cm. The long-wavelength absorption edge of an approximately Gaussian band was found which increases without peak to 215 mμ (ultraviolet limit of the monochromator). Preliminary analysis indicates a peak between 200 and 210 mμ with an extinction coefficient of 10^3 to 10^4. A numerical analysis of the method of measurement used to obtain the erroneous data referred to above is in progress, and it appears that the trouble was undoubtedly a large stray-light error. (AEC Activity 5420)

Radiation Effects on Oxidation. — Previously reported studies of the oxidation characteristics of niobium showed that a correlation existed between the shape of the oxidation rate curves and the formation of cracks in the oxide film. The oxidation rate curve for tantalum at 510°C has now been determined, and, as in the case of niobium, a transition from protective to nonprotective oxidation was observed. Electron micrographs of the oxidized specimens showed that this transition was associated with crack formation in the oxide. It was concluded that the oxidation mechanisms for niobium and tantalum are similar and that the cracking of the oxide films is produced by the stresses generated in the films as a result of anion diffusion. (AEC Activity 5420)

PROGRAM 6000 — BIOLOGY AND MEDICINE

BIOLOGY

Pathology and Physiology. — The mortality of mice 30 to 120 days after the administration of 900 r of x radiation to the whole body was increased in F_1 recipients of parental-strain bone marrow when the parental marrow

donors were previously sensitized against the recipients; in irradiated (101 x C3H), or reciprocal, F_1 hybrids injected with 101 or C3H marrow, the mortality was increased from about 10 to 100%, and in irradiated (C57BL x 101) F_1 recipients injected with 101 marrow, mortality was similarly increased from about 10 to 100%. In (C57BL x 101)F_1 recipients injected with C57BL marrow, mortality was increased from 85 to 100%. Presensitization to rat antigens did not affect mortality. It is noteworthy that the rate of delayed mortality induced by parental marrow in F_1 recipients varied from one parental donor to the other and was lower than that induced in the homologous parental recipient. These results indicate the importance of the antibody-forming capacity of the inoculated marrow cells, as well as the extent of histocompatibility differences between donor and recipient, in the pathogenesis of the delayed foreign bone marrow reaction. (AEC Activity 6130)

Biophysics: Electron Spin Resonance Studies. — Glycine was adsorbed from solution on colloidal silica, the glycine-silica system was allowed to set to a gel, and, after desiccation, the system was irradiated with cobalt gamma rays. Magnetic resonance signals observed in these systems are considerably less than those found from equal amounts of glycine irradiated in the pure crystalline state. From these preliminary results it appears that the spin resonance induced in crystalline amino acids probably arises from radiation-induced lattice defects or from other magnetic centers in the crystals rather than from molecular free radicals. Further study of these systems is continuing. Preparation of amino acid microcrystals in the submicron range is in progress; they will be used in similar studies.

The preliminary relative biological effectiveness (RBE) measurements for the long-lived electron spin resonance phenomena in crystalline amino acids gave the following results:

Acid	RBE (in Comparison with X Rays)		
	250-kvp X Rays	Co^{60} Gamma Rays	14-Mev Neutrons
Glycylglycine	1.00	0.82	0.56
DL-α-Alanine	1.00	1.21	0.51
Glutamic acid	1.00	1.02	0.22

These results are qualitatively similar to previous values found by Kirby-Smith for glycine. (AEC Activity 6130)

Biophysics: Glycine Dosimetry. — In connection with the investigation of paramagnetic resonance in single crystals of glycine, considerable coloration of the irradiated crystals has been observed. Qualitative study of the increase in optical density induced by radiation (Co^{60} gamma rays and 14-Mev neutrons) in glycine crystals has shown that they can be used as dosimeters in the high dose ranges. The close tissue equivalence and the small dimensions of such dosimeters make them an attractive possibility for radiation measurements. (AEC Activity 6130)

Mammalian Recovery. — S,2-Aminoethylisothiourea (AET) protects the intestine of mice exposed to 900 or 1500 r of x rays. Some protection was observed after 2000 r. Protection was ascertained by histological and cytological examination and by wet and dry weights of the intestine.

There are indications that radiation-induced cell hypertrophy in the intestine of irradiated mice is dependent on the x-ray dose. Similar studies with testis are in progress.

Testis weight loss in the irradiated mouse during the first four weeks after exposure is not significantly offset by AET. (AEC Activity 6230)

Radiation Immunology. — Electrophoretic analysis of mouse serum samples obtained at intervals from one to eight weeks after exposure to 950 r of x rays and treatment with isologous bone marrow (950 r—IBM), rat bone marrow (950 r—RBM), or S,2-aminoethylisothiourea (AET—950 r) revealed that the serum protein composition of only the 950 r—RBM mice deviated significantly from the normal control. The albumin concentration remained low from the second through the seventh week, and the lipoprotein concentration increased during this period. These changes are associated with the in vivo antigen-antibody reaction.

Further studies on transplantation of isologous spleen from immunized donors into 950-r mice showed that as few as 0.5×10^6 to 1×10^6 nucleated spleen cells can be detected in terms of antibody production. In contrast, approximately 1000 times as many cells must be used in the rabbit for a comparable study. Continuation of these studies will permit an accurate determination of the LD_{50} of antibody-producing cells, the existence of radioresistant and radiosensitive stages, and the development of the recognition mechanism. (AEC Activity 6230)

PROGRAM 8000 — WORK FOR OTHERS

International Reactor Courses at ORNL. — Students for the international courses in Nuclear Reactor Operations Supervision and Nuclear Reactor Hazards Evaluation have been selected and were notified of their acceptance by the Division of International Affairs, AEC. Twenty-six students representing 15 foreign countries reported to ORNL late in January. (AEC Activity 8717)

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