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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JUNE 1959

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

June 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

Distribution and Excretion of Uranium in Man. — In a study of the effect of size of dose upon the deposition and retention patterns of uranium, uranyl nitrate was injected intravenously into rats at three dose levels: 10, 100, and 1000 μg of uranium per kilogram of body weight. The rats were killed at 1, 2, 4, 8, and 12 weeks, and the amounts of uranium remaining in various tissues were determined. The percentage of the initial dose retained in the kidney varies inversely with the quantity initially injected. (AEC Activity 2120)

Power Reactor Fuel Reprocessing: Pilot Plant. — The plant was on stream 30 days during the month, and a total of 12.5 metric tons of BNL irradiated uranium fuel containing 6.4 kg of plutonium was processed.

Operation of two extraction columns in the plant with the organic phase continuous resulted in a ten- and threefold increase in decontamination in the codecontamination cycle and the final uranium cycle extraction columns, respectively. These changes eliminated the need for silica gel treatment of the final uranium product. Both the uranium and the plutonium products meet fission product specification for subsequent processing. On the basis of measured losses from the plant, the product recoveries for uranium and plutonium were 99.3 and 99.7%, respectively. (AEC Activity 2351)

Dissolver Solution. — The studies of the extraction of uranium into hexone from "acid deficient" aluminum nitrate solution indicate that this procedure will be a valuable supplement to the triisooctylamine extraction procedure. The hexone procedure was evaluated by analyzing standard uranium solutions. The relative standard deviation was found to be 0.5% when 10 mg of uranium was determined.

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The triisooctylamine-extraction coulometric-titration procedure for the determination of uranium has been applied to the analysis of synthetic Darex dissolver solutions. The relative standard deviation, based on 15 determinations, was found to be 0.4%. This procedure was also applied to the analysis of synthetic Sulfex dissolver solutions; the relative standard deviation, based on 12 determinations, was found to be 0.3%.

Development work on the anion exchange separation procedure for plutonium has been completed. The procedure consists in adsorbing Pu(IV) from 1:1 (by volume) HNO_3 onto 3 ml of Dowex 1-X4 (50-100 mesh) and washing the column with 25 ml of the acid. The plutonium is then eluted directly into a cell with 15 ml of hot 0.5 M HCl for titration. Radiometric studies of this separation indicate that the loss in adsorption and washing is less than 0.1% and that 99.9% of the plutonium is eluted in the first 15 ml of eluant. This is true for both pure plutonium solutions and synthetic uranium fuel solutions. (AEC Activity 2724)

PROGRAM 4000 – REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. – HRT run 20 was started after installation of the multiple-hydroclone assembly (reported last month).

While a sample was being taken soon after power operation began, a valve stem broke in the isolation chamber of the blanket high-pressure sampler, and the sampler assembly had to be removed for repairs.

After replacement of the sampler, operational testing was resumed. The reactor has operated at 1400 psig and 260°C at power levels up to 1.4 Mw, so that component performance could be evaluated. All the reactor equipment has operated satisfactorily except the fuel recombiner. Incomplete recombination of radiolytic gas in the fuel low-pressure system has prevented continuous power operation. An attempt is being made to reactivate the platinum catalyst by heating it to a temperature above 600°F. (AEC Activity 4103.3)

HRT Chemical Pilot Plant. – The effect of faster processing on the rate of collection of solids by the hydroclone system in the HRT chemical plant is being tested by use of an assembly of thirteen 0.6-in.-dia hydroclones in parallel. The pressure differential across the reactor heat exchanger provides the driving force to circulate about 10 gpm of fuel through the multiclone unit. From the multiclone unit 1 gpm of concentrated underflow goes to the original one-hydroclone system, where the solids are finally collected. In the first 50-hr run the rate of collection of solids was 5.5 g/hr, as compared with an average of about 1.5 g/hr during a comparable period of reactor operation with a single 1-gpm hydroclone. In the second run, of 90 hr duration, the collection rate was 3 g/hr. However, it will not be possible to accurately compare the performance of the multiclone unit with previous experience until an equilibrium removal rate is established after several hundred hours of reactor operation. (AEC Activity 4103.1)

Fuel Processing. – In three runs with 0.4 m UO_2SO_4 in H_2O containing 0.045 m nickel, 45% of the nickel was electrolytically removed from 15 gal of solution by 10,000 amp-hr of current. The cell was glass-lined and had a platinum anode and a mercury cathode. Nickel was still being removed when the experiment was terminated. In laboratory experiments the removal of nickel from D_2O solutions was about 30% faster than from H_2O.

Regeneration of the mercury cathode for re-use may be possible with a slurry of mercurous sulfate in 1 M H_2SO_4. Agitation of the used cathode in this reagent for 3.5 min removed 95% of the nickel and copper. Mixtures of nitric acid and hydrogen peroxide cannot be used for regeneration of the mercury cathode if the nitric acid and hydrogen peroxide concentrations are greater than 1 M and 1%, respectively, because of excessive attack on the mercury. (AEC Activity 4103.1)

Thorium Oxide Slurry Development. – In a flame-calcined thorium-uranium-aluminum oxide (1.5–2.0 μ average particle size) with 8 wt % uranium oxide and 2.5 to 10 wt % aluminum oxide, more than 50% of the particles observed in an electron micrograph were spherical. Yield stresses for slurries of spherical particles, 2 g/cc density, were 0.02 to 0.05 psf, as compared with 0.2 to 1.0 psf for nonspherical ThO_2 or ThO_2-UO_2 slurries of the same density. With 1 wt % aluminum oxide the particles were random-shaped aggregates of nonspherical particles; 3 wt % Al_2O_3 in 94% ThO_2 and 3% UO_2 may be most favorable for production of spherical particles.

Samples of a silica-stabilized thoria sol, particle size 1000 to 1500 Å, formed a small cake after 486 hr of heating at 300°C in a stainless steel ampoule, but the bulk of the sol appeared unchanged. Another sample broke to a soft gel after 3.5 hr of heating and stirring at 300°C; analysis of a dried sample showed more than 1000 ppm of corrosion products. In a sample heated for 286 hr in a quartz ampoule a small amount of material flocculated but was readily redispersed by shaking.

A rolling-ball viscometer was constructed for determining the viscosities of small samples (<10 ml) of irradiated aqueous thoria slurries at temperatures up to 300°C. The terminal velocity of fall of a steel ball through a fluid is measured magnetically over a range of angles of inclination of the tube. In room-temperature tests of a 6-ml viscometer with a 0.2425-in.-dia tube and 0.1885- to 0.225-in. balls, results were accurate with 1- to 400-centipoise Newtonian liquids and with slurries of 650°C-fired thoria at concentrations up to 800 g/liter and with yield stresses up to 0.3 psf.

With a slurry of 1000°C-fired ThO_2 at a concentration of 500 g of Th per kg of D_2O, containing 55 ppm of platinum and 0.05 m MoO_3, and preheated under D_2 at 270°C, the D_2-O_2 recombination rate was 8 moles of D_2 per liter per hour at 160°C and a D_2 partial pressure of 100 psi. Subsequent heating of the slurry at 280°C under oxygen lowered the rate to 3 moles of D_2 per liter per hour at 173°C. On the assumption that 100 psi is an acceptable partial pressure of radiolytic deuterium (excess oxygen is usually present) at 280°C in a slurry blanket or core, the catalytic activity of a thorium or thorium-uranium oxide slurry without additives is sufficient for a reactor power level of several tenths of a kilowatt per liter, depending on slurry concentration. The catalytic activity of 0.001 m MoO_3 in ThO_2 slurries is sufficient for a 1-kw/liter power density; in thorium–0.5% uranium oxide slurries it is sufficient for 0.1 kw/liter. The equilibrium activity of 0.001 m palladium is sufficient for 2 to 3 kw/liter (based on out-of-pile tests or in-pile tests of more than 500 hr duration) in any thoria-urania slurry of current interest. (AEC Activity 4103.1)

HRP Chemistry: Heterogeneous Equilibria in Aqueous Systems. – Temperatures at which two liquid phases occur in compositions containing UO_2SO_4, $CuSO_4$, $NiSO_4$, and D_2SO_4 in the molar ratio existing in a typical homogeneous reactor fuel were determined as a function of D_2O content. The data indicate that a second liquid phase, rather than a solid phase, would always be the initial phase produced upon concentrating such a fuel solution by localized

boiling within the reactor. Subsequent higher temperatures would convert the second liquid phase to solid.

The distribution of cupric ion between the two liquid phases of the system UO_3-CuO-SO_3-H_2O was determined at 300, 325, and 350°C; the copper was distributed in approximately the same mole ratio as uranium.

An investigation of the effect of nitrate ion concentration on the two-liquid-phase temperature in the system UO_3-SO_3-N_2O_5-H_2O indicated a considerable elevation of this phase-transition temperature as the NO_3/SO_4 ratio was increased. (AEC Activity 4103.1)

HRP Chemistry: Reactions in Aqueous Solutions. – The energies of activation were calculated for the homogeneous catalysis of the activation of D_2 by dissolved cupric salts in two acid D_2O solutions. Experiments at 100, 110, 120, 130, and 140°C with 0.01 M D^+, 0.1 M Cu^{++}, and a pressure of 20 atm of D_2 gave results which indicated an activation energy of 27,500 kcal/mole; similar experiments with 1.0 M D^+ gave results indicating an activation energy of 24,700 kcal/mole. The rates of reaction in the solution of higher acidity were 39 and 31%, respectively, of the rates in the solution of lower acidity at 100 and 130°C. These results are based on the assumption of constant D_2 solubility and are subject to correction when better solubility information becomes available.

Vapor-pressure measurements of the recently discussed (ORNL-2736) crystalline compound $UO_4 \cdot 4H_2O$ gave a value of 12 ± 2 mm Hg at 27 ± 1°C, which could be obtained from above or below; this suggests that a true equilibrium probably exists in accordance with the following equation:

$$UO_4 \cdot 4H_2O \rightleftharpoons UO_4 \cdot 2H_2O + 2H_2O$$

(AEC Activity 4103.1)

HRP Chemistry: Gaseous Fission Product Disposal. – A brief study of propagation of deuterium-oxygen explosions through Columbia G activated carbon, 8 to 14 mesh (the charcoal used in the HRT adsorber system), indicated that the limits of flammability for D_2-O_2 mixtures in charcoal were not appreciably different from those obtained previously in a more extensive investigation with H_2-O_2 mixtures.

Measurements of apparent thermal conductivities for Columbia G charcoal with oxygen and helium sweep gases were made by both a direct and an indirect method. In the latter, heat generation in the charcoal resulting from beta decay of adsorbed fission gases was simulated by resistance heating of the charcoal, a fair conductor of electricity. Krypton holdup times were used to evaluate the resulting temperature profiles in the charcoal; from these, in turn, the corresponding values of thermal conductivity were deduced, uniform heat generation being assumed. The lowest values thus far obtained by

these methods are 0.06 $Btu \cdot hr^{-1} \cdot ft^{-2} \cdot (°F/ft)^{-1}$ when oxygen is the sweep gas and 0.12 $Btu \cdot hr^{-1} \cdot ft^{-2} \cdot (°F/ft)^{-1}$ when helium is the sweep gas. (The value 0.06 is twice that which was employed in design calculations for the HRT fission-gas disposal system.) (AEC Activity 4103.1)

HRP Analytical Chemistry. – Molybdenum can be determined in impure ThO_2-UO_x slurries by a derivative polarographic method based on the reduction wave of molybdenum at either -0.48 or -0.64 v. vs the S.C.E. in 0.15 M nitrilotriacetic acid at pH 3. By utilizing the wave at -0.64 v and applying a standard addition technique, molybdenum in the range 20 to 200 μg/ml can be estimated with a coefficient of variation of approximately 5%. Uranium(VI) and chromium(VI) can be tolerated in concentrations up to ten times that of the molybdenum.

Basic spectral data were collected for use in further evaluation of the applicability of flame photometry to the estimation of the rare-earth elements, except cerium and promethium, in the mixtures occurring in simulated blanket slurries or fuel solutions. Detailed flame spectra of the several elements were taken by means of Hilger prism and Jarrell-Ash grating spectrographs. By this means, characteristic bands and lines of the different elements were located accurately. This information was then utilized to identify the principal bands and lines of the flame spectra that were subsequently recorded with an ORNL model 1457A flame photometer, equipped with a shield to mask out interference by the luminous inner cone of the flame. (AEC Activity 4103.1)

Slurry Component Development and Slurry Testing. – A new 50-gpm Byron-Jackson pump circulated slurry at 300°C through the 200A loop with satisfactory mechanical performance. The slurry was prepared from 800°C-fired 48-hr-digested thoria of 1.8 μ average particle size; during a 295-hr run the particles were degraded to between 1.0 and 1.5 μ. Nonhomogeneous distribution in horizontal pipes at 5 fps was indicated by samples. (AEC Activity 4103.1)

Slurry Blanket System Development. – Run SM-7, in the 300-SM blanket test facility, was completed. In this run, a thoria slurry containing 8% uranium was circulated at 40 to 200°C, and its distribution in a 30-in. core vessel was studied. Concentration gradients were observed at the higher temperatures but were not apparent at 40°C. Following this run, weak points in the system were strengthened to permit operation at 2000 psi.

In 1640 hr of slurry circulation during runs SM-6 and SM-7, the Zircaloy pump impeller lost only 0.5 g out of 1421 g. Bearing wear was negligible.

Construction of the 300-SM low-pressure system was completed, and the performance of the low-pressure equipment with water is being studied. (AEC Activity 4103.1)

Engineering Research. – Additional data obtained with ThO_2 suspensions having average particle diameters up to 13 μ continue to indicate that the yield stress, on the average, is proportional to the cube of the volume fraction of solids. However, the yield stress appears to be inversely proportional to the 1.7 power of the particle diameter, rather than the second power as previously reported. The minimum average particle diameter required to ensure a yield stress less than 0.1 psf at a volume fraction of solids of 0.10 is about 2.5 μ.

Cake formation resulted when ThO_2 spheres (produced in a 200A loop run) were circulated in the 30-gpm loop in the presence of CrO_3 equivalent to 1500 ppm (based on thorium). A heat transfer unit designed for use in determining film thickness in the multidiameter sample barrel indicated an oxide film thickness of 1.8 mils in the 5-fps section after the run with 1500 ppm CrO_3. By weighing oxide obtained from a measured surface area, the indicated film thickness was 1.6 mils. (AEC Activity 4103.1)

Core and Blanket Vessel Development. – Velocity and heat transfer measurements made at the wall of the re-entrant 48-in. core model were shown to be consistent. An expression was derived, combining boundary-layer theory and the velocity measurements, for predicting wall shear stress in the vessel.

In operation of the 30-in. slurry core vessel with slurry containing 500 g of ThO_2 per kg of H_2O, uniform slurry distribution was attained at flow rates above 60 gpm in the range 25 to 90°C. Surface heat transfer coefficients were approximately equal to those measured earlier with water. It was found that sampling bias from a 3-in. pipe was increased from 4% at 25°C to 10% at 90°C in a poorly oriented sample probe.

The settled-bed density of flocculated ThO_2 slurries decreased linearly with temperature in the range 25 to 200°C. Density at 200°C averaged two-thirds of that at room temperature. (AEC Activity 4103.1)

Centrifugal Circulating Equipment. – The HRT replacement fuel circulating pump was fitted with a stainless steel 400-gpm impeller, and performance data were taken at room temperature over its operating range. The pump was also pretreated and cleaned in consecutive runs by circulating trisodium phosphate solution, dilute nitric acid, and hot oxygenated water.

The 300A slurry pump in a 2800-hr run at 280°C, circulating approximately 460 g of ThO_2 per kg of H_2O, lost only 2.5 g from its titanium impeller, which was in excellent condition. No wear could be detected in the mechanically mounted alumina bearings. A subsequent run was terminated as a result of failure of experimental flame-plated alumina bearings.

Operation of the 20-cfm blower was suspended following a large leak of the cooling water into the stator cavity. (AEC Activity 4103.1)

Miscellaneous Equipment. – Development of equipment for inspection and modification of the HRT core was continued. Devices fabricated and tested successfully were a remote fly cutter and a remotely driven milling cutter for cutting screens loose, improved screen-manipulating tools, and a tool for making an impression of the hole in the core tank. Testing of mechanisms and procedures for removing the screens from the reactor was nearly completed. In a demonstration, screens were cut loose and a hole was plugged in a mockup of the reactor vessel. (AEC Activity 4103.1)

Heat Removal Equipment. – The design of the heat exchanger test facility was completed, and construction was begun. Tubing manufacturing for the test units was completed.

The cleaning of a plugged U-tube bundle by combined use of vibration and hydraulic pressure was found to be incomplete, although many plugged tubes were cleaned. (AEC Activity 4103.1)

Gas Handling Equipment. – The natural-circulation high-pressure recombiner completed a 2500-hr run at 1500 psi and was dismantled for examination. The internal appearance was excellent. (AEC Activity 4103.1)

Fuel Systems Development. – Following the completion of run CS-24, a dump-tank weighing system and other minor auxiliaries were installed in the HRT mockup to aid in improving the determination of the uranium inventory. Run CS-25, to study the problems of determining the fuel inventory, was started on June 9. (AEC Activity 4103.1)

Feed Pump Development. – Successful operation of two prototype HRT feed-pump heads was continued beyond the two-year mark. Two slurry heads have operated without diaphragm failure for more than 5000 hr. Although negligible wear was observed on check-valve balls made of high-purity high-density alumina, seats of this material experienced severe mechanical damage. Beryllia seats were eroded rapidly.

Two diaphragm failures, caused by foreign matter, were repaired on the three-stage contaminated-service oxygen compressor. The test of the unit was continued.

A slurry dump tank, statically pressurized to 20 psi, was operated successfully without any means of agitation. The feed pump was coupled to the tank with a short suction line which was inclined 45° or more from the horizontal. Slurry of density 2.4 was circulated without difficulty following a 24-hr shutdown. (AEC Activity 4103.1)

MOLTEN-SALT REACTOR (MSR) PROGRAM

Chemistry. – Further investigations of the $NaF-BeF_2-PuF_3$ system, which has adequate PuF_3 solubility at 550°C to be of interest as a molten-salt fuel, show that a new compound, probably $NaF \cdot PuF_3$, forms a eutectic with NaF at 727°C and 22 mole % PuF_3; this is the lowest liquidus temperature attainable in the $NaF-PuF_3$ binary system, which has now been explored with cooling curves from 0 to 30% PuF_3.

The feasibility of using graphite as a moderator in direct contact with fluoride fuels is being investigated. An impervious graphite was found to contain no salt, as judged by weight change, after exposure for one year to $LiF-BeF_2-UF_4$ (62-37-1 mole %) at a flow rate of 1.1 gpm at 1300°F and 13 psig (3 psig helium cover plus 10 psi effective head due to pumping) in an INOR-8 loop. Weight losses varying from negligible to 0.05% and averaging 0.02% for 21 rods (70- and 10-g samples) were attributed to desorption of residual gases from the graphite; no dimensional changes or other evidence of erosion could be detected. The chromium content of the salt increased from 135 ppm to 550 ppm during the year of operation. The graphite had been degassed in the loop under vacuum for 24 hr at 1100°F and repressurized with argon before the loop was charged with salt.

Tests have shown that graphite is not wetted by the fluoride fuels, but pressures of 25 to 30 psia cause penetration of 2 to 3 vol % when ordinary graphites such as AGOT are soaked in $LiF-BeF_2-ThF_4-UF_4$ (62-36.5-1-0.5 mole %) for 48 hr. With breeder fuels containing 15 mole % ThF_4, the permeation of ordinary graphite by the salt increases to 9 vol % under the same conditions; some of the increase is due to a lower surface tension of the melt, but increased solubility of the UO_2 formed by reaction with oxide surface complexes in the graphite pores also seems to play an important part.

Another indication that precipitation of UO_2 will be much less prone to occur in blanket fuels containing high ThF_4 concentrations than in burner fuels without ThF_4 was obtained when 600 liters of dry air was passed through $LiF-BeF_2-ThF_4-UF_4$ (71-15-13-1 mole %) during five days at 600°C without apparent precipitation of UO_2. The main effects noted were attack of the nickel container and a corresponding consumption of oxygen to give an insoluble precipitate, tentatively identified as NiO.

In comparing the precipitation of UO_2 from UF_4 dissolved in LiF-NaF (60-40 mole %) at 750°C by structural metal oxides, Fe_2O_3 was found to remove 3 moles of U^{4+} for 4 moles of Fe_2O_3 added, while NiO reacted but little and Cr_2O_3 was almost inert.

Periodic sampling of the $LiF-BeF_2-UF_4$ (62-37-1 mole %) being circulated in an INOR-8 forced-circulation loop has been continued. The loop has now operated more than 4000 hr. For the last 3000 hr the Cr^{++} concentration in the fuel has remained at 500 ppm, which is probably a steady-state "equilibrium" concentration which could persist indefinitely. A similar INOR-8

loop containing $LiF-BeF_2-UF_4$ (70-10-20 mole %) has shown a Cr^{++} increase from 160 ppm to 210 ppm, in the first 1000 hr; this increase in chromium concentration is less than expected in view of the high UF_4 content.

In the continuing study of the corrosion behavior of fluorides, chromium was found to react with a molten mixture of SnF_2 and LiF to yield a product identified as Li_3CrF_6 by x-ray diffraction:

$$2Cr + 3SnF_2 + 6LiF \rightarrow 2Li_3CrF_6 + 3Sn$$

The corresponding treatment of vanadium metal gave a product with an x-ray pattern slightly shifted from that of Li_3CrF_6, so presumably it was Li_3VF_6.

Commercial zirconium chloride, known as Zirklor, has recently been advertised as a lubricant and packing material for use at temperatures up to 1000°C with no mention of the need for an inert atmosphere. Samples tested in the laboratory were attacked, however, upon heating to 325°C in air, and they reacted completely during a 15-hr period in air at 800°C. (AEC Activity 4103.1B)

Physical Properties. – The viscosities of two salt mixtures have been redetermined. For mixture 123 ($NaF-BeF_2-UF_4$, 53-46-1 mole %) the viscosity is given by the equation

$$\mu = 0.0234e^{5506/T},$$

and for mixture 126 ($LiF-BeF_2-UF_4$, 53-46-1 mole %) by the equation

$$\mu = 0.0104e^{6488/T},$$

where μ is in centipoises and T is in degrees Kelvin. (AEC Activity 4103.1B)

Fuel Processing. – A new modification of a molten-salt reactor fuel processing method gives promise of recovering uranium by a low-temperature volatility method and decontaminating from cesium the Li^7F to be recycled. Mixtures of HF and ClF_3 were used to achieve fluorination of UF_4 and, at the same time, dissolution of Li^7F. The UF_6 formed in this manner at a low temperature and with little corrosion could be flashed off and recovered by distillation or absorption from the HF that is to be recycled. In a separate test it was shown that when a 90% HF solution containing dissolved LiF and CsF was evaporated to lower the HF concentration from 90 to 42%, 97% of the LiF precipitated, leaving over 65% of the cesium in solution. (AEC Activity 4103.1B)

In-Pile Loop. – Operation of the second MSR in-pile loop in the MTR was terminated June 10, 1959, when the pump seized. The loop had circulated fuel for 987 hr, 766 of which were with the reactor at power. (AEC Activity 4103.1B)

Pump Development. – Three tests have been made of salt-lubricated journal bearings having three helical grooves (instead of the axial grooves used in previous tests). In order to improve the load-carrying ability, a dam was installed at the top of each groove in the third test bearing, and it has operated for more than 200 hr at loads up to 500 lb.

A 650-gpm pump has now operated continuously at temperatures of 1200°F or above with a molten fluoride salt for more than two years (17,500 hr). A second pump equipped with a bellows-mounted seal has operated continuously for more than 14,800 hr with NaK at 1200°F. (AEC Activity 4103.1B)

Design Studies. – A reference design for a 10-Mw (electrical) experimental reactor has been completed. The design chosen is a single-region, homogeneous burner, employing a semidirect maintenance concept and operating on a Loeffler steam cycle. It is estimated that the total cost of construction would be approximately $16 million, including indirect overhead and contingency costs aggregating $7 million. The design prepared can be used as a reference from which the cost and design problems of smaller or larger molten-salt reactors can be estimated.

The total fuel-salt volume in the reactor is 171 ft^3, of which 113 ft^3 is in the 6-ft-dia spherical core. The initial inventory of U^{235} in a thorium-free fuel salt is 62 kg, and it would increase after two years of operation without reprocessing to 107 kg. The burnup of uranium during this period would be 29 kg.

From previous studies it was known that the greatest expected operational strain in the system would come from a sudden stoppage of the single fuel-circulating pump. Simulator studies have shown that with complete stoppage of the fuel flow and no heat losses the temperature could rise only 110°F – a transient easily within the capability of the structure to withstand. (AEC Activity 4103.1B)

GAS-COOLED REACTOR PROJECT

Experimental Gas-Cooled Reactor (EGCR) Development. – Site work for the EGCR was initiated, and detailed design work is being done. A special meeting of the Gas-Cooled Reactor Subcommittee of the AEC's Advisory Committee on Reactor Safeguards was held in Oak Ridge at which an oral presentation of the hazards analysis of the EGCR was presented. A review is being made of the proposed research and development program for the EGCR. (AEC Activity 4110)

Reactor Physics. – Multigroup supercell calculations were performed to ascertain the power generated in various representative experiments installed in 5-in.-dia EGCR loops. Although the loops are not concentric with the reactor, the reactor power distribution in four directions from the loop axis (in both directions along a reactor diagonal and in both directions along a line passing through the next 5-in.-dia loop) was practically symmetrical about the loop axis, as determined by two-dimensional

diffusion-theory calculations of the reactor with the four 5-in.-dia loops. It was therefore thought reasonable to calculate the effect of various loop contents by using a cell calculation, with a flat-flux boundary condition about 15 in. from the loop axis. The loop and its contents were represented exactly, whereas the surrounding core material was treated as being homogeneous. Cases calculated were: (1) empty loop tube; (2) tube filled with graphite of density 1.15 g/cm^3; (3) graphite plus uranium; (4) graphite plus boron; (5) an EGCR fuel element, including sleeve, enriched to 10% U^{235}. For a carbon-to-uranium atom ratio of 200 (case 3) in all four loops, the positive reactivity contribution would be about 0.03, if a power of 1040 kw in each loop were allowable. However, if the experimental assembly were reduced in length to limit the power in each loop to 500 kw, the combined reactivity of the four loops would be 0.02.

The gamma-ray heating in a 5-in.-dia loop due to reactor core gamma rays and to gamma rays produced in loop experiments was calculated. The contribution from the core gamma rays in the reactor mid-plane, with an empty loop tube, is 0.32 w/cm^3. The average heat generation rate in the tube wall due to neighboring fuel channels is 0.23 w/cm^3, and the integrated power generation in the tube, over its full length, is about 1.7 kw.

For a calculation of heat generation in the tube due to an experimental fuel element inside the loop, the experimental element was arbitrarily taken to be an EGCR element producing twice the power of the hot elements in the reactor, that is, $2 \times 492 = 984$ kw. However, it is presently assumed that 500 kw will be the maximum power generation in the smaller loops, and the integrated gamma-ray heat in the tube was reduced accordingly. The calculated result is then 2.7 kw. Maximum heat rates in the tube are 0.23R (w/cm^3) due to core gamma rays and 0.73R (w/cm^3) due to gamma rays produced in the experimental element, where R is the ratio of peak to average power density in the axial direction. (AEC Activity 4110)

EGCR Hazards Evaluations. – A detailed calculation of the direct radiation at a distance of 1000 ft from the EGCR containment vessel following the maximum credible accident was made in which all fission products were considered. The calculation yielded a dose of ~3 r in 8 hr, which is to be compared with the value of 2 r in 8 hr found in the EGCR hazards report on the basis of four isotopes selected because of their contribution to the dose in previous studies. (AEC Activity 4110)

Tests of Coated Graphite. – Siliconized silicon carbide coatings on graphite-clad UO_2 fuel elements were tested alternately in a radiation field and in high-temperature air (1100°C). Two samples irradiated to a total dose of 6.4×10^{20} nvt remained intact after 467 hr at 1100°C in air. No weight changes occurred. (AEC Activity 4110)

Closed-Cycle Experiment in ORR. – A leak in the system has been located, and the system is being repaired. Test operation will be resumed when the repair work is completed. (AEC Activity 4110)

Out-of-Pile Component Tests. — A 1000-hr test of a full-scale model of an EGCR fuel assembly (seven-rod cluster complete with hangers and graphite sleeve) was completed in a forced-circulation loop. Argon was circulated in this loop at 1400°F, 30 psig, and a velocity of 300 fps. Argon was used to give mass flow rates equivalent to those for helium at reactor design pressure in a low-pressure facility. (AEC Activity 4110)

Mechanical Properties of Type 304 Stainless Steel. — Tube-burst tests of type 304 stainless steel have continued. One test at 1300°F and 500 psi resulted in a time to rupture of 1769 hr with an average strain of 1.4% and a maximum strain of 4.64%.

The reactions of type 304 stainless steel with carbon monoxide are being studied in the absence of stress. The amount of carbon absorbed is quite large (0.54%) for the exposure times involved (up to 200 hr), and, according to creep data presently available, will severely alter the properties of the material. The carbides formed have been identified as $Cr_{23}C_6$, and hence a carbon content of 0.5% will tie up about 20% of the chromium present in type 304 stainless steel. This can markedly alter the corrosion and mechanical properties of the material. (AEC Activity 4110)

SMALL-SIZE NUCLEAR POWER PLANT

The Oak Ridge Operations Office (AEC) was assigned responsibility for formulating and implementing a new program for the utilization and development of small-size (5 to 40 Mw electrical) nuclear power plants. The overall objective is to develop small-size reactors that can produce reasonably economic power at an early date in small publicly owned power systems in the United States and for specialized foreign applications. This program is a part of the AEC's over-all civilian nuclear power program for fiscal year 1960.

An ORNL Advisory Group was established in April 1959 to provide technical assistance in the accomplishment of this work. This group has worked closely with an OROO Task Force Committee in the selection and determination of the feasibility of a first-generation prototype reactor which can be constructed at an early date by using reasonably available technology. The initial effort of the ORNL Advisory Group was an evaluation of the suitability of several reactor types. Results and recommendations from this study were transmitted to OROO. (AEC Activity 4163)

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR-1). — Operation of the APPR-1 at Fort Belvoir, Virginia, was resumed early in June 1959 following an extended shutdown for core examination and measurements. The five boron-iron control-rod absorber sections were replaced by europium oxide—stainless steel sections. One control-rod fuel element, one stationary fuel element, and all reactor vessel studs were replaced.

Two boron-iron control-rod absorbers, one control-rod fuel element, and one stationary fuel element, which had operated approximately 10.5 Mw-years in the first core, were shipped to ORNL. These elements are currently being subjected to postirradiation examination in ORNL hot-cell facilities.

Twelve miniature europium oxide—stainless steel control-rod specimens were fabricated at ORNL, and approval was obtained for irradiating them in the beryllium reflector of the MTR. Specimens containing 20, 30, and 40 wt % europium oxide will be irradiated for periods corresponding to 1/2, 1, 2, and 3 core lives in APPR-1. (AEC Activity 4601)

GENERAL REACTOR RESEARCH

Basic Reactor Research. — The effect of the (n,2n) reaction of beryllium on the fast-neutron multiplication in a reactor was calculated. Preliminary to the calculation, a review of the available cross-section data was made, and upper and lower estimates of the (n,2n) cross section were obtained. The fast multiplication effect was computed by calculating the spectrum in an infinite homogeneous beryllium moderator, on the assumption that the source neutrons had a fission neutron distribution. The entire angular distribution of the elastically scattered neutrons was considered. The calculated upper limit of the net effect is 7.6% and the lower limit is 5.1%. Attention was also given to the poisoning effect created by the daughter products of the (n,α) reaction. At fluxes of about 5×10^{13} neutrons$\cdot$cm$^{-2}\cdot$sec^{-1} it lowers the reactivity gain approximately 5% after about two years.

The fission cross section of Np^{237} was measured as a function of neutron energy in the range from 0.9 to 8.0 Mev. The measurements above 1.7 Mev were made relative to the known U^{238} fission cross section. Thin deposits of Np^{237} and U^{238} on platinum backings were placed back to back in an ionization chamber, and fission fragments from each deposit were counted in a 2π geometry. The neutrons incident on the deposits were from the $H^3(p,n)He^3$ and $H^2(d,n)He^3$ reactions. The masses of the various neptunium and uranium deposits were determined in separate experiments by alpha counting. In the range from 0.9 to 2.9 Mev, a secondary neutron monitor was used and the results were normalized to those of the comparison counting experiment in the overlapping energy range. The results indicate that the cross section increases above threshold to a maximum value of 1.7 barns at $\sim$2 Mev, decreases to 1.36 barns at $\sim$5 Mev, and increases again to 2.08 barns at $\sim$8 Mev. The estimated uncertainty in the absolute magnitude of the fission cross section of neptunium is ±8%. These results differ somewhat in both magnitude and shape from those in the second edition of BNL-325. (AEC Activity 4202)

Basic Shielding Research. – An experiment designed to determine the importance of the different materials required in the production of barytes concrete is currently in progress at the Lid Tank Shielding Facility. Measurements of fast-neutron and gamma-ray dose rates and thermal-neutron fluxes have been made behind 16, 32, and 48 in. of barytes aggregate and behind 8, 16, 24, 32, 40, and 48 in. of a mixture of barytes aggregate and portland cement, one part of cement to eight parts of the aggregate by volume. Preliminary analysis shows that there is essentially no change in the fast-neutron dose rate in the two cases; however, both the gamma-ray dose rate and the thermal-neutron flux appear to decrease somewhat more slowly in the case of the aggregate-cement mixture than in the aggregate alone. (AEC Activity 4202)

Power Reactor Fuel Reprocessing: BORAX IV Fuel. – The presently preferred method for processing BORAX IV fuel is removal of the aluminum cladding with boiling 2 M $NaOH$–1.78 M $NaNO_3$, dissolution of the lead bond in 1 to 2 M HNO_3, and dissolution of the sintered ThO_2-U_3O_8 core in boiling 13 M HNO_3–0.04 M NaF–0.1 M $Al(NO_3)_3$. The initial rate of dissolution of lead foil increased from 3.3 to 15.9 $mg \cdot min^{-1} \cdot cm^{-2}$ as the nitric acid concentration was increased from 0.5 to 2.0 M.

Decladding with mercury-catalyzed nitric acid was unsatisfactory because a mercury concentration of 0.5 M is required to assure an adequate dissolution rate. This is about ten times the concentration normally required for dissolution of aluminum. In 8 M HNO_3–0.05 M $Hg(NO_3)_2$, titanium corroded at a maximum rate of 0.8 mil/month in 456 hr of exposure. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Darex Process. – The Darex reference flowsheet was used successfully on APPR and Yankee Atomic prototype fuel solutions to remove chloride to 70 ppm. The presence of titanium had no adverse effect. An average over-all heat transfer coefficient of 250 $Btu \cdot hr^{-1} \cdot ft^{-2} \cdot (°F)^{-1}$ was determined for the system consisting of condensing steam, 0.090-in. titanium, and Darex solution in a Darex still having a capacity of 0.3 kg of U^{235} per day.

Approximately 40% of the silica remaining after Darex dissolution of APPR fuel had a particle size larger than 13 μ; it was removed by medium-size fritted glass but not by 30-mesh sand. The remainder of the silica passed through fine fritted glass. Addition of 0.4 g of gelatin per liter of dissolver solution at 60°C permitted removal of 50% of the silica by medium-fritted glass immediately and an additional 10% after 24 hr. Addition of 0.005 M HF during dissolution and two days standing permitted 100% removal of the silica by medium-fritted-glass filtration; 60% was removed when the solution was filtered immediately. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Mechanical Processing. — In tests to find a satisfactory method of disposing of the NaK released in SRE fuel decladding, the reaction of NaK with steam was evaluated. The 98.4 ml contained in one fuel rod produced 1 liter of hydrogen per minute in reaction with steam at 0.4 lb/hr. Reaction was complete in 35 min.

In shearing-leaching tests the percent (P) of powdered UO_2 produced in shearing 0.4-in.-dia UO_2-filled stainless steel tubes was related to the length of cut (L) by $P = 13/L^{1.2}$ for 0.5- to 5-in. lengths. In leaching tests it was demonstrated that stainless steel particles up to 1000 μ in diameter can be satisfactorily jetted from a batch dissolver. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Graphite-Containing Fuel. — By a grind-leach process, more than 99.9% of the uranium was recovered from specimens containing 5.5, 9.7, and 14.0 wt % uranium in graphite. The samples were ground to -200 mesh and leached twice for 4 hr with boiling 15.8 M HNO_3, and the residue was washed thoroughly with cold water. The iron content of these specimens varied from 0.07 to 0.4%. Only 99.7% of the uranium was recovered from a fuel containing 11.2 wt % uranium and 0.8% iron.

Soxhlet extraction of plates of the General Atomic 1.5% uranium-7.1% thorium-graphite fuel with azeotropic nitric acid offered no advantages over a single leach with boiling 15.8 M HNO_3, in which 10% of the uranium and thorium was insoluble. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Perflex Process. — In the Perflex process, U-Zr alloy fuels are dissolved in aqueous HF-H_2O_2. In corrosion studies with Monel and INOR-8, neither was sufficiently corrosion-resistant for use as a process vessel. In 1 M HF-0.06 M H_2O_2, Monel corroded at a maximum rate of 157 mils/month in 4 hr; the rate was decreased to 57 mils/month by contact with dissolving zirconium and blanketing with nitrogen. In 1 M HF-0.06 M H_2O_2, INOR-8 corroded at a maximum rate of 47 mils/month in 24 hr. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Sulfex Process. — In tests on depassivation of the cladding of prototype Yankee Atomic fuel specimens (stainless-steel-clad UO_2), iron wire in contact with the passive cladding surface consistently initiated complete dissolution of the cladding in boiling 6 M H_2SO_4. The passive film was also destroyed by 15-min exposure of the pins to concentrated chromous sulfate at 85°C. Soluble uranium and plutonium losses during the 30 min required for the decladding of irradiated Yankee fuel specimens with boiling spent 6 M H_2SO_4 were a maximum of 0.05% for fuel irradiated to 1440 Mwd per ton of uranium. However, uranium and plutonium losses were as high as 20% when irradiated core material was exposed to boiling fresh decladding solution, in the presence of oxygen, for 24 hr.

The rate of dissolution of sintered 95.5% ThO_2-U_3O_8 pellets (Consolidated Edison fuel) in a 200% excess of boiling 13 M HNO_3-0.04 M NaF-0.1 M $Al(NO_3)_3$ decreased from 50% in 1 hr with no sulfate present to 1% in 1 hr when the sulfate concentration was 0.3 M.

Radiation-induced uranium losses from UO_2-ThO_2 in 6 M H_2SO_4 were less than 0.3% in 3 hr. The percentage increase in losses was inversely dependent on temperature, with 70% increase in loss at 35°C but only 40% increase at 75°C. In a single run in boiling 6 M H_2SO_4 with a crushed reactor-irradiated pellet, uranium losses were about 3% after 3 hr. Much acid-insoluble thorium sulfate was formed during this run.

The presence of boric acid, a possible neutron poison, did not affect the dissolution time of ThO_2-UO_2 pellets in Thorex dissolvent (13 M HNO_3, 0.1 M Al, 0.04 M F). The solubility of boric acid in Thorex dissolvent increased from 0.26 M at 23°C to 1.18 M at 85°C. Thorex dissolvent was steam-stripped to acid deficiency at 120°C. Without steam-stripping the solutions must be heated above 150°C to remove the free acid. The corrosion rate of Ni-o-nel was 2.3 mils/month after 15 Sulfex-Thorex cyclic dissolutions. (AEC Activity 4301)

Waste Disposal Research and Engineering: Disposal of ORNL Radioactive Wastes. — Cesium sorption curves obtained for sodium vermiculite treated with potassium to yield vermiculite with less than 100% potassium saturation indicate that the sorption of cesium is reduced by an amount equivalent to the amount of potassium previously sorbed. This is regarded as further evidence that cesium and potassium occupy similar sites between the lattice layers. The sorption of cesium and potassium is not easily reversible, resulting in an apparent reduction of the interlayer charge as measured by the calcium exchange capacity. Both potassium and cesium cause a structural collapse of the vermiculite lattice to a crystal lattice similar to that of illite. This collapse is accompanied by an extreme increase in the affinity of the vermiculite for cesium. These results are further indication that structure plays a primary role in the sorption of cesium by naturally occurring clay minerals.

The second of five core holes 200 ft deep was completed in Bear Creek valley. Pressure tests on well No. 1 indicate that below a depth of 164 ft the rock penetrated is essentially impermeable. The rock above this point contained numerous cavities up to 0.3 ft thick. To determine whether the cavities encountered in the well were hydraulically connected with Bear Creek, fluorescein dye was injected into the well under pressure. The dye was detected in the creek at points about 300 and 1200 ft from the well. (AEC Activity 4352)

Waste Disposal Research and Engineering: Corrosion Studies. — Tests of Haynes alloy No. 25, welded at ORNL, in salt-saturated acid Purex waste resulted in severe intergranular corrosion in the base metal adjacent to the welds. Metallographic examinations showed that severe carbide precipitation was present and that all the corrosion had taken place along the grain boundaries in the affected area.

Consequently, a revision was made in the welding procedure for fabrication of the Haynes alloy No. 25 covers to be used for sealing the cavities for the field-scale experiment in the salt mine at Hutchinson, Kansas. The revised procedure calls for (1) a single pass for each weld, (2) the use of coated rod, and (3) quenching of the weld with dry ice as quickly as possible. This restricts the time during which the base metal is at welding temperatures to an absolute minimum. Corrosion tests to evaluate welds made under these conditions are in progress. (AEC Activity 4352)

Waste Disposal Research and Engineering: Disposal into Natural Salt Formations. – Invitations to bid on the preparation of 6000 gal of synthetic Purex waste for the field experiments in the Hutchinson mine were submitted to interested vendors. The bid of the Associated Sales and Supply Company, St. Louis, Missouri, was accepted, and delivery of the waste at the mine is expected on or about July 1, 1959.

Two one-fifth-scale models of the field test cavities have been excavated in pure salt in the Hutchinson mine, and a third one-fifth-scale model has been excavated in the floor of the mine. These models will be used to check the thermal calculations. The two large cavities have been filled with brine, and no significant leakage has been observed. All the test equipment is scheduled for completion on July 1, 1959. Final installation will begin upon delivery of the metal covers, power supplies, and instruments. (AEC Activity 4352)

Waste Disposal Research and Engineering: Hazards Evaluation. – The concentrations of radioactive material at the Benjamin Franklin Bridge that were based upon the model studies of an intermittent release opposite the Philadelphia Naval Base were compared with the results of model studies of a continuous release opposite the New York Shipbuilding Yard. The concentrations from the release at the Naval Base were transposed, and the theory of superposition was used to determine the effects of a continuous release opposite the New York Shipbuilding Yard. The results were within 25% of those found in the model studies for low flow and within a factor of 2.5 for the high flows.

A comparison of the geologic structure of the tank farm site in the Ohio River valley with similar areas for which permeability data are available indicates that the rate of movement of ground water in the till is probably less than 1 ft/day. However, in the underlying limestone, movement rates of the order of 1 fpm are expected. This high rate occurs in channels in the bedrock and is evidenced by the presence of numerous sinkholes, springs, and caves in the immediate area of the site.

Between the mouth of the Ohio River and the tank farm site there are approximately 35 towns with a total population of 740,000. Of this number, approximately 630,000 depend upon surface sources for their water needs, while the remainder utilize community wells, private wells, and springs. An additional 54,000 inhabitants utilize other surface sources that drain the general area of the site. (AEC Activity 4352)

THERMONUCLEAR PROJECT

DCX-1. – Reliable operation of DCX-1 was seen during the month, with observations that have been most illuminating in that a dominating influence of the carbon arc upon the trapped plasma has been revealed. When the anode of the arc and the anode baffle through which the arc passes were moved close to the volume occupied by the trapped 300-kev protons, high efficiencies for the dissociation of molecular ions were achieved (40% vs 3 to 8% in the previous arc configuration) – which was the objective of the change. However, the mean residence time of the trapped ions turned out to be low (1 to 2 msec at 10^{-6} mm Hg ambient pressure). When the arc was off and the residual gas was used for dissociation of the molecular ions, better containment was achieved (10 msec at 10^{-6} mm). Apparently, under these particular electrode conditions, the arc was loading so much carbon into the containment volume that it became a major loss factor for the protons – worse, in fact, than the residual gas. The mechanism was clarified when a detector was placed in a position appropriate for the registration of protons that become neutralized in one of their many passages through the arc. This detector accounted for most of the particles that were being injected into the trapping volume. In fact, for the first time a particle balance has been found at low pressures between input and output – a reassuring fact in that large losses from the ends need not now be assumed in order to account for all of the input trapping rate.

This loss rate by neutralization in the arc is not in contradiction with old measurements, which set a limit of about one charge exchange per 10^5 passages through the arc. The observed rate of neutralization agrees approximately with that limit.

The measures to be taken to remove this loss mechanism are clear:

1. The arc should be run under conditions that minimize charge exchange. This may mean that a dominating population of C^{+++} and C^{4+} ions should be sought, or that an admixture of H_2 into the carbon arc should be attempted. (There are no electrons on H ions.) A sacrifice in molecular ion dissociation efficiency may be involved, but there is still reserve beam available in the injection system.

2. The orbit geometry should be changed slightly so as to introduce precession; the trapped protons would then miss the arc in most of their revolutions. This alteration has long been contemplated, but now there is a compelling experimental reason for making it.

The arc geometry has now been changed from the conditions that were so revealingly bad. Withdrawal of the anode and baffles has restored the best previous conditions; the arc neutralization loss has been reduced by a factor of 3 (although it is still large), and the mean residence time has been increased to 5 msec at 10^{-6} mm Hg. The dissociation efficiency, however, has gone down.

One promising feature of the situation should be noted. With gas breakup (arc off) the mean residence time was 10 msec at 10^{-6} mm Hg, and if the arc losses could be made negligible, this figure might be reached with the arc on. Charge exchange in the residual gas would then be the dominating loss mechanism, so that if the pressure were reduced to operating conditions already reached (2×10^{-7} mm Hg), then a mean residence lifetime of 50 msec should be attained. Such a figure would be most interesting from the point of view of burnout. (AEC Activity 4851)

Carbon Arc Studies. – Three significant things have been learned about carbon arcs:

1. The lowest pressure at which the arc had been run in any of the several experimental tanks in use had so far been 2×10^{-7} mm Hg. The question whether this indeed sets a lower limit to the ambient pressure, and hence to the usefulness of the arc, has now been answered. An arc has been run at an ambient pressure of 5×10^{-8} mm Hg, and there is no indication that a limiting low pressure has been reached.

2. The breakup mechanism of H_2^+ ions in the carbon arc is not connected with radiation in the arc. Dissociations apparently are caused by collisions with the positive ions.

3. There is a population of C^+, C^{++}, and C^{+++} ions fringing the luminous column of the arc. The population falls off with radial distance, in the case of C^+ to about 1% of the central density at a radial distance of 1 in. in a magnetic field of 4000 gauss. The fringing population shrinks in extent with increasing magnetic field strength. (AEC Activity 4851)

PROGRAM 5000 — PHYSICAL RESEARCH

ISOTOPE PRODUCTION

Radioisotope and Stable Isotope Production. — Thirty thousand curies of Cs^{137} was separated and 43 sources were fabricated.

The isotopes of mercury and strontium have been processed through the calutrons, and those of neodymium, nickel, and copper are now being processed. Through the use of a mixture of nickel chloride and metallic copper as feed material, a simultaneous separation of the isotopes of nickel and copper is being accomplished. Ion currents equivalent to a total of 192 g have been received during this reporting period.

Charge materials of strontium metal, mercury sulfide, neodymium chloride (fused lumps), mixed lithium metal and lithium chloride, and mixed nickel chloride and copper metal were prepared and supplied for isotope separations.

Three hundred twenty grams of potassium chloride, slightly enriched in K^{40}, was recovered from radioactive waste solutions and purified and is being held for future charge material.

Ten new lots and three returned lots of isotopes were refined chemically. In addition, 29 samples of strontium, mercury, calcium, and neodymium were recovered from pockets, homogenized, and partially purified to obtain mass assay data.

New lots of Ca^{46}, Ca^{48}, Cu^{63}, Cu^{65}, Gd^{154}, Gd^{155}, Gd^{156}, Gd^{157}, Gd^{158}, Rb^{85}, and Ru^{101} and returned lots of Ba^{137}, Cu^{65}, Eu^{151}, Eu^{153}, Nd^{143}, Nd^{145}, Pb^{204}, Se^{74}, and Si^{30} were added to inventory.

Five isotopic samples of silicon that had been used by Wright Air Development Center were further refined to remove a very small (<0.1%) sodium impurity.

The special services laboratory performed 28 conversions and prepared 22 targets. Other work included the purification of two lithium samples by distillation and the development of a method for the preparation of pure, ductile titanium metal.

Radioisotope Research and Development. — Test runs were completed in the equipment for the separation of Ce^{144} and Y^{91} by batch extraction, which was installed in cell 9 of the Fission Product Pilot Plant. Three full-scale runs were made in which rare-earth cuts obtained from BNL slug processing waste were used. Although no Y^{91} was present in this aged waste, the process functioned as required in the removal of Ce^{144}. Approximately 98.6% of the Ce^{144} was removed in one solvent extraction step with a loss of only 1% of the Pm^{147}.

Processing of waste solution resulting from BNL slugs which were aged about three years is in progress in the Fission Product Pilot Plant. The recovery of Sr^{90} from this Redox-type waste was found to be very poor (~25%) on the first main-stream precipitation step (a magnetite precipitate brought down at pH 3.0). The recovery was improved to 80% on this step by adding an additional small amount of iron to the solution after the magnetite had precipitated and precipitating hydrated ferric oxide, on which strontium is carried more efficiently.

The installation of equipment for the continuous countercurrent mixer-settler separation of rare earths is 70% complete. Tests and required modifications of the mixer-settler units are complete.

Design is in progress on purification equipment for Tc^{99}, a purification cell for Sr^{90}, and revisions to Building 3026C cells.

A second full-scale sample of sulfur was irradiated in the ORR and processed in the regular P^{32} production equipment. Twenty-five curies of P^{32} product (as of one week following discharge) resulted and met normal product specifications. Additional analyses for aluminum are in progress. (AEC Activity 5100)

Stable Isotope Development. — The focal patterns produced by various grid-type electrodes were determined. Parallel beams were not produced by any arrangement investigated.

An analysis of the separation data accumulated during the processing of mercury isotopes showed that background ions were the major source of product contamination. In addition to component refrigeration, the attainment of a higher vacuum would be desirable in future separations of this element.

A small laboratory-type facility has been set up and is being used in an isotope retention program. In this work the sputtering angle and distribution of both the target species and the ionic species are being measured. (AEC Activity 5100)

PHYSICS

High-Voltage Experimental Program. — Polarization-direction measurements have been made on the gamma-ray transitions in Pb^{207} resulting from the decay of Bi^{207}. These measurements were made in order to obtain additional information on both the spin of the level at 2.34 Mev and the multipole character of the decay gamma ray. The observed polarization eliminates the possible spin assignment 9/2. It is concluded that the spin assignment is 7/2 and that the 1.77-Mev gamma ray is predominantly M1.

Neon-20 ions have been accelerated to energies in the range 5 to 11 Mev with the 5.5-Mv Van de Graaff. These projectiles were used to investigate Coulomb excitation in light nuclei. The following states

were observed to be excited: Li^7, 478 kev; F^{19}, 110 and 197 kev; Na^{23}, 440 kev; Ti^{47}, 159 kev; V^{51}, 323 kev; Mn^{55}, 128 kev; Fe^{57}, 136 kev; and Ta^{181}, 136 kev. Values for the reduced electromagnetic E2 transition rates were obtained which in general have an accuracy of ±20%. The largest source of uncertainty in these absolute measurements is the stopping power of the various target materials for Ne^{20} ions. (AEC Activity 5220)

The 86-Inch Cyclotron: Nuclear Physics. — Bombardment of enriched Hf^{174} with protons produced Ta^{173}, which decays by electron capture to Hf^{173} with a half life of 2.5 hr. The most intense transitions observed with internal-conversion spectrographs form an anomalous rotational band based on the predicted ground-state spin $I_0 = 1/2$. Rotational levels are present at 69.8 kev $(I_0 + 1)$, 81.5 kev $(I_0 + 2)$, 241.9 kev $(I_0 + 3)$, and 262 kev $(I_0 + 4)$. The low-level structure of Hf^{173} is analogous to that of Yb^{171}, both of which are nuclides with odd A and odd N (N=101). It is apparent that the behavior of the odd neutron in the low energy region is not changed by the addition of two protons. (AEC Activity 5220)

The 86-Inch Cyclotron: Applied Physics. — The isotopes Co^{56}, Co^{57}, As^{74}, Sr^{85}, $Y^{87,87m}$, Dy^{159}, and $Bi^{205,206}$ were produced in service irradiations for outside customers, and Tc^{95m} was produced for the ORNL Chemistry Division. A 3 × 4 × 0.050 in. iron sheet, soft-soldered to a water-cooled copper base, was operated at a current of 1000 μa for the Co^{56} production.

Two test targets of rhodium sheet 3 × 1 5/8 × 0.003 in. were bombarded for the production of Pd^{103}. One was soft-soldered to the copper base and the other silver-soldered. The soft-soldered target melted at 900 μa and became inoperable. With the silver-soldered target evidences of melting were encountered at 1100 μa, but when the cyclotron was adjusted to a slightly off-resonance condition (altering the beam distribution), satisfactory operation was continued at 1500 μa. (AEC Activity 5220)

Heavy-Particle Physics. — Preliminary data on the elastic scattering of nitrogen from aluminum, taken from 50 to 124° in the center-of-mass system, show a smooth angular distribution dropping rapidly from $0.7\sigma_{Coulomb}$ at the smallest angle to $0.05\sigma_{Coulomb}$ at 124°. Since the coincidence technique is not useful at smaller angles, other techniques are being investigated for extending the measurements to small angles.

A search is in progress for the radiative capture of nitrogen by phosphorus nuclei. The reaction $P^{31}(N^{14}, \gamma)Ti^{45}$ is identified by chemical separation of the radioactive Ti^{45}. Preliminary results indicate that the cross section may be as large as 20 microbarns.

In an investigation of the inelastic scattering of nitrogen from carbon, scattering to the 3.95-Mev state in nitrogen and to the 4.43-Mev state in carbon was established. A closer examination of these reactions awaits the perfection of detectors having a higher resolution. (AEC Activity 5220)

Relativistic Isochronous Cyclotron. — The preparation of the engineering drawings and specifications for the ORIC building and facilities by the Catalytic Construction Company is now in the title-II phase.

The full-scale ORIC magnet is being installed temporarily in the Y-12 area in order to obtain more precise magnetic field measurements. The steel yoke, weighing 200 tons, is now in place. One main coil was wound, and the second coil is being wound in the Y-12 shops. Some faults were found at the center of the 1070-ft lengths of hollow aluminum conductors, where two ingots had been joined in the extrusion process. It was necessary to remove short lengths of the conductor in the extrusion-weld region. A Heliarc welding technique was developed which satisfactorily corrects the difficulty.

Installation of a new quarter-scale model magnet is complete; it is being test-operated. It will provide accurate magnetic field data to assist in the detailed design of the auxiliary coils for the cyclotron. A test resonator, to be used in design studies for the shorting bar (spider), was completed and is being tested. Measurements on a quarter-scale r-f model with a single 180° dee were completed; this system has now been selected for ORIC, and a full-scale model will be designed for more exact measurements. (AEC Activity 5240)

Special Separations. — Two U^{234} collections yielded a recovered weight of 168 mg of U234, which assayed 97.53%. Two additional runs yielded 96.8 mg of U^{234}, which presently is being assayed.

The separation of U^{238} to provide enriched charge material for future second-pass operations has produced approximately 725 g in 11 runs.

Three plutonium runs were completed and ion currents equivalent to 1.65 g of Pu^{239}, 722 mg of Pu^{240}, and 85 mg of Pu^{241} were collected.

Two U^{234} charges were prepared from an initial 28-g feed batch by chemical recycling.

Approximately 10 kg of UCl_4 was provided as the charge for U^{238} separation. Recycle of this material has progressed into extraction and peroxide purification. (AEC Activity 5250)

CHEMISTRY

Chemical Engineering Research. — The apparent integral diffusivity of uranyl nitrate (50 g of uranium per liter) from aqueous solution to 30% tributyl phosphate in Amsco was 6.7×10^{-7} cm^2/sec. When the organic phase contained 10 ppm of detergent (Tide), the diffusivity dropped to 4.4×10^{-7} cm^2/sec, indicating some blocking of the interface by the detergent. The experiments were made by allowing the aqueous uranyl nitrate solution to diffuse from a capillary tube (30 mm^3) into a large volume (300 cc) of the organic phase. (AEC Activity 5310)

Americium Recovery. — Approximately 9.5 g of Am^{241} was recovered from various waste solutions containing large amounts of Ca, La, Fe, Cr, and Ni and smaller amounts of other impurities. After purification by oxalate precipitation followed by citrate elution from cation exchange resin, the americium product contained less than 1% impurities. (AEC Activity 5310)

Transuranics. — Investigations were continued on the use of mono-2-ethylhexylphosphoric acid in xylene diluent as a solvent for the separation of americium and curium from rare-earth fission products in a chloride system. Distribution coefficients (organic to aqueous) between 0.5 M mono-2-ethylhexylphosphoric acid and 12 N HCl are 0.12 for cerium and 0.025 for americium, giving a separation factor for cerium of 5. The separation factor increased with increasing atomic weight of the rare earths, being, for example, 7 for neodymium, 8.5 for promethium, and 10 for europium. At constant chloride concentration the distribution coefficients can be increased by replacing the hydrogen ion with lithium. With 11 M LiCl—1 M HCl, the distribution coefficients were 0.55 for cerium and 0.11 for americium. Over this range of conditions the cerium-americium separation factor was approximately constant. As the remaining hydrogen ion is replaced by lithium, the distribution coefficients continue to increase; however, the cerium-americium separation factor decreases to 3 with 12 M LiCl. These results were obtained with tracer americium and rare earths. With macro amounts the distribution coefficients were approximately proportional to the inverse cube root of the rare-earth concentration. (AEC Activity 5310)

Feed Materials Processing. — Studies leading to the development of a simple, quickly applied test for predicting the surface area and sinterability of UO_2 powders were continued. The amount of methylene blue adsorbed by a UO_2 sample was shown to be directly proportional to the total surface area when adsorbed from a solution buffered at pH 8 by KH_2PO_4-NaOH and containing 0.001 M $Na_2P_4O_7$ and 0.16 g of methylene blue per liter. This property was also shown to be a linear function of the sintered densities of six samples of ceramic-grade UO_2 powders.

The sinterability of UO_2 powders was shown to increase with increase in the percentage of particles less than 1 μ in size. Powders with more than 22% of the particles below 1 μ sintered to more than 93% of theoretical density.

In a study of the precipitation of uranyl peroxide, rounded aggregates of hydrated UO_4 were precipitated from 0.15 M uranyl nitrate or fluoride solution at 90 to 95°C by 2 M H_2O_2—0.2 M HNO_3 solution.

Fast-settling and -filtering, free-flowing precipitates of ammonium diuranate were produced from 0.15 M uranyl nitrate solution at 95°C by the slow addition of 7 M NH_4OH.

In attempts to increase the thermal conductivity of UO_2, nickel phosphide was plated onto UO_2 pellets and powder from electroless nickel plating solution. (AEC Activity 5310)

Equipment Decontamination. — A dilute solution of a mixture of tri- and tetravalent uranium in dilute sulfuric acid was shown to be an effective reagent for removing scale deposited on stainless steel by uranyl sulfate solutions. Specimens of HRT pipe were descaled and decontaminated from fission product gamma activity by a factor of 10^3 by a two-stage treatment consisting in 90 min at room temperature in this reagent and 5 min in warm (50°C) 4 M HNO_3–3% hydrogen peroxide. The descaling was by dissolution of the scale, not by attack on the metal beneath the scale and loosening of large particles of scale. Solutions containing only tetravalent uranium were not effective; reduction of uranium to the trivalent state by zinc in sulfuric acid was insignificant at pH 2.0 but proceeded readily at pH 1.3.

Preliminary studies indicated that the corrosiveness of chromous sulfate–sulfuric acid solutions at 85°C is not solely a function of sulfuric acid concentration but is affected by the total ionic concentration and the proportion of chromous ion to sulfuric acid.

The descaling of large equipment by chromous sulfate may not be as successful as that of small pieces. A section of pipe from corrosion loop E, which had operated with various concentrations of uranium solutions for several thousand hours, the final run being with uranyl perchlorate, was not descaled when treated with chromous sulfate solution. This piece of pipe was about 100 times the size of previous test specimens. (AEC Activity 5310)

Chemical Physics. — The relative yields of atomic hydrogen were measured in various concentrations of sulfuric, perchloric, and phosphoric acids after cooling the acids to liquid-nitrogen temperature and irradiating with gamma rays from Co^{60}. The paramagnetic resonance method was used. After the irradiated acids were warmed, the molecular hydrogen resulting from the irradiation was collected and assayed. A striking correspondence was found between the relative atom yields and final molecular yields. An absolute assay was completed on 0.129 mole fraction sulfuric acid (the reference material for the relative atomic hydrogen assays), and the yield of atomic hydrogen was found to be about twice the molecule yield (0.62 molécule of H_2 per 100 ev absorbed). This demonstrates essentially a direct correspondence between the yields of atomic hydrogen (the intermediate) and molecular hydrogen (the final chemical product). (AEC Activity 5330)

Analytical Chemistry Research. — A gas chromatographic procedure has been established for the analysis of the gases that are evolved when graphite is heated. These gases contain N_2, O_2, H_2, CO, CO_2, CH_4, complex mixtures of higher hydrocarbons, traces of the inert gases, and water vapor. The gas sample is taken at pressures as low as 100 μ and compressed in a 1-cm^3 vessel to approximately atmospheric pressure. It is then carried by a stream of helium into a chromatographic column packed with Linde 5A molecular sieve, which separates H_2, O_2, N_2, CO, and CH_4. The concentration of each eluted constituent is determined by comparing its conductivity with

that of the carrier gas; the coefficient of variation is about 2%. Samples having volumes of 20 to 500 μl can be analyzed by this method.

Changes that have been made in the x-ray fluorescence unit include an increase of voltage to 75 kv, whereby the excitation of more elements is possible. The unit is being used for the analysis of alloys of niobium with rare earths and with zirconium and for the nondestructive identification of small amounts of precipitates. A new timer-scaler was installed on the x-ray absorption apparatus, which reduces the counting time on samples of low concentration by a factor of 6. In addition, a cell was designed for the determination of americium and other alpha emitters by x-ray absorption. For the determination of zirconium, the method of x-ray absorption has been found, so far, to be accurate within 1 to 3% over the concentration range 0.05 to 15 mg/ml, regardless of the presence of impurities.

METALLURGY AND SOLID STATE PHYSICS

Ceramics Research. — The oxides of all the lanthanide elements except promethium, most of them 99% pure, in the form of dry-pressed 1/8-in. cylinders were fired in oxygen and also in hydrogen at 1300, 1500, and 1600°C. The resulting densities were compared with theoretical. In oxygen, all the oxides showed fair or poor sinterability at 1600°C except Sm_2O_3 (good sinterability at 1300°C) and Tm_2O_3 and Lu_2O_3 (good sinterability at 1600°C). Similar treatment in a hydrogen atmosphere increased the refractoriness of Lu_2O_3 considerably; the behavior of the other oxides was not affected notably.

X-ray lattice parameters were obtained for the 13 oxides. (AEC Activity 5420)

Fundamental Investigations of Radiation Damage in Solids: Effects in Nonmetallic Materials. — Several systems of electron spin resonance lines have been observed in gamma-irradiated natural and synthetic quartz crystals. The correlation of one system of lines with an optical absorption band at 2300 Å has been established. The correlation was made by observations on irradiated crystals of differing origin and by optical bleaching experiments carried out at 78°K. The system is composed of two lines when the c axis is parallel to the magnetic field and six lines when the [100] orientation is parallel to the field. Estimates of the eigenvalues of the g tensor are $g_1 \approx 2.0004$, $g_2 \approx ?$, $g_3 \approx 2.0020$. This ESR system and the 2300-Å band have also been observed in neutron-irradiated crystals and grow with increasing irradiation; thus impurities are not the source of the magnetic defect. The anisotropy of this ESR system and the magnitude of the g value suggest that this defect may be an unsaturated silicon bond which can trap an electron. (AEC Activity 5420)

Fundamental Investigations of Radiation Damage in Solids: Radiation Effects on Electronic Components. — The thermoelectric power of a single-crystal n-type germanium sample was measured as a function of temperature

and temperature differential before and after exposure to 9.6×10^{13} fast neutrons/cm^2. Measurements of the Hall coefficient, made on a plate from the same ingot which was exposed simultaneously, indicated that the carrier concentration at liquid-nitrogen temperature changed from 1.09×10^{15} to 0.914×10^{15} cm^{-3}. After the irradiation, the thermoelectric power was found to have decreased for all points measured and to vary linearly with temperature in the extrinsic region. The Seebeck coefficient is found to be a function of temperature and temperature differential. The dependence on temperature differential changed on irradiation to a $1/\Delta T$ behavior. (AEC Activity 5420)

PROGRAM 6000 — BIOLOGY AND MEDICINE

BIOPHYSICS

Radiation Dosimetry Programs. — An electromagnetic wave having its wave vector parallel to the direction of the steady magnetic field H dissipates its energy in plasma very effectively when the frequency ω is equal to the ionic resonance frequency Ω_g of the plasma. A small localized periodic disturbance initiated in a plasma at $t = -\infty$ and having a frequency $\omega = \Omega_g$ produces, along the direction of the magnetic field, a wave motion characterized by a complex wave vector

$$k \sim (\Omega_o \, \Omega_g / c^2 u_{oi})^{1/3} (\sqrt{3} + i),$$

where Ω_o is the Langmuir frequency and u_{oi} is the mean thermal velocity of the ions in the plasma. The energy absorption per wavelength at the resonance frequency is substantial, since $\mathrm{Im}\, k/\mathrm{Re}\, k = 1/\sqrt{3}$. However, the energy absorption per unit of length is not maximum at ionic resonance, since it increases for increasing frequencies when ω passes through the resonance. This is shown by the fact that $\mathrm{Im}(dk/d\omega) > 0$ for $\omega = \Omega_g$, since at resonance $\mathrm{Im}(dk/d\omega) = K_1/u_{oi} + (K_2/u_o^2 u_{io})^{1/3}$, where K_1 and K_2 are appropriate positive constants and u_o is the velocity of the magnetohydrodynamic wave. (AEC Activity 6140)

Ecological Studies. — The radiation above the contaminated soil of the bed of White Oak Lake has been characterized in terms of the horizontal and vertical distribution of the dose rates in air over most of the bed. The surveys disclosed that the radiation field was predominantly (90%) gamma and was nonuniform over the lake bed, ranging from less than 1 mr/hr to a maximum of 100 mr/hr.

The equation

$$D = \Sigma \frac{2\pi}{\mu_i} Nn_i G_i \left[1 - E_2(\mu_i h_i) \right],$$

where $i = 1, 2, 3, \ldots$, determines the expected dose rate D above a large slab source of thickness h_i in which radionuclides i of concentrations Nn_i are uniformly distributed; μ_i is the coefficient of absorption in the slab source, G_i is the dose rate in air at 1 cm from a point source of nuclide i, and E_2 is the E function of second order. When this equation was applied to a section of the lake bed, the solution, after scattered radiation was considered, gave a theoretical value of 18 mr/hr, which compared favorably with the measured mean value of 16 mr/hr. (AEC Activity 6480)

PROGRAM 8000 — WORK FOR OTHERS

Professional Health Physics Training Program. — The AEC Radiological Physics Fellows completed their work at Vanderbilt University. Seven of the group went to the National Reactor Testing Site for their summer training and one to Brookhaven National Laboratory. The remainder, with the addition of a Fellow from the University of Rochester and three Air Force officers, making a total of 30, are in training at ORNL. (AEC Activity 8780)

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

June 1959

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