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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

FEBRUARY 1959

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

February 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

Metal Recovery: Thorex. – Modifications of the power reactor fuel reprocessing facilities in Buildings 3505 and 3019 were completed, and the processing of irradiated fuels was started on January 8, 1959. During this report period six tons of NRX-irradiated uranium and five NRX plutonium-aluminum-alloy fuel assemblies containing 1.2 kg of plutonium were processed.

The average dissolution rate of the alloy was in excess of 10 $mg \cdot min^{-1} \cdot cm^{-2}$ in 6 M HNO_3 catalyzed with mercuric nitrate; the final dissolver solution was 0.5 M in aluminum nitrate and 4.0 M in nitric acid. A solvent extraction flowsheet modified from the Purex second plutonium cycle was used for recovery; the gamma-beta decontamination factor was 10^3 (limited by the low activity level of the feed solution). The Building 3019 (Thorex) plant was used for first-cycle operations; final purification was accomplished in the Metal Recovery Plant. (AEC Activity 2351)

Chemical Plant Criticality Studies. – Experiments are in progress to determine the nuclear properties of arrays of 0.94% U^{235}-enriched uranium metal slugs in water. Each slug is a 6-in.-long hollow cylinder, having an outside diameter of 1.37 in. and an inside diameter of 0.49 in. There is no cladding on the slugs.

In one series of tests to evaluate the nuclear safety of a slug dissolver, 7174 slugs (160 kg of U^{235}, 18.8 tons of uranium) were randomly placed in a cylindrical vessel 52 in. in diameter. This randomness was achieved by dumping the slugs from a height of more than 5 ft and resulted in a water-to-metal volume ratio of 1.06 ($H:U^{235} = 156$) which was reproducible in several such operations to within 5%. The height of the array was 53 in. Only a small apparent source neutron multiplication was observed with the entire assembly immersed in and reflected by water.

In other experiments the slugs, placed end to end to form vertical rods of variable length, were arranged in triangular lattices in a water moderator and reflector. With one pattern, having a water-to-metal volume ratio of 1.70 ($H:U^{235} = 252$; rods arranged on 2.00-in. centers), it was possible to make five pseudocylindrical assemblies of different height-to-diameter ratios critical. These data yield a value of 28×10^{-4} cm^{-2} for the material buckling, using an extrapolation distance of 7 cm. The minimum

mass, in near equilateral geometry, is about 42 kg of U^{235} (5.0 tons of uranium). In a second triangular array the water-to-metal volume ratio was 1.14 ($H:U^{235}$ = 169; 1.78-in. center-to-center rod spacing). The buckling was about 19 x 10^{-4} cm^{-2} in each of two assemblies. The minimum critical mass with this lattice spacing is about 105 kg of U^{235} (12.4 tons of uranium).

Measurements are also being made of the source neutron multiplication by lattices of units of 93% U^{235}-enriched uranium metal in air. Each unit is about 1 x 8 x 10 in. and, on the average, has a mass of 24.5 kg of uranium (22.8 kg U^{235}). One assembly consisted of a 3 x 3 x 5 array of 45 units, located on 12-in. centers, within a 1-in.-thick shell of Plexiglas which provided nominal neutron reflection. The source multiplication was 2.7. In a second configuration, which consisted of a cubical array of 125 units placed on 15-in. centers within a reflecting shell, the multiplication was 3.0. (AEC Activity 2712)

Dissolver Solution. — Work has continued on the three phases of the dissolver program mentioned in the December monthly report (ORNL-2664).

The amine-extraction coulometric-titration procedure for uranium, which has been outlined in previous reports, has been applied to nonradioactive dissolver solutions of the zirconium-uranium fuels, and it was found to have a relative standard deviation of 0.2%. Studies are now being made to determine the effects of the fission products on the extraction, on the titration, and on the entire procedure.

A supplementary procedure for uranium has also been developed. It involves (1) extraction of uranium from 5 ml of acid-deficient aluminum nitrate solution into 5.00 ml of methyl isobutyl ketone, (2) transference of 4.00 ml of the organic material into the titration vessel, (3) removal of the organic material by evaporation, (4) dissolution of the uranium residue in HNO_3 and H_2SO_4, and (5) coulometric titration of the uranyl sulfate. This procedure requires 35 to 45 min per single analysis. Studies of its precision are being conducted.

A procedure for coulometric titration of plutonium has been developed. It requires two electrolyses: a preliminary reduction at +0.125 v (vs Hg, Hg_2SO_4), which converts the plutonium to Pu(III), and then a coulometric oxidation at +0.425 v (vs Hg, Hg_2SO_4), which quantitatively converts Pu(III) to Pu(IV). Studies of the precision and interference tolerance of this method are in progress. (AEC Activity 2724)

PROGRAM 4000 – REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. – Reactor run 18, which started in December and had as its purpose the investigation of the relationship between reactor power and fuel stability, was concluded on February 2, when the reactor scrammed after 1281 hr of operation. During the run 363 Mwhr was accumulated, at power levels up to 1.6 Mw, with no indications of power disturbances. The scram, the first in the reactor's history, occurred when fresh fission gas was trapped in a closed instrument line near the O_2 activity monitors. There was no release of activity from the reactor piping. Shielding was placed around the instrument line, and operating procedures were modified to prevent future shutdowns for the same reason.

After the scram, the fuel circulating pump was found to have an open electrical lead. The shutdown for repair of the lead was extended to include repair of a leaking flange in the reactor steam system and replacement of the blanket dump valve and three diaphragm pump heads. The maintenance work was completed seven days ahead of schedule.

Preparations are being made for run 19, in which the investigation of fuel stability will continue as the primary objective. (AEC Activity 4103.3)

HRT Chemical Pilot Plant. – Collection of solid corrosion and fission products by the HRT chemical plant hydroclone dropped from the 2-g/hr rate observed for the first 300 hr of the current reactor run to 0.3 g/hr for the next 340 hr. The 0.3-g/hr rate corresponds to 50% of the estimated solids production rate in the reactor during this period. The high collection rate observed at the beginning of reactor runs is believed to result from the high initial solids concentration produced by scale removed from the core vessel and circulating system by thermal cycling during shutdown and startup operations. (AEC Activity 4103.1)

Gaseous Fission Product Disposal. – In further studies of the effect of linear gas flow rate on the effectiveness of rare-gas adsorption by long charcoal beds, the optimum superficial velocity was 0.5 fpm with pipes containing Columbia G charcoal at room temperature. Varying the bed diameter from 0.8 to 2.7 in. did not change the efficiency of the bed. (AEC Activity 4103.1)

Thorium Oxide Slurry Development. – Spherical particles of mixed thorium-uranium-aluminum oxide, with an average diameter of 2.5 μ, were produced experimentally for possible use in aqueous homogeneous reactor slurries by spraying an ethyl alcohol solution (10 parts Th, 1 part U, 1 part Al; all as nitrates) into an acetylene flame, equipped with a zirconia reflector, at 1400 to 1500°C. Atomization of a thoria sol into a lower-temperature flame produced rounded agglomerates of fine particles, which on refiring to 1000°C fused into glassy, spherical ThO_2 particles.

A slurry viscometer, which measures the terminal velocity of a metal ball rolling down an inclined slurry-filled tube, was tested for use with small samples of irradiated slurries. Data on effective slurry viscosity were reproducible within 10%. Yield-stress measurements in the neighborhood of 0.1 psf were also possible. (AEC Activity 4103.1)

HRP Analytical Chemistry. – A flame-photometric method was applied to the estimation of palladium which had been added to ThO_2 slurries in microgram quantities as a recombination catalyst. Palladium was separated from interferences by extraction of the α-furildioxime complex with hexone, after which the radiant intensity of the palladium in the hexone solution was measured flame photometrically at 363 mμ. For palladium in the range of 1 to 6 μg/ml, the coefficient of variation averaged about 3%.

A method was devised for dissolution of ThO_2-UO_x mixtures without atmospheric oxidation of uranium and for subsequent estimation of U(IV). The sample is dissolved by refluxing it with 7 M H_3PO_4 which contains $K_2Cr_2O_7$ in excess, after which the excess dichromate is determined by conventional titration with standard $FeSO_4$.

A new method of free-acid analysis has been studied and is now being tested in the "hot" cells. The method involves sorption of the cations on a Dowex 50-X8 resin column and subsequent elution with dilute lithium sulfate solution. In this process, hydrogen ion is selectively eluted as free acid. The separated free acid is then titrated photometrically with standard base. A series of standards has been analyzed by use of remote facilities, and the data at present indicate good precision and accuracy. The method will be adopted for "hot" samples. (AEC Activity 4103.1)

Slurry Blanket System Development. – The 300-SM blanket test facility was equipped with a new high-pressure (2000-psi) vessel designed specifically for handling slurry in any one of several different flow patterns. The vessel interior shape is that of a 30-in. hemisphere mounted on a 60° inverted cone. Two interchangeable head closures permit the vessel to be used for the study of either a slurry core system or a blanket system, as one head closure includes a 13-in. centrally located dummy core vessel.

In connection with the design of a slurry critical experiment (HRE-4), the vessel was set up for core-system performance studies with re-entrant flow at the bottom. On the basis of preliminary operation at 200°C and 500 psi with a slurry containing 500 g of Th per kg of H_2O, it is apparent that slurry flow within the vessel is close to the design criteria required of a reactor, a condition not achieved in any vessel heretofore. The slurry was maintained in suspension over a range of flows from 125 to 400 gpm; there was no evidence of settling-out on the vessel wall. The circulation of slurry could be started at will by merely turning on the pump. (AEC Activity 4103.1)

Engineering Research. – Heat transfer under nucleate-boiling conditions was studied for ThO_2-H_2O slurries containing up to about 1000 g of Th per liter. For concentrations up to about 200 g/liter, the presence of thoria

does not appear to influence the rate of heat transfer; at a concentration of 1000 g/liter, the maximum heat transfer rate (at burnout conditions) was 210,000 $Btu \cdot hr^{-1} \cdot ft^{-2}$, compared with 490,000 $Btu \cdot hr^{-1} \cdot ft^{-2}$ for water under corresponding conditions.

Thoria caking studies indicated that slurries have less tendency to form "cakes" and "spheres" at 50°C than at temperatures above 200°C, and also less tendency at a thorium concentration of about 40 g per kg of H_2O than at concentrations greater than 100 g/kg. Cakes are more easily formed on surfaces exposed to low fluid velocities (5 fps) than on those exposed to high velocities (20 fps), but if cakes are formed on all surfaces, those formed in the higher-velocity regions are more adherent. (AEC Activity 4103.1)

Core and Blanket Vessel Development. — Preliminary tests were made with a promising modification of the annular-inlet, concentric-polar-outlet model of the HRT replacement core. The stream impinging against the core wall from the annular inlet was stabilized against the entire core wall by means of a secondary jet entering at the pole opposite the annular inlet. Measurements of fluid-mixing in the model indicated that this geometry could be used to maintain cool fluid near the core wall in a reactor.

The carbon-steel model of the present HRT core was operated with forward and reverse flow while the number of diffuser screens was varied. The only combination tested which provided uniformly good heat transfer at the core wall plus complete removal of sedimentary solids was reverse flow with at least five screens removed. This flow geometry was recommended for future HRT experiments. (AEC Activity 4103.1)

Centrifugal Circulating Equipment. — The 300A slurry loop has been operated at 280°C for 1800 hr, with an indicated titanium loss from the pump hydraulic parts of less than 1 g. The quantity of 1600°C-fired thoria slurry charged to the loop was calculated to provide a circulating concentration of 460 g of ThO_2 per kg of H_2O; however, slurry deposition in the pressurizer reduced the actual circulating concentration to 200 g/kg. Methods of overcoming the deposition problem are being investigated.

A floating-bushing shaft seal for canned-motor pumps, fabricated from corrosion-resistant nitrided titanium, was shown to be free of galling when operated in water. The unit will be evaluated on the 300A pump. (AEC Activity 4103.1)

Miscellaneous Equipment. — The feasibility of the remote operations required to remove the diffuser screens from the HRT core and to plug the hole in the core tank was demonstrated, and progress was made in developing the necessary tools. A remote saw can be used to detach the screens from the wall. The Zircaloy screens can then be cut into small removable strips with a Heliarc torch, or they can be anodically disintegrated in 8 M HNO_3. A cover plate can be mounted over the hole and a bolt-toggle assembly tightened to provide a mechanical seal. (AEC Activity 4103.1)

Heat Removal Equipment. — Much of the tubing ordered for the Griscom-Russell test steam generators was rejected because of surface irregularities. There are a sufficient number of tubes, however, for tube-joint development and qualification. Layout sketches of the test loop for these units were completed. (AEC Activity 4103.1)

Gas Handling Equipment. — The natural-circulation high-pressure recombiner was operated at 1500 psi with a stoichiometric $H_2 + 1/2\ O_2$ input of 0.6 scfm up to a total of 1180 hr. There were no experimental difficulties or loss of catalyst efficiency. (AEC Activity 4103.1)

Feed Pump Development. — Prototype HRT fuel feed pumps have operated for more than 20 months without difficulty. Two increased-capacity solution diaphragm heads have continued to pump properly.

Two slurry feed-pump diaphragm heads have circulated ThO_2 slurries for 3150 hr; however, replacement of check-valve trim was required during this period. Materials for check valves on hand for testing include alumina, beryllia, zirconia, and thoria. When the feed-pump loop was operated with a gas surge volume in the high-pressure system, foam was produced in the letdown line where gas bubbles came out of solution. The foam was kept under control by mild boiling in the dump tank, and was eliminated by the addition of a trace of phosphoric acid. (AEC Activity 4103.1)

GENERAL REACTOR RESEARCH

Basic Reactor Research. — Critical experiments with aqueous solutions of U^{233} and U^{235} in a 69.2-cm-dia sphere to which boron was added as a neutron absorber were completed. A comparison of the results increased the average value of η of U^{233} for thermal neutrons from 2.27, determined with unpoisoned solutions, to 2.28. A 122-cm-dia aluminum sphere was also made critical when filled successively with aqueous solutions of $U^{233}O_2(NO_3)_2$ and $U^{235}O_2(NO_3)_2$. From these experiments a value of η for U^{233} of 2.29 was derived by using world-consistent cross sections from BNL-325 (2nd edition) and 2.076 as the thermal-neutron η for U^{235}. (AEC Activity 4202)

Power Reactor Fuel Reprocessing. — Because of the many types of fuel elements that will be used in power reactors, several dissolution and feed preparation methods are being developed for the production of satisfactory feed solutions for solvent extraction. In the case of stainless-steel-clad fuel elements having a sintered UO_2 and ThO_2 core, the jackets can be removed chemically by dissolution in 6 $\underline{M}$ H_2SO_4 (the Sulfex process); the cores can be dissolved separately in Thorex dissolver solution, 13 $\underline{M}$ HNO_3–0.05 $\underline{M}$ HF–0.2 $\underline{M}$ $Al(NO_3)_3$. The Darex process, involving dilute mixed acid (5 $\underline{M}$ HNO_3–2 $\underline{M}$ HCl) in a titanium-containing system, will be used for fuels containing mixtures of stainless steel and fissionable oxides. For Zircaloy-clad fuel elements having either an oxide core or a uranium-niobium-zirconium alloy core, the Zirflex process, in which 6 $\underline{M}$ NH_4F–1 $\underline{M}$ NH_4NO_3 is used as a dissolving agent, is being studied. Mechanical disassembly of fuel elements will be required to some extent for all fuels; mechanical

disassembly and cutting of active sections of fuel elements followed by selective leaching of core materials may compete with the all-chemical feed preparation procedures. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Sulfex Process. – Corrosion tests indicate that welded Ni-o-nel (heat treatment: 1850°C for 0.5 hr, air quench) may be satisfactory as a construction material for both the boiling sulfuric acid dejacketing step and the subsequent core dissolution with Thorex dissolver solution (TDS). The corrosion rate in boiling TDS was approximately 1 mil/month when the aluminum nitrate concentration was 0.2 M; the rate varied from 20 to 2 mils/month as the aluminum concentration was varied from 0.04 to 0.15 M. Increasing the aluminum concentration from 0.04 to 0.15 M (with the fluoride concentration held constant at 0.04 M) increased the dissolution time for ThO_2-UO_2 core material by 50%.

Stainless steel cladding which had been exposed to steam at 1200 psig and to oxygen at 800 psig did not react with boiling 6 M H_2SO_4 until it was activated by contact with unoxidized stainless steel. After the reaction started, the stainless steel cladding dissolved at a rate of 15 to 20 mg·cm^{-2}·min^{-1} in excess H_2SO_4 (3.3 times the stoichiometric requirement).

Criticality control may be required during decladding. While the stainless steel is being dissolved, core oxides are exposed to the decladding solution, in which they have some solubility; fines from cracked and dissolving core material can be suspended in the decladding solution. The addition of 0.25 M Cd^{++} and 0.25 M borate as homogeneous poisons for criticality control in decladding solutions decreased the dissolution rate of stainless steel in 6 M H_2SO_4 by about 20%; no precipitates formed in the product solution.

The dissolution rate of mixed ThO_2 (96 wt%) and UO_2 (4 wt%) pellets in 13 M HNO_3–0.05 M HF–0.2 M $Al(NO_3)_3$ required from 4 to 15 hr to complete, depending upon the excess of dissolvent over the stoichiometric ratio. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Zirflex Process. – Zircaloy-2 jackets can be dissolved in 6 M NH_4F–1 M NH_4NO_3 and the exposed UO_2 core in 10 M HNO_3. The losses of uranium resulting from the dissolution of UO_2 in the decladding solution were 0.02% for unirradiated fuel and 0.2% with irradiated samples.

For zirconium-clad uranium-zirconium-niobium alloy fuels, the jacket and the alloy can be dissolved together in 6 M NH_4F to which $Al(NO_3)_3$ and HNO_3 are added to complex the fluoride and to provide nitrate ion for extraction of the uranium into tributyl phosphate. By this dissolution technique, which can be run in conventional stainless steel equipment, enriched fuels such as those for the STR or EBWR core 1 can be processed. Uranium, zirconium, and niobium can be precipitated from the dissolver solutions as hydrous oxides with ammonium hydroxide and, after filtration or centrifugation, dissolved in nitric acid to yield solvent extraction feed solutions of low fluoride and high uranium concentration. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Darex Process. — In continuous dissolution tests with unirradiated APPR fuel, uniform dissolution rates of 275 g of fuel element per minute were obtained with a constant rate of addition of mixed acid of 4.5 liters/min. Braze alloy (by Coast Metals, Inc.) was insoluble in the dissolver solution, but not in fresh 5 M HNO_3–2 M HCl. No difference in dissolution rate was noticed between clean and oxidized stainless steel in this acid system.

Filtration of Darex dissolver solution with the Separan and gelatin technique studied at Savannah River was attempted with success. The Separan-gelatin mixture precipitated and coagulated the silica in the dissolver solution, so that filtration, with the aid of 10 g/liter of diatomaceous earth, was accomplished through a sand bed at a rate of 40 to 100 liters/hr. The filter cake required approximately 23 liters of 0.05 M HNO_3 for removal of the uranium. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Mechanical Processing. — Tests of the use of abrasion disks for sawing stainless steel rods indicated that at a disk speed of 10,770 surface feet per minute and a cutting rate of 10 in./min, approximately 4500 cuts through a 0.5-in. stainless steel rod would reduce the diameter of the 10-in. cutting disk by a factor of 2. The "sawdust" contained one part wheel dust to six parts metal fines. It was also shown that a stainless-steel-clad porcelain core (stand-in for UO_2) could not be cut successfully with a 24-in. abrasive disk running at 11,000 surface feet per minute.

A 36-tube porcelain-filled stainless steel tube bundle was sheared into short sections by using a 125-ton Manco hydraulic shear with "line of contact" blades. After 65 cuts through the entire bundle, the clearance between the shear blades increased from 2 mils to 18 mils without changing the efficiency of the shear. In tests with UO_2-filled single tubes it was shown that about 30% of the UO_2 contained in a 0.5-in. tube approximately 1 in. in sheared length was dislodged by the shearing action as dust, 50% of which was less than 150 μ in diameter.

In leaching tests on sheared 3/8-in.-dia stainless-steel-clad UO_2-core fuel sections 1.5 in. long, the sections of tubes were leached free of UO_2 by 10 M HNO_3 in approximately 25 min. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Hermex Process. — In studies to explore the use of mercury and amalgams as processing agents, further determinations of the solubility of fission products in mercury have been made. The solubility of gadolinium, chosen as representative of rare-earth fission products, increases from 0.0335 to 0.254 wt% as the temperature increases from 100 to 200°C.

Pellets of UO_2 fired at 1700°C can be reduced with a 25% excess of magnesium metal in the presence of $MgCl_2$ at 1000°C with a yield of 60%. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Solvent Extraction Studies. — A two-cycle flowsheet is being developed for recovering enriched U^{235} and U^{233} from solutions of thorium, uranium, and aluminum. This process is intended for use with Consolidated Edison fuel. Since recovery of thorium is not required, 2.5% tributyl phosphate is used. Both cycles are run at high solvent saturation; however, the first-cycle feed is 1.0 M in HNO_3, and the second-cycle feed is 0.1 M acid-deficient. (AEC Activity 4301)

Fused Salt—Fluoride Volatility Processing. — Experiments on dissolution of Zircaloy-2 dummy fuel elements in molten sodium fluoride—lithium fluoride in a 1/8-scale INOR-8 dissolver indicated average corrosion rates at the interface of 0.1 to 0.2 mil per 8 hr of dissolution time at 675 to 700°C. Dissolution rates were approximately 0.2 $lb \cdot hr^{-1} \cdot ft^{-2}$ with HF utilizations of 40%. There was a severe entrainment of solids in the off-gas. The superficial flow rate of the HF was approximately 30 $lb \cdot hr^{-1} \cdot ft^{-2}$. (AEC Activity 4302)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes by Sintering. — Mixes containing different amounts of kaolinite per unit volume of neutralized Purex waste were used to prepare sinters. Kaolinite was selected, since preliminary tests showed this clay to be nonswelling when heated with waste solutions. None of the kaolinite-waste mixes were entirely satisfactory for retaining cesium. When boric acid was substituted for a portion of the kaolinite, retention characteristics improved; this behavior of improved retention through addition of boric acid was also noted with Conasauga shale—waste mixes. Although kaolinite sinters do not have the same degree of retentivity as sinters prepared from shale, their attractive feature is the nonswelling character of the sinter on firing. Thus far, the kaolinite—boric acid—waste mixes investigated represent low kaolinite and boric acid concentrations (or high waste concentration per unit weight of clay). Sinters containing higher clay and boric acid contents are now under investigation. (AEC Activity 4352)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes into Deep Wells. — Several cores of Berea sandstone were coated with an epoxy resin and mounted for a study of the transmissibility of Purex waste and the sorption and extraction of Cs^{137} within the core. The neutralized citrate-complexed waste was pumped through the core with a Pulsafeeder. The concentrations of the Purex waste studied were a 9:1 and a 30:1 dilution using 1 M NaCl. The sorptive capacity of Berea sandstone for Cs^{137} was 5.6 and 3.2 curies/ft^3 for the 9:1 and 30:1 dilutions, respectively. These values were as expected on the basis of previous slurry experiments.

It is significant that the sorptive capacity of Berea sandstone for cesium in Purex waste is within the range of 2 to 8 curies/ft^3. Heat calculations show that a disposal aquifer may safely contain this amount. Moreover, cesium sorption can be controlled within the core by adjusting the sodium-to-cesium ratio. (AEC Activity 4352)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes into Salt Formations. — To determine the effect of irradiation upon the mechanical properties of rock salt, samples were hand-excavated from bedded deposits in Kansas and from dome deposits in Texas and irradiated in the cobalt storage facility.

Due to the shale inclusions the bedded salt samples had a wider variation than did the dome salt samples. Nevertheless, the dome salt had a higher compressive strength at room temperature than the bedded salt loaded both perpendicular and parallel to the planes of stratification. For a dose of 5×10^8 r, there were no differences in compressive strength between bedded and dome samples, although all were weaker than the unirradiated samples. At 200°C the differences between bedded and dome samples were reduced, although the unirradiated samples still had a higher compressive strength. At room temperature both unirradiated and irradiated samples of dome salt showed the least creep. The modulus of elasticity, yield strength, and apparent elastic limit were increased at a dose of 5×10^8 r, although the results were not significantly different from those obtained for the unirradiated samples. (AEC Activity 4352)

Waste Treatment. — All free acid was recovered from synthetic Darex (stainless steel dissolved in HNO_3) wastes when the volume was reduced by 77%. When Darex wastes were calcined in an 8-in. pot, 93% of the iron was found in the solids; the remainder was lost by entrainment. The calcined cake was 7% of the original waste volume.

Laboratory studies showed that the voids in calcined Darex and TBP-25 (aluminum) wastes were 80 and 90%, respectively; thermal conductivities were estimated as 0.2 and 0.1 $Btu \cdot hr^{-1} \cdot ft^{-1} \cdot (°F)^{-1}$ for the solids.

Estimates of temperature rise expected in calcined wastes during permanent storage indicate that the decay characteristics of fission product mixtures have little effect on the maximum temperature attained. For equal heat generation rates but varying fission product distributions, the differences in maximum temperature rise were less than 3% between one- and three-year-decayed wastes and less than 7% between one- and ten-year-decayed wastes. At a heat generation rate of 60 $Btu \cdot hr^{-1} \cdot gal^{-1}$ for the solid, the maximum temperature rise in a 0.75-ft-radius cylinder [$k = 0.1\ Btu \cdot hr^{-1} \cdot ft^{-1} \cdot (°F)^{-1}$] was 1270°F in "average soil," 1150°F in "average rock," and 1020°F in salt. The temperature rise in the center of one-year-decayed solid TBP-25 waste stored in a 55-gal drum in a granite cave was calculated as 694°F. (AEC Activity 4352)

MOLTEN-SALT REACTOR (MSR) PROGRAM

Chemistry. — The equations for the vapor pressure of UF_4, previously reported, have been revised by use of a different value for the heat capacity of the liquid and the inclusion of a heat capacity correction for the solid. The new equations for solid UF_4 and liquid UF_4, respectively, are

$$\log p \text{ (mm Hg)} = \frac{-20{,}060 \pm 240}{T} - 7.05 \log T + 38.01 \pm 0.22, \qquad (1)$$

$$\log p \text{ (mm Hg)} = \frac{-16{,}840 \pm 44}{T} - 7.549 \log T + 37.086 \pm 0.03. \qquad (2)$$

From these equations, the heats of sublimation and vaporization at 0°K were evaluated and found to be 91.8 ± 1.1 and 77.1 ± 0.2 kcal/mole, respectively. The normal boiling point calculated from Eq. 2 is 1723°K, and the heat and entropy of vaporization at the boiling point are 51.2 ± 0.2 kcal/mole and 29.7 e.u., respectively. The heat of fusion is 16.0 kcal/mole.

Studies of the phase equilibria in the system $NaF-BeF_2-ThF_4$ have established the composition limits and liquidus contours of the $NaF \cdot ThF_4$ primary phase field. This primary phase field is contiguous with those of β_2-$2NaF \cdot ThF_4$, $3NaF \cdot 2ThF_4$, $NaF \cdot 2ThF_4$, $2NaF \cdot BeF_2$, and $NaF \cdot BeF_2$. Temperatures and compositions of each of the associated ternary invariant points are known. The reproducible $\alpha \rightleftharpoons \beta$ transition of $NaF \cdot ThF_4$ at 535°C on the $NaF-BeF_2-ThF_4$ liquidus indicates that β-$NaF \cdot ThF_4$ is an equilibrium phase in the system $NaF-ThF_4$.

The solubility of neon gas in an $LiF-BeF_2$ (64-36 mole %) mixture has been determined over a pressure range of 0 to 2 atm, and the Henry's law constants have been determined experimentally in the temperature range 500 to 800°C.

The solubility of HF in two compositions of the $NaF-BeF_2$ system has been determined over the pressure range of 0 to 2.5 atm. The Henry's law constants for the compositions $NaF-BeF_2$ (49-51 and 58-42 mole %) have been measured at 600, 700, and 800°C.

Investigations of the permeability of reactor-grade graphite and special grades of graphite have included treatment of AGOT graphite, after evacuation at 1250°F, with $LiF-BeF_2-UF_4$ (62-37-1 mole %) at this same temperature. The specimens showed 1 to 2% gains in weight. Machine cuttings have been submitted for analytical determination of the depth of penetration. Long-term soaking of some special grades of graphite rods impregnated with an $LiF-MgF_2$ mixture is continuing at 1250°F in the $LiF-BeF_2-UF_4$ (62-37-1 mole %) mixture to determine possible penetration. (AEC Activity 4103.1B)

Physical Properties. — The enthalpy and heat capacity of the salt mixture $LiF-BeF_2-ThF_4-UF_4$ (62.0-36.5-1.0-0.5 mole %) have been experimentally determined in the liquid state over the temperature range 500 to 800°C. The enthalpy above 30°C can be expressed as

$$H_T - H_{30°C}(\text{cal/g}) = -2.1 + 0.545t - (4.03 \times 10^{-5})\, t^2,$$

where t is the temperature in °C. The heat capacity is then

$$c_p\ [\mathrm{cal \cdot g^{-1} \cdot (^\circ C)^{-1}}] = 0.545 - (8.06 \times 10^{-5})\ t.$$

The surface tension of the same salt mixture was experimentally determined in the temperature range 500 to 800°C to be given by

$$\sigma\ (\mathrm{dynes/cm}) = 272.2 - 0.143t$$

to a precision of ±5%. This value is approximately 5% above the result previously reported for the related mixture $LiF-BeF_2-UF_4$ (62-37-1 mole %). (AEC Activity 4103.1B)

Fuel Processing. — Following uranium recovery from $LiF-BeF_2-UF_4$ by the fluoride-volatility process, it may be feasible to recover the Li^7 by dissolution in anhydrous HF. It was previously found that LiF dissolves appreciably along with some BeF_2 in 100% HF and that rare-earth fluorides are almost insoluble. Further solubility measurements indicate that NpF_3 is soluble to only 0.0002 mole % in HF saturated with $LiF-BeF_2$. Thus it is possible to free the costly LiF of two major neutron poisons, rare earths and neptunium, generated in a molten-salt reactor burning U^{235}. (AEC Activity 4103.1B)

Dynamic Corrosion Tests of INOR-8 and Inconel in Molten Salts. — Metallographic examination of an INOR-8 alloy thermal-convection loop which circulated salt 126 ($LiF-BeF_2-UF_4$, 53-46-1 mole %) for one year at a hot-leg temperature of 1250°F revealed no observable attack. Two Inconel thermal-convection loops which operated for one year at hot-leg temperatures of 1350°F showed moderate to heavy intergranular attack to a depth of 6.5 mils by salt 128 ($LiF-ThF_4$, 71-29 mole %) and similar attack to a depth of 7 mils by salt 129 ($NaF-ZrF_4-UF_4$, 55.3-40.7-4 mole %). (AEC Activity 4103.1B)

Mechanical Properties of INOR-8. — Fatigue studies are being conducted on INOR-8 alloy. These include rotating-beam tests on smooth-bar specimens which have been programed at 1200 to 1500°F and frequencies of 100 and 3000 rpm. The tests at 1500°F have been completed, and the results indicate the 10^6-cycle fatigue stress for INOR-8 to be approximately 30,000 psi, as compared with 18,000 psi for Inconel. Tensile tests have shown higher strengths for notched INOR-8 specimens at room temperature and 1500°F than for smooth-bar specimens. It thus appears that INOR-8 is not notch-sensitive. (AEC Activity 4103.1B)

Graphite Compatibility with Molten Salts. — INOR-8 specimens exposed to salt 130 ($LiF-BeF_2-UF_4$, 62-37-1 mole %) and graphite for 4000 hr at 1300°F showed no signs of carburization upon metallographic examination, and exhibited mechanical properties similar to those of control specimens when tested in an argon atmosphere for similar times and at similar temperatures. Degassed graphite specimens have been completely penetrated within 100 hr by

salt 30 ($NaF-ZrF_4-UF_4$, 50-46-4 mole %) at 1300°F under an argon pressure of 150 psi. No penetration occurred in similar tests with salt 130 ($LiF-BeF_2-UF_4$, 62-37-1 mole %). The effects of thermal cycling and freezing, with subsequent melting, on the properties of salt-penetrated graphite are being studied. (AEC Activity 4103.1B)

In-Pile Loop Tests. — The first MSR in-pile loop, which started operating in the MTR on December 3, 1958, was terminated January 8, 1959, after 864 hr of operation, when a high radiation field accompanied by fission-gas release developed in the vicinity of the loop as a result of partial plugging of the pump-sump purge-outlet line. As a result of the reduced purge flow, fission gases back-diffused up the inlet line to a leak in a bad silver-solder joint between the loop cubicle and the instrument panel. The second in-pile loop is under construction. (AEC Activity 4103.1B)

Pump Development. — Several tests of INOR-8 journals and bearings operating in salt 130 were conducted in the molten-salt-lubricated journal bearing tester. These tests were conducted over a speed range of 600 to 2700 rpm at loads of 10 to 500 lb. Visual inspection after the tests revealed a slight amount of rubbing. The facility for testing a molten-salt-lubricated thrust bearing has been completed and is ready for operation. A layout of a test pump that incorporates a submerged molten-salt-lubricated hydrodynamic bearing has been approved, and detail design work has begun.

A centrifugal pump has accumulated 14,400 hr of continuous operation with molten salt 30 at 1200°F, 645 gpm, and 2700 rpm. Another pump equipped with a bellows-mounted seal has operated 11,800 hr with NaK at 1210°F, 1200 gpm, and 2800 rpm. (AEC Activity 4103.1B)

Forced-Circulation Loop Corrosion Tests. — Fifteen forced-circulation loops continued to operate during this period. An Inconel loop which was terminated after a year of operation was replaced by another Inconel loop. An INOR-8 loop was shut down temporarily after 5000 hr of operation for removal of machined inserts for weight-loss determinations.

The accumulated operating times are 24,000 hr for Inconel loops and 57,000 hr for INOR-8 loops. (AEC Activity 4103.1B)

Thermal-Convection Loop Corrosion Tests. — During this period, four Inconel loops and five INOR-8 loops have been terminated after a year of operation. Six new Inconel loops and six new INOR-8 loops were placed in operation. (AEC Activity 4103.1B)

Frozen-Lead-Seal Pump. — A small frozen-lead-seal pump consisting of a centrifugal pump mounted vertically over a fractional horsepower motor drive has operated continuously for more than 6200 hr. The pump circulates a molten salt mixture isothermally at 1200°F. (AEC Activity 4103.1B)

CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR

PEGCPR Design Review. — Four-group two-dimensional diffusion-theory calculations have been made to determine the power distribution in the Kaiser-ACF prototype reactor (PEGCPR). The following configurations were studied: (1) the central rod inserted to the reactor mid-plane, (2) the central rod and the next four rods fully inserted, and (3) just the four rods nearest the center one fully inserted. Peak-to-average power density ratios are given below:

Configuration of B_4C Control Rods	P_{max}/P_{av}	δk
No rods	1.78	0
Central rod half inserted	1.81	0.0076
Central rod fully inserted*	1.42	0.020
Four rods fully inserted	1.77	0.062
Five rods fully inserted	1.46	0.080

*One-dimensional, multigroup result.

The four-rod case has a peak at the center of the reactor. The five-rod case has a distribution that is very flat, except immediately around the rods. (AEC Activity 4110)

Mass-Transfer Experiment. — Two essentially identical runs have been completed for a peripheral rod in a septafoil arrangement of 1-in.-OD rods of 1.4-in. center-to-center spacings contained within a 4.0-in.-ID channel. At a Reynolds modulus of ~ 60,000, both runs yielded over-all mass-transfer data within 20% of that predicted by the correlating equation

$$j_m = 0.023 N_{Re}^{-0.2} .$$

The close agreement of the ratio j_{local}/j_{mean} for the two runs indicates that the apparatus and technique employed are capable of high experimental precision. For the peripheral rod studied, two definite minima were observed: the first at the circumferential location closest to the channel wall for which j_{local}/j_{mean} was found to be essentially 0.74, the second at the position closest to the central tube with a j-factor ratio of nearly 1.0. A slight asymmetry in the flow was observed, although care was taken to ensure nearly ideal approach conditions (a 6-ft calming length of 4-in.-ID pipe preceded by a flow-straightening section). (AEC Activity 4110)

Graphite Coatings. — A blind-end coated graphite tube which was joined to an Inconel tube was heated at 100°C with helium at a pressure of 100 psi on the inside of the tube. After 250 hr at this temperature and pressure, there are no indications of leaks through the graphite coating. Irradiation of the coated graphite specimens continues, since there is no evidence of breakthrough of fission products. (AEC Activity 4110)

Static Pot Tests. — Final analyses of the gaseous impurities in static test No. 9, which contained type AGOT graphite, showed the total impurity concentration to be reduced from a maximum of 6.5% to the order of 0.01% after 900 hr at 1400°F. Visual examination of the metal test specimens revealed a uniform gray coating on all samples. The specimens are undergoing detailed examination. (AEC Activity 4110)

Mechanical Properties of Type 304 Stainless Steel. — Tube-burst tests are being conducted on 0.800-in.-dia specimens. The wall thickness of this tubing is 0.020 in. Results of the tests to date are summarized below. All specimens ruptured in the tube wall.

Type of End Closure	Test Temperature (°F)	Internal Pressure (psi)	Time to Rupture
Butt weld	1500	1500	5.0 sec
Edge fusion	1500	1500	6.7 sec
Butt weld	1500	1000	184.9 sec
Edge fusion	1300	1500	18.5 min
Edge fusion	1300	2000	16.4 sec

A hot-streak test at a uniform temperature of 1300°F with a hot streak at 1500°F and an external pressure of 300 psi was discontinued at the end of 2000 hr. The diametral clearance between the inner surface of the tube wall and the inserts was originally 0.010 in. Collapse of the tube wall occurred, but a wrinkle was not formed. The diameter of the tube along the hot-streak section was 0.010 in. greater than the diameter 90° from the hot streak. (AEC Activity 4110)

Evaluation of Beryllium Tubing. — Extensive evaluation tests have been completed on some beryllium tubing samples (0.040-in. nominal wall thickness). Wall thickness measurements revealed variations over the range 0.037 to 0.050 in. Apparent local density increases of up to 10% were observed radiographically. These variations are attributed to the presence of concentrations of impurities in the beryllium. Fluorescent-penetrant examinations revealed the presence of many small pinholes in the samples. (AEC Activity 4110).

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR-1). — Eight control rod fuel sections for core II of APPR-1 were manufactured. As a result of recent development work, the fuel sections have integral flux suppressors containing Eu_2O_3, and the absorber sections utilize Eu_2O_3 as the nuclear poison. Shipment of seven sets to Fort Belvoir and one set to the Martin Company was made during the week of February 8. (AEC Activity 4601)

PROGRAM 5000 — PHYSICAL RESEARCH

ISOTOPE PRODUCTION

Radioisotope and Stable Isotope Production. — The elements Gd, Mg, Bi, Tl, Hg, and Rb were processed in the calutron for collection of isotopes.

Nineteen new lots of isotopes of Ca, Cu, K, and Lu and one returned lot of Si^{29} were refined chemically. New lots of K^{39}, K^{40}, K^{41}, Ca^{42}, and Ca^{44} were prepared for inventory following chemical refinement and spectrographic and mass analysis. Some of the K^{40} samples attained an isotopic purity of 85%. This material has the highest purity, both chemically and isotopically, ever achieved for K^{40} at ORNL.

Radioisotope Research and Development. — The ion exchange column separation equipment in Building 3028 was completed and checked. The capacity of this equipment was further increased by installing two 3-in.-dia "roughing" columns and using the two tantalum columns for final separation passes. Two additional 4-1/2-in.-dia columns are being installed in cell 10 of the Fission Product Pilot Plant.

A process was developed for the separation of Y^{91} from the rare-earth fraction in short-decayed waste by batch extraction with di-2-ethylhexylphosphoric acid. Cerium-144 is also extracted quantitatively in this process step by oxidation to Ce(IV) with $KMnO_4$. After the Y^{91} is stripped into 10 N HNO_3, the Ce^{144} is reduced and stripped into 1 N HNO_3. The equipment for this process has been designed and is being installed in cell 9 of the Fission Product Pilot Plant.

An alternative process for the purification of Pm^{147} is continuous extraction in a 12 N HNO_3–tributyl phosphate system in mixer-settler cascades. The pertinent distribution coefficients for the system were determined, and a flowsheet utilizing available equipment was developed. Design of equipment for installation in the unused tank farm evaporator cell is in progress.

The temperature of sulfur in a 3/4-in.-dia can positioned against the face of the ORR lattice was 235°C. On the basis of this value, use of a 1/2-in.-dia can for P^{32} production will be allowable in the isotope stringer position. (AEC Activity 5100)

Stable Isotope Development. — The search for a chemical method applicable to the enrichment of Ca^{46} was resumed. An ion exchange system which has been reported to have an over-all separation factor of 4.4 for calcium isotopes is in operation at room temperature. Before separation factors are determined, the column will be heated to 90 or 95°C.

A calutron separation of Bi^{208} and Bi^{210} is being made from irradiated feed containing approximately 1 ppm Bi^{208} and traces of Bi^{210}. The operation involves the simultaneous collection of all the isotopes of

bismuth and also those of thallium, which are being used as monitoring aids. The minimum enrichment of the desired product should exceed 20. (AEC Activity 5100)

PHYSICS

High-Energy Physics. — A study was made of about 90,000 pictures obtained with the Duke University liquid-helium bubble chamber, which was exposed to a low-energy K^- beam at the Berkeley Bevatron. The chamber is situated in a magnetic field of 14.3 kilogauss. About 9000 K^- mesons were observed, of which about 2500 were stopped in the chamber. The following features of the K^--helium interaction are being studied: (1) the neutral-to-charged-hyperon production ratio, (2) relative numbers of Λ_0's produced both directly and by $\Sigma \rightarrow \Lambda_0$ conversion, (3) angular correlations in Λ^0 decay, pertinent to the question of parity conservation in Λ^0 production, and (4) hyperfragment production rates. Preliminary scanning shows that Λ^0 production predominates over charged Σ production; this is expected from the large Σ-Λ^0 conversion observed in K^- absorption in deuterium. (AEC Activity 5210)

High-Voltage Experimental Program: Coulomb Excitation of the First 2^+ State in Some Medium-Weight Even-Even Nuclei. — Coulomb excitation of the first 2^+ state has been measured for the 14 even-even nuclei Ni^{58}, Ni^{60}, Ni^{62}, Zn^{64}, Zn^{66}, Zn^{68}, Zn^{70}, Ge^{72}, Ge^{74}, Ge^{76}, Se^{76}, Se^{78}, Se^{80}, and Se^{82}. Coulomb excitation was effected by alpha particles with energies of 3 to 10 Mev. Over this energy range the thick target yield of Ge^{76} excitation varied by a factor of 20,000. Up to an alpha energy of 8 Mev, the yield followed that expected for Coulomb excitation. Above 8 Mev there were small but significant deviations. The position of the first 2^+ state in Zn^{70} (887 ± 9 kev) was not previously known. The value obtained for the 2^+ state in Se^{82} (670 ± 10 kev) is different from that previously reported. The absolute B(E2) values are determined to ±10%. For the three nuclei Ni^{60}, Ge^{72}, and Ge^{74}, the Coulomb excitation gamma-ray lifetimes may be compared to those obtained by Metzger, who used the resonance fluorescence method. There is good agreement. The enhancements over the single-particle transition estimate (taking $r_0 = 1.20 \times 10^{-13}$ cm) vary from a low of 11 for Ni^{58} to a high of 47 for Se^{76}. The previously observed constancy of the mass parameter $B_2/(B_2)_{irrot}$ is also found for the nuclei reported here (Phys. Rev. 110, 489, 1958). (AEC Activity 5220)

High-Voltage Experimental Program: Cross Section for the $Al^{27}(n,\alpha)Na^{24}$ Reaction as a Function of Neutron Energy. — The $Al^{27}(n,\alpha)Na^{24}$ cross section has been measured as a function of neutron energy from 6.2 to 8.3 Mev and at 14.8 Mev. The measurement was made relative to the fission cross section of U^{238} by using samples of aluminum mounted adjacent to and concentric with a thin deposit (~100 $\mu g/cm^2$) of U^{238}. The number of $Al^{27}(n,\alpha)$ events was determined by counting gamma rays from the residual radioactive Na^{24} nuclei. The gamma-ray detector was an NaI(Tl) crystal whose efficiency was measured in auxiliary experiments by use of coincidence techniques and 4π beta counting. The number of neutrons incident on the aluminum sample was determined by counting fission fragments from the U^{238} deposit in a 2π ionization

chamber. While a number of peaks appear in the curve for cross section vs energy, there is a general increase in cross section with increasing neutron energy, from about 3 millibarns at 6.2 Mev to about 46 millibarns at 8.3 Mev. The measured cross section at 14.8 Mev is 117 ± 8 millibarns. (AEC Activity 5220)

High-Voltage Experimental Program: s-Wave Detector of Deuteron Polarization and 14-Mev Polarized Neutron Source. — A new method has been proposed for analysis of the polarization of a deuteron beam. The fact that isotropy does not necessarily follow for a reaction induced by s-wave deuterons may make possible, for example, the use of the $H^3(d,n)He^4$ reaction as a sensitive detector of the polarization, or alignment, of the incident deuteron beam. It is shown that for a pure $m_d = +1$ beam, longitudinally polarized, the angular distribution of the neutrons in the region of the 107-kev resonance will be $(2/3)\ K(2 + 3 \sin^2 \theta)$, where $K \doteq |U|^2/16k^2$ is independent of angle, U is the relevant element of the collision matrix, and k is the deuteron wave number. The 14-Mev neutrons emitted at 90° (C.M.) will be 80% transversely polarized opposite to the beam direction. It is apparent that with a low-voltage open-air accelerator there need be no limitation on the size of the deuteron polarizing apparatus, and the easy accessibility of such apparatus has many advantages. (AEC Activity 5220)

Classification of Low-Lying Nuclear Energy Levels. — The decay of 8.3-day Ag^{106} ($I_0 = 6$) has been investigated with scintillation spectrometers. The Ag^{106} was produced by an (α,n) reaction on Rh^{103}. The single gamma-ray spectrum and the spectra in coincidence with the 0.513-, 0.725-, 0.808-, 1.051-, and 1.54-Mev gamma rays were measured. The energies (and relative intensities) of the gamma rays are 0.215 (11), 0.41 (26), 0.45 (37), 0.513 (100), 0.618 (26), 0.725 (49), 0.808 (41), 0.85 (12), 1.051 (34), 1.128 (11), 1.213 (21), 1.54 (26), and 1.77 (3) Mev. Energy levels have been proposed in Pd^{106} at 0.513 (2^+), 1.131 (2^+), 1.56 (2), 1.94, 2.06, and 2.78 Mev. Of these the levels at 0.513, 1.131, and 1.56 Mev have also been found to be populated in the decay of Rh^{106}($I_0 = 1$). The ratio of the cascade to crossover gamma rays from the second 2^+ state is 2.3 ± 0.3. This result is in agreement with the value 2.1 ± 0.6, which was obtained from the decay of Rh^{106} (ORNL-2664). (AEC Activity 5220)

The 86-Inch Cyclotron: Nuclear Physics. — The energy distributions of tritons from (p,t) reactions on Zr^{90}, Zr^{91}, and Zr^{92} were examined; very few levels are excited in the residual nucleus. The energies of these levels are consistent with the interpretation that the reaction proceeds as an interaction of the proton with only two neutrons in the target; the proton picks them up, leaving the rest of the nucleus undisturbed. No compound-nucleus contribution to the reaction was observed.

The electron capture of Lu^{173} (1.30-year) to levels in Yb^{173} was investigated with scintillation spectrometers and permanent-magnet spectrographs. Levels in Yb^{173} were observed at 78.7 (7/2), 179.5 ($9/2^-$), 351.2 ($7/2^+$), and 636.8 ($7/2^-$) kev. The first two excited states may be interpreted as members of a rotational band based on the ground state

[(512) $5/2^-$]. The levels at 351 and 637 kev are probably the intrinsic levels [(633) $7/2^+$] and [(514) $7/2^-$], respectively, predicted by Nilsson (Kgl. Danske Videnskab. Selskab, Mat.-fys. Medd. 29, No. 16, 1955). L-electron capture was observed to be the primary mode of population of the 637-kev level. (AEC Activity 5220)

The 86-Inch Cyclotron: Applied Physics. — The isotopes Be^7, V^{48}, As^{74}, Sr^{85}, Y^{88}, Hg^{197}, and Cd^{109} were made in service irradiations for outside customers. (AEC Activity 5220)

Heavy-Particle Physics. — The study of elastic scattering of nitrogen from carbon was extended to lower bombarding energies. At 23.5 Mev the differential cross section is, on the average, twice as large as it is at 27.5 Mev. It still exhibits considerable structure; for example, there is a maximum at 105° (C.M.) and a sharp minimum at 125° (C.M.).

The total cross section for the exchange transfer reaction $Al^{27} + N^{14} \rightarrow Mg^{27} + O^{14}$ was found to be $2.3 \begin{smallmatrix}+2.0\\-1.0\end{smallmatrix} \times 10^{-30}$ cm^2. This reaction may proceed by a simultaneous transfer of a proton from one nucleus and a neutron from the other. It is the first such reaction to be discovered. (AEC Activity 5220)

Spectroscopy Research Laboratory. — The infrared spectra of thin films of solid NH_3 and ND_3 have been studied as a function of temperature and of the conditions under which the films were deposited. Observations of the spectra were made at prism resolution, using a low-temperature cell of conventional design. Spectra have been obtained from the two metastable phases reported by the x-ray studies of Mauer and McMurdie, and from the cubic stable phase. The metastable phases were prepared by the deposition of solid films at 77 and 112°K. The stable modification was obtained by deposition at 160°K, and its spectrum was studied at temperatures down to 77°K. The spectrum of the stable phase is in good agreement with that predicted from the established cubic structure (Bull. classe sci., Acad. roy. Belg. 11, 655, 1925; Z. Krist. 61, 532, 1925). No evidence of inversion doubling was found in any phase. (AEC Activity 5230)

Relativistic Isochronous Cyclotron. — In cooperation with the Catalytic Construction Co., the architect-engineering firm selected for the ORIC, design information is being developed for preparation of the title I engineering; the target date is March 16. The over-all requirements for the building are practically complete; detail specifications of laboratory layouts and services are 90% complete; designs for the various penetrations through the concrete shielding were prepared.

Flowsheets for the water and vacuum systems of the cyclotron are 80% complete. The finished magnet yoke and pole bases were inspected at U.S. Steel, Pittsburgh, and accepted for shipment. A quarter-scale model magnet is being designed for use in computing magnetic field requirements for the deflector system. (AEC Activity 5240)

High-Energy Accelerator Shielding. — A high-energy accelerator shielding program has been initiated to provide data to aid in the design of experimental and biological shields for high-energy particle accelerators. The program will consist in a Monte Carlo calculation of the angular distributions and energy spectra of mesons and nucleons that penetrate shields having slab geometry where the incident radiation is limited to mesons and nucleons with energies below 3 bev. It is further intended to extrapolate existing physical data into the higher energy ranges in an attempt to provide useful data for incident particle energies up to 50 bev.

In order to provide the necessary differential cross-section data for the penetration calculation, it is first necessary to perform a nuclear cascade calculation similar to that done by Metropolis et al. (Phys. Rev. 110, 204,1958). The Monte Carlo method of treating this problem will be altered to automatically produce average angular distributions and energy spectra for the cascade particles leaving the nucleus, the average values being the most useful for the penetration problem. The elemental cross sections for the cascade calculation are now being collected, and the problem is being coded for the IBM-704 computer.

It is anticipated that experiments will be performed with the Brookhaven Cosmotron as a definitive check for the penetration calculation. (AEC Activity 5240)

Special Separations. — Calutron collections of U^{233} were completed. The collected samples contain less than 5 ppm of other uranium isotopes.

Runs are in progress to collect Ca^{46} and Ca^{48}. Six tanks are being utilized in these collections. An estimated 17 mg of Ca^{46} and 250 mg of Ca^{48} have been collected.

The recovery of high-purity U^{233} feed material was started in December and is 95% complete. Of the original 765 g available for calutron charge, 636 g was returned to inventory as the metal. Recovery of the rest of the material is in progress. (AEC Activity 5250)

Mass Spectrometry and Related Techniques. — The ions produced by alpha particles in several polyatomic gases (acetylene, methyl alcohol, carbon dioxide, and n-butane) were identified and measured with a mass spectrometer. The relative proportions of various ions thus produced are strikingly different from those produced by 75-ev electrons. This preliminary work indicates that the use of alpha particles to produce ions in a mass spectrometer may be advantageous for mass-spectral analysis in addition to the obvious application to radiation chemistry. The principal advantage for analytical application is the much simplified mass spectrum observed. (AEC Activity 5250)

CHEMISTRY

Raw Materials Research and Development. — The uranium-thorium ores from the Blind River district in Canada are processed for uranium recovery by anion exchange; thorium is expelled in raffinate as a sulfate solution. In most plant raffinates, there are also significant concentrations (1 to 2 g/liter) of nitrate ion which result from the tailings of uranium elution with NH_4NO_3 solutions. Recovery of thorium by extraction with a secondary amine (the Amex process) has in the past been seriously hindered by nitrate ion. This interference can be circumvented by using primary amines, which have a stronger affinity for thorium. As a result, a simple, economical solvent extraction flowsheet similar to those widely used for uranium can be devised for thorium.

The possibility of processing carbonate leach liquors of uranium ore by solvent extraction has increased recently because of the commercial availability of a new quaternary ammonium compound, Aliquat 336, which, at a concentration of 0.08 M in 90% kerosene—10% tridecanol, gives uranium distribution coefficients of approximately 30, and separates rapidly enough from carbonate leach liquors for process use. A suitable low-cost stripping method must be developed, however. A solution of sodium hydroxide plus sodium carbonate has shown the most promise. (AEC Activity 5310)

Chemical Engineering Research. — The integral diffusivity of uranyl nitrate in water was measured in a capillary diffusion cell and found to be approximately 6×10^{-7} cm^2/sec at 23°C. Rapid measurements of uranium concentrations of the order of 0.1 to 0.2 g/liter were made with a gamma scintillation counter; total counting periods of approximately 15 min gave results precise to within ±5%. This technique significantly decreased the time required for making accurate diffusivity measurements. (AEC Activity 5310)

Transuranics. — Laboratory-scale studies of solvent extraction methods for recovery of plutonium, americium, and curium from highly irradiated aluminum-clad plutonium indicate that more than 99.9% of the plutonium can be recovered by using 5% tributyl phosphate (TBP) in Amsco as the solvent, when the aqueous phase is 4 M in nitric acid. Americium and curium can then be separated from all fission products except rare earths by using a 40% solution of TBP in Amsco, when the aqueous phase is nearly neutral with respect to acid. (AEC Activity 5310)

Extraction Reagent Performance. — In studies of the effect of radiation on solvents used for purification by solvent extraction, an over-all G value of 4.1 molecules per 100 ev absorbed was determined for the decomposition of tri-n-octylphosphine oxide at a total dose of 400 whr/liter; of this amount, 2.8 molecules are converted to a polymer. At the same total absorbed dose, Primene JM-T, a primary amine, is decomposed with a total G value of 2.25 molecules per 100 ev, of which 1.50 molecules per 100 ev formed an unidentified polymer. Tributyl phosphate which has been equilibrated with 12 M HNO_3 is decomposed by radiation with a total G value of 3.1 to 4.0 molecules per 100 ev, as compared with a value of 2.3 for

TBP not saturated with HNO_3. The complexing power for rare earths of the products from radiolysis of TBP saturated with HNO_3 is 4.2 to 5.4 equivalents per mole of TBP decomposed, as compared with 2.5 equivalents per mole for TBP not equilibrated with HNO_3. (AEC Activity 5310)

Equipment Decontamination. — The effectiveness of the removal of corrosion scale with chromous sulfate as a decontamination procedure for components of aqueous homogeneous reactors was tested on a canned rotor pump removed from the HRT Chemical Plant. An over-all decontamination factor of 10 was obtained when a mixture of chromous sulfate and sulfuric acid was passed through the pump at 85°C, followed by water and acidic peroxide. This was sufficient to permit manual dismantling. Inspection indicated that the reagents had cleaned the surfaces with which they had come in contact and that the bulk of the residual activity was located in regions where the solution had not reached the metal surfaces. The bearings of this pump were frozen, so that it was not possible to use the pump itself to obtain better contact. (AEC Activity 5310)

Ion Exchange Technology. — Analysis of the results of two experiments to determine the kinetics of the sorption of uranium(VI) by 18-mesh Dowex 21K resin from 0.05 M sulfate solution at pH 2 indicated that the half-time is approximately 30 min at a superficial flow rate of 70 cm/min. (AEC Activity 5310)

Solvent Extraction Research. — Multistage continuous tests of the separation of rare earths by distribution between tributyl phosphate and nitric acid have shown the applicability of this method to the recovery and separation of promethium from fission-product rare earths. In a two-cycle system with 25 stages in the first cycle and 34 stages in the second, it is estimated that promethium product can be obtained at a yield >90% and a purity >85%.

Several solvents have shown potential utility for recovering neptunium from acid nitrate waste streams. Either 0.3 M triisooctylamine (TIOA, available commercially) or 0.01 M tri-n-octylphosphine oxide (TOPO, available as a specialty chemical) extracts Np(IV) with a distribution coefficient of ~20 from 8 M HNO_3, ~50 from 1 M HNO_3, or >200 from a solution that is 1 M in HNO_3 and 6 M in total nitrate. Di(2-ethylhexyl)phosphoric acid at 0.01 M extracts neptunium together with zirconium; the distribution coefficient for Np(IV) is ~500 from 1 M HNO_3 (with or without additional nitrate salt) and 3000 from a solution that is 0.4 M in HNO_3 and 6 M in total nitrate. The extracted neptunium can be stripped from all three reagents by dilute sulfate, oxalate, or carbonate solutions.

A process scheme is proposed for the purification and separation of neptunium and plutonium according to which Np(IV) and Pu(III) are extracted with 0.3 M TIOA from 2 M $Al(NO_3)_3$ at <0.1 M HNO_3. Data indicate that the Pu(III) can be selectively stripped with 2 M HNO_3 containing a holding reductant, and the Np(IV) can be subsequently stripped with dilute sulfate or oxalate. (AEC Activity 5310)

High-Temperature and Structural Chemistry. - A high-temperature copper-block drop calorimeter was built for measuring the heats of fusion of salts. The heats of fusion are needed for the interpretation, in terms of solute species, of the depression of the freezing point brought about by the addition of a metal to the salt.

The heats of fusion ΔH_f of several salts were determined from heat content measurements carried out above and below the melting point. The heat of fusion of lithium iodide was found to be 3.50 ± 0.1 kcal, approximately three times the literature value, which is thus shown to be in error. The heat of fusion obtained for bismuth trichloride, 5.45 ± 0.15 kcal, agrees fairly well with a value derived from cryoscopic measurements. It confirms the previously tentative assumption that bismuth metal enters into solution in bismuth trichloride as a dimer of the "subhalide" BiCl. The corresponding value for bismuth triiodide, 9.40 ± 0.2 kcal, is consistent with the Bi-BiI_3 phase diagram and with vapor pressure measurements found in the recent literature. It supports the surprising conclusion from the vapor pressure measurements that the solvent BiI_3 in the solution of bismuth appears to obey Raoult's law up to concentrations as high as 30 mole % Bi. (AEC Activity 5330)

Molten-Salt Thermodynamics. - Activity coefficients for $AgNO_3$ in the molten reciprocal salt system KNO_3-$AgNO_3$-KCl have been obtained from emf measurements at five different temperatures. Substitution of these values into the lattice model equation (ORNL-2636)

$$\gamma_{AD} = \frac{(1 - X)^Z \left[1 + \frac{X}{\beta(1 - X)} \right]^{Z - 1}}{1 - N_{C^-}}$$

where $\beta = e^{-\Delta E/RT}$,

X = fraction of anions lattice positions adjacent to A^- ions which are occupied by a C^- ion,

and N_{C^-} is the ion fraction of C^-,

permits estimation of ΔE, the energy of formation of the ion pair Ag^+Cl^-, for any arbitrarily chosen value of the lattice coordination number Z. If a value of 6 is assigned to Z, the value obtained for ΔE is 5.65 kcal/mole, constant over the temperature range from 370 to 436°C. The constancy of ΔE lends confidence in the validity of the theory based on the lattice model.

Vapor pressure measurements by a boiling-point technique are being used to evaluate the activity of CsF in binary melts containing 20 mole % of various alkaline-earth fluorides. Preliminary tests with MgF_2 and BaF_2 indicate that the former depresses the CsF activity to a greater ex-

tent. This is in accord with the concept that the larger the charge-to-size ratio of the alkaline-earth cation, the stronger the interaction with CsF in solution. (AEC Activity 5330)

Analytical Chemistry Research. — The particle-size distribution of ThO_2 and $Th(C_2O_4)_2$ in the range 1 to 8 μ has been determined on pilot-plant samples by a rapid, incremental sedimentation method. Measurements of the natural gamma activity of thorium in samples withdrawn from a specific level at various time intervals are utilized to estimate the fraction of solids remaining at the sampling level. From these data and calculations based on Stokes' law, the size distribution can be established. In making particle-size analysis of ThO_2 in the range 100 to 1000 μ, a similar method is utilized, but prior to the sedimentation test the sample is activated by neutron bombardment. During the sedimentation test, which is carried out in a medium of high viscosity, the concentration of ThO_2 at a fixed point below the surface is established as a function of time by measuring the gamma activity of Pa^{233}.

A method involving the counting of the gamma activity of F^{18} produced by the neutron reactions $Li^6(n,\alpha)H^3$ and $O^{16}(H^3,n)F^{18}$ has been investigated and applied, with some accuracy, to (1) the determination of Li^6 at concentrations as low as 10^{-4} M in aqueous solutions and (2) the analysis of oxide films 1 Å in thickness (0.01 $\mu g/cm^2$).

METALLURGY

Fundamental Investigations of Radiation Damage in Solids. — Comparison of the low-temperature magnetic susceptibility of unirradiated and radiation-damaged silicon yields quantitative information about the density of irradiation produced or irradiation removed magnetic centers. Correlation, or lack of correlation, between those centers and irradiation-induced electrical traps, as well as changes of introduction rate of centers, can then be interpreted in terms of a model of the radiation damage site.

A 0.03-ohm-cm sample (original donor concentration of 6×10^{17} arsenic atoms per cubic centimeter) has now been irradiated up to 4×10^{17} nvt in four steps. After an initial decrease and partial return of the original magnetic center density occurring at irradiations of less than 2×10^{17} nvt, no further change takes place, and the number of magnetic centers remains at about one-half to two-thirds of the original concentration. Measurements of the magnetic susceptibility of n-type silicon as a function of irradiation dose are continuing. (AEC Activity 5420)

PROGRAM 6000 - BIOLOGY AND MEDICINE

BIOPHYSICS

Radiation Dosimetry Programs. The electron flux within a medium under X-irradiation is determined by measuring the derivatives of the current-voltage curve of the electrons emitted by two closely spaced parallel plates in a vacuum. Previously the derivatives were evaluated directly from the curve (Health Phys. 1, 219, 1958). Considerably increased accuracy, sensitivity, and electron energy range are obtained by an a-c method. Two signals of equal amplitude and of frequencies 300 and 324 cps modulate the d-c plate voltage. The beat-frequency amplitude is proportional to the second derivative of the current-voltage curve. First derivatives are obtained by integrating the second derivatives, and both are used to determine the flux (Bull. Am. Phys. Soc. 3, 300, 1958). Fluxes in copper and aluminum have been measured to an energy of about 50 ev. The spectra follow the inverse square dependence on energy noted before (ORNL-2521), to about 20 ev, where they begin to fall off more rapidly. For X-ray energies from 30 to 200 kev, the total yield is proportional to the X-ray energy absorbed in the surface of the plates as calculated from mass absorption coefficients. (AEC Activity 6140)

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