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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

DECEMBER 1958

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

December 1958

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. — A program to check certain biological parameters which are of controlling importance in calculating the maximum permissible concentration (MPC) of uranium was begun. The fraction of an oral dose that is absorbed into the bloodstream from the gastrointestinal tract, f_1, directly controls the MPC in water and is important in estimating MPC in air for soluble compounds. Values of f_1 now appearing in the internal-dose handbooks are 0.0006 and 0.0001 for soluble uranium compounds. A study has been carried out in which enhanced UO_2F_2 was administered as a single oral dose (0.5 mg of U per ml of water solution) to seven mongrel dogs. The total dose for each animal was 0.7 mg of U per kg of body weight. For the compound and the dosage used, f_1 was found to be 0.015 (standard deviation = 0.002). Additional work now in progress includes studying the effect of dose level (0.007 mg/kg), compound (uranyl nitrate), and animal species (rat). (AEC Activity 2120)

Dissolver Solution. — The procedure for the determination of uranium outlined in the October monthly report (ORNL-2636) has been applied to the analysis of synthetic ThO_2- UO_2 fuel (Consolidated Edison reactor fuel). It was found necessary to increase the triisooctylamine concentration in xylene to 20% to obtain quantitative recovery of uranium. The relative standard deviation was found to be 0.3% for solutions containing 11.30 mg of uranium per milliliter. An evaluation study of methyl isobutyl ketone (hexone) as an alternative extraction agent has been started. Polarographic and coulometric titration studies of plutonium in synthetic dissolver solutions have also been started. (AEC Activity 2724)

PROGRAM 4000 — REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. — Run 18, which has as its purpose a continued investigation of fuel stability, is in progress. Initially the reactor was operated at 260°C and 1500 psig on D_2O condensate. In order for the new chemical processing plant entrance line to have ample opportunity to remove solids left in the high-pressure system after run 17, the chemical plant was started with the reactor and remained on stream for 106 hr (see next section, "HRT Chemical Pilot Plant"). For 29 hr during the heating of the system, the temperature was maintained at 150°C since more solids are known to be suspended at that temperature than at higher temperatures.

While the fuel was held in the fuel dump tanks, it was sampled many times so as to minimize the error in the measured inventory of uranium in solution prior to pumping uranium to the high-pressure system. One kilogram of U^{238} was added to the fuel so that the isotopic dilution can be used as an additional means of determining the uranium inventory.

On December 9, the run was interrupted while the chemical plant circulating pump was replaced.

The reactor is currently critical and operating at 260°C and 1500 psig. Zero power operation will continue while the uranium loss, if any, is established, and critical concentrations are being determined at several temperatures. (AEC Activity 4103.3)

HRT Chemical Pilot Plant. — Removal rates for corrosion-product solids in the first 106 hr of operation in run 18-7, with the new entrance line adjacent to the core wall of the reactor, were approximately the same as those during the first period of run 17. The 202 g of corrosion products collected contained 22% Fe, 46% Zr, 3.4% Cr, and 1% Ti. Difficulties have been experienced in determining the amount of uranium associated with the solids. The weight ratio of insoluble uranium to corrosion products collected has varied in the last four runs from 0.17 to 0.65, while the composition of corrosion-product solids has remained quite constant.

Following run 18-7, the circulating pump in the hydroclone system seized and could not be started. The pump was replaced with the cell flooded, and the plant is again in operation in conjunction with the reactor. (AEC Activity 4103.1)

Fuel Processing. — Further tests of the electrolytic method for removing nickel and manganese from simulated HRT fuel solution concentrated to about 60 g of U per kg of D_2O have confirmed that, with a mercury cathode, manganese as well as nickel can be satisfactorily removed. With a stainless steel cathode a slow increase in both nickel and manganese concentration was observed when the uranyl sulfate solution contained about 1 g of each metal per kg of D_2O. When a stainless steel container holding a pool of mercury served as the cathode in an electrolytic cell, nickel was satisfactorily removed from solution, but manganese was not. (AEC Activity 4103.1)

Gaseous Fission Product Disposal. — Studies relating to the ignition and subsequent combustion of charcoal in bed A of the HRT adsorber system during the early part of run 17 were completed. In one group of experiments, hydrogen-oxygen explosions in Columbia G charcoal were initiated, and oxygen was allowed to flow through the charcoal immediately thereafter. On a number of occasions, reaction of the flowing oxygen with hydrogen caused combustion of the charcoal. (AEC Activity 4103.1)

Thorium Oxide Slurry Development: Oxide Preparation. — Seven hundred pounds of mixed oxide of 2.5-μ average particle size and containing 8% uranium was prepared by precipitation of uranium carbonate on 650°C-fired thorium oxide and subsequent firing. The product was fired at 1050°C and

classified to remove oversize (>5-μ) particles. One hundred pounds of thorium oxide was prepared by a 70°C precipitation of thorium oxalate and thermal decomposition at 650°C. In addition, 160 lb of 1600°C-fired, classified thorium oxide (2.6-μ average size) was prepared.

Alteration of flame denitration equipment to permit use of a vertical reflector greatly increased yields of mixed thorium-uranium oxides at temperatures of 800 and 1000°C. Mixed oxides containing molybdenum recombination catalyst were prepared at 1200 and 1450°C with thorium-uranium-molybdenum nitrate solutions in CH_3OH-H_2O as feed. All conditions tested for fluidized-bed denitration of $Th(NO_3)_4$ have produced porous agglomerations of small ThO_2 particles instead of growth of dense ThO_2. (AEC Activity 4103.1)

Thorium Oxide Slurry Development: Slurry Irradiations. — A slurry (500 g of Th per kg of D_2O) of thorium—3% natural-uranium oxide solids from the Westinghouse pump loop (pumped 552 hr at 450°F; argon overpressure) containing a palladium oxide catalyst (122 ppm based on slurry solids) was irradiated in the LITR under a deuterium gas atmosphere. No radiolytic gas pressure was observed. The run was terminated because of a leak after only six days of irradiation. Irradiation in the ORNL Graphite Reactor of a slurry (500 g of Th per kg of D_2O) of thorium—2.8% uranium oxide, prepared with enriched uranium, without a catalyst, and under a D_2 atmosphere, did not produce significant amounts of radiolytic gas (<25 psi) at temperatures above 240°C.

Reaction rates of D_2-O_2 mixtures in a D_2O slurry prepared from the Westinghouse solids decreased from 3.7 moles of D_2 reacted at 200°C to 0.9 mole of D_2 reacted at 280°C per hour per liter of slurry at a partial pressure of 100 psi D_2. This behavior was not materially changed by preheating the slurry in an overpressure of D_2 or argon at 280°C. The effect of varying the initial pressure of reactant gases (D_2 + 1/2 O_2) charged to the reaction vessel from 300 psi to 900 psi at room temperature was also minor. The reaction rate, however, was dependent on the pressure of O_2 in excess of the stoichiometric quantity. For D_2:O_2 ratios of 1:1 and 1.5:1, the reaction rates at 280°C were too rapid to be measured. The rate for a D_2:O_2 ratio of 1.25:1 was 5.1 moles of D_2 reacted per hour per liter of slurry at 280°C and a partial pressure of 100 psi D_2. (AEC Activity 4103.1)

HRP Chemistry: Fuel Studies. — A new method of free-acid analysis was examined. The method involves sorption of cations (including H^+) on a Dowex 50-X8 resin column, initially in the lithium form, followed by elution with 0.05 M lithium sulfate solution. This elution selectively removes the hydrogen ions as free acid in an excess of neutral lithium sulfate. The separated free acid in the column effluent is titrated with standard base. Tests of the procedure with simulated HRT fuel solutions gave accurate results of good precision.

Equilibrium compositions at 350°C of the two immiscible liquid phases in the system UO_3-SO_3-H_2O have been determined. From initial solutions having concentrations of 0.8 to 2 m stoichiometric UO_2SO_4, the resultant light phases show compositions in the range of 0.05 m uranium and contain 30 to 50

mole % excess free acid; the heavy-phase compositions are in the range of 12 m UO_2SO_4 and contain essentially no free acid. The exact final compositions depend on the specific molalities of the initial solutions.

Binary curves at 300°C in the systems UO_3-NiO-SO_3-H_2O and NiO-CuO-SO_3-H_2O have been determined. In the first system the saturating solid phases are $NiSO_4 \cdot H_2O$ and $UO_3 \cdot UO_2SO_4$, whereas in the second system the solids are $NiSO_4 \cdot H_2O$ and $3CuO \cdot SO_3 \cdot 2H_2O$. Skeletal structures at 300°C can be presented for the solid-liquid equilibria in the five-component system UO_3-NiO-CuO-SO_3-H_2O (D_2O) from 0.02 to 0.5 m SO_3.

Solutions having HRT core and blanket fuel compositions and containing approximately 200 psi O_2 overpressure have been heated to 590°C and held at that temperature as long as 18 hr. The second liquid phase (at 340 to 350°C) and the critical phenomena (at 374°C) of the light phase appeared as expected; however, no solids precipitated from the solutions.

Because of the probable existence of temperature gradients and the possibility of local boiling at the HRT core-tank walls, studies were initiated to investigate stabilities of possible HRT solid products to high temperature and to water-vapor pressures of 40 to 170 atm, corresponding to 250 to 350°C water. Preliminary experiments with UO_2SO_4 solid under 60-atm water-vapor pressure indicate almost complete stability at 880°C over a 20-hr period. It is possible, however, that a small amount had decomposed to produce free H_2SO_4, which stabilized the bulk of the solid as UO_2SO_4. Similar experiments with UO_2SO_4 at 880°C in an open tube showed considerable decomposition after 4 hr. (AEC Activity 4103.1)

HRP Analytical Chemistry. — A cathodic stripping technique similar to the method introduced by Zittel et al. (ORNL-2453, p 16) was further investigated as a means of determining microgram quantities of chloride in solutions of uranyl sulfate. For this application an ORNL high-sensitivity polarograph was substituted for the Electronic Coulometric stripper previously utilized by Zittel. When the microelectrode, which consists of a drop of mercury, is made the anode, a quantity of mercurous chloride that is directly proportional to the concentration of chloride in the electrolyte is deposited on the anode. The mercurous chloride is then redissolved by scanning the potential at a constant rate in the negative direction. The peak-height current (I_p) of the voltage-current curve (Anal. Chem. 29, 933, 1957), which is recorded during the dissolution period (cathodic stripping cycle), is directly proportional to the mercurous chloride deposited on the anode and, consequently, to the concentration of chloride initially present. Chloride is, therefore, determined by referring I_p to a calibration curve. A 0.1 M KNO_3, 0.1 M citric acid solution is used for the supporting electrolyte. From 2 to 20 μg of chloride in a final volume of 10 ml can be measured by this method with a coefficient of variation of about 15%. (AEC Activity 4103.1)

Slurry Testing and Evaluation. — Run 200A-18 (200-gpm loop) was undertaken to evaluate the behavior of a mixed urania-thoria slurry of suitable composition for use as fuel in a small, experimental slurry reactor. The

mixed oxide of about 1.5-μ particle size and containing 8.5% uranium (based on the thorium) was prepared by depositing ammonium uranyl carbonate on 650°C-fired thoria and refiring to about 1050°C.

A slurry of about 200 g of Th per liter was circulated at 224°C and 750 psi under oxygen atmosphere for 1026 hr, and the following conclusions have been drawn:

1. There was no noticeable separation of uranium from the thorium.

2. The pH of the slurry was in the range of 4.0 to 5.6, with most values reported being about 5.4.

3. There was no noticeable degradation of the particles, and the generalized attack rate was less than 0.1 mil/year.

4. There was no visual evidence of attack on any parts of the pump, including the titanium impeller.

5. The behavior of the slurry was generally comparable in all other respects to that of other degradation-resistant compositions.

(AEC Activity 4103.1)

Slurry Blanket System Development. — Preliminary tests to determine the behavior of thoria fluidized beds have been concluded after about 500 hr of circulation time at a bed density of 2900 g of Th per liter. The low attrition rate for the 40-μ thoria spheres (Houdry process) previously reported (0.26 wt % per day) was confirmed. The following conclusions have been drawn:

1. Dense thoria spheres (20 to 60 μ) do not degrade significantly under liquid fluidization conditions at 250 to 300°C.

2. The small particles carried over from the bed can be removed by hydroclones.

3. The nonuniformly sized particles and possibly the nonuniform flow distribution caused a moderate density gradient from top to bottom of the fluidized bed.

4. Further work on the flow distributor is needed.

(AEC Activity 4103.1)

Engineering Research. — A low-temperature loop is being built for determining the average fluid velocity required to maintain thoria-water slurries in suspension. The test sections have pipes which are 2 and 4 in. in diameter. Because of difficulties expected in removing gas from the loop, an 8-in.-dia 3-ft-long vertical pipe was installed to act as a gas-separator section. Preliminary tests indicate that gas bubbles are adequately removed in this section.

The capillary tubes of the high-temperature (200°C) vertical-tube viscometer were successfully calibrated with mercury, water, and Karo syrup in laminar-flow tests at room temperature.

Several 30-gpm loop runs were completed. A slurry containing 65 g of Th per kg of H_2O (thoria from batch LO-20) did not cake when circulated at 280°C; this is in contrast to the caking behavior observed when the thoria concentration was about 500 g of Th per kg of H_2O. At 45°C, however, caking was not observed during circulation of the 500-g/kg slurry. (AEC Activity 4103.1)

Core and Blanket Vessel Development. — Based on experimental flow measurements, it was calculated that the core-wall temperature of an HRT replacement vessel of the re-entrant type could be maintained below the fluid temperature by means of cooling from the blanket side. A re-entrant system which satisfied all other known criteria for use in the HRT was selected for this application.

Additional runs were made with the alternate vaned-annular-inlet configuration. It was found that a stable flow system with a strong, cool boundary was achieved when most of the inlet flow was bypassed directly to the opposite pole. Although this concept is not useful for application in the HRT, because the internal volume is not cooled properly, for a large reactor the concept has some promise if a larger flow can be utilized to cool the interior without disturbing the boundary layer.

Runs were made in the model of the present HRT core tank to investigate possible revisions. Preliminary results indicate that substantial improvements can be attained in wall cooling and solids removal if the screens are removed; flow may be either forward or reversed.

On the basis of water runs with a 30-in. cone-bottom slurry core model, more favorable wall shear conditions are obtained with re-entrant flow than with straight-through flow. The optimum location of the inlet nozzle was found to be at the projection of the spherical surface into the cone. (AEC Activity 4103.1)

Centrifugal Circulating Equipment. — The 300A slurry loop began routine operation. The pump components being tested in the first run consist of various vane shapes in the titanium impeller, and mechanically mounted aluminum oxide bearings.

Performance curves were obtained for the 200Z pump, which contains a hydraulic design believed to be the best obtainable with current knowledge of slurry pumps. The performance was consistent with that expected in design. The pump stator later failed electrically, apparently as a result of long-term deterioration, and it requires repair. (AEC Activity 4103.1)

Miscellaneous Equipment. — Specimen recovery operations at the HRT were concluded. In all, 12 of 22 corrosion samples were recovered; the balance are inaccessible in the screen diffuser system.

A dip tube for removing a continuous stream from the core to the HRT chemical plant was developed, along with the procedures to permit its installation.

In order to improve the usefulness of future work in the Y-12 maintenance mockup of the HRT core and pressure vessel, a dummy shield plug was designed and is being fabricated. A device for positioning an ultrasonic core-thickness-measuring crystal at any core location was designed, and fabrication was started. Development work was started also on the problems of removing the HRT screens, plugging the core defect, and cleaning up solid deposits. (AEC Activity 4103.1)

Heat Removal Equipment. — Manufacturing release was granted to Griscom-Russell in order that they might proceed with the fabrication of carbon-steel components of their test steam generators. An order was placed for Inconel—stainless steel composite tubing for one or two of the test units. The rest of the tubing failed a chloride test and requires further cleaning.

Tests of a plastic inverted-L slurry heat exchanger with flocculated slurry were completed in the concentration range 200 to 1000 g of ThO_2 per liter. No difficulties were experienced with flow distribution or plugging. Tests of a dispersed slurry in the same system are planned. (AEC Activity 4103.1)

Gas Handling Equipment. — After 180 hr of satisfactory operation, the natural-circulation high-pressure recombiner was shut down because of the failure of a Bourdon tube which had been in use in this system for several years. The tube is being replaced. (AEC Activity 4103.1)

Fuel Systems Development. — The HRT mockup, containing a titanium let-down heat exchanger and a titanium pressurizer in which the uranyl sulfate is boiled, was operated for 520 hr since the last report period (total time now 2246 hr). During operation at a pressurizer temperature of 330°C (the two-liquid-phase formation temperature), the uranium and copper concentrations in the light phase dropped and the free acid rose slightly, thus indicating formation of the heavy liquid phase. The copper and uranium concentrations and free acid returned to normal when the temperature was reduced to 321°C, as was expected upon disappearance of the heavy phase.

The stainless-steel-to-titanium transition joints still showed no visible corrosion after the above operation, although the estimated generalized stainless steel corrosion rate (based on iron increase in solution) was about 2.3 mils/year, as compared with 1.3 mils/year during operation below the two-phase formation temperature. (AEC Activity 4103.1)

Feed Pump Development. — Prototype HRT fuel feed pumps have operated for more than 19 months and prototype purge pumps have operated the equivalent of 19 months without difficulty. After 13,000 hr of operation on the HRT mockup, an experimental 1/4-hard 0.021-in.-thick diaphragm failed. Increased-capacity solution diaphragm heads of the double diaphragm and of the larger contour types have continued to operate satisfactorily.

The duplex-diaphragm slurry feed pump has operated for about 800 hr with aluminum oxide valve trim, although there is evidence that the trim is deteriorating.

The three-stage contaminated-service oxygen compressor passed a 250,000-cycle test at the factory, compressing 2.9 scfm of atmospheric air to 2500 psi. The compressor is now being set up at ORNL for long-term testing. (AEC Activity 4103.1)

MOLTEN-SALT REACTOR (MSR) PROGRAM

Design. — Design work on a 30-Mw (thermal) molten-salt test reactor was initiated. The reactor core of this design is 6 ft in diameter and the cell is 46 ft high. The fuel enters the core from the heat exchanger through an 8-in.-dia line, exits from the core to the pump, and returns to the core through the fuel-to-sodium heat exchanger. The tubes in the tube-and-shell heat exchanger are helical.

The layout of this design provides for semiremote maintenance. The heat exchanger and its headers and the reactor portion of the off-gas system can be pulled out of the cell at the top. Similarly, the fuel pump and the gas heating and cooling system can be removed from the top. The fuel valves, the sampling system, the fuel-level-indicating mechanism, and some components of the off-gas system external to the reactor may also be removed.

The reactor shell is double everywhere except around the heat exchanger to provide an annulus for preheating the system and for removing afterheat. A dip line is provided to transfer the fuel from the core to the drain tank. Several types of fuel pumps are being considered. (AEC Activity 4103.1B)

Nuclear Calculations. — Investigations of nuclear characteristics of graphite-reflected and -moderated reactors were continued. For nonblanketed, heterogeneous, graphite-moderated reactors fueled with thorium-bearing fully enriched U^{235} salts, initial Oracle calculations show critical mass and breeding ratio combinations that range from a critical mass of 21 kg and a breeding ratio of 0.34 to a critical mass of 79 kg and a breeding ratio of 0.66. With a low-enrichment ($\sim$1.5%) non-thorium-bearing U^{235} salt, the critical mass was 440 kg and the breeding ratio was 0.82. Optimum combinations of the variables are being sought.

The Cornpone program is being used to compute the nuclear characteristics of a spherical, graphite-reflected reactor. For these calculations it is assumed that the core salt is compatible with the graphite. The most promising system found thus far has a core 10 ft in diameter and the fuel salt contains 2 mole % ThF_4. This reactor has an initial critical inventory of 550 kg of U^{235} and an initial regeneration ratio of 0.64. (AEC Activity 4103.1B)

Physical Properties of Molten Salts. — The viscosities of two beryllium-containing fluoride salt mixtures, $LiF-BeF_2-UF_4$ (62-37-1 mole %) and $LiF-BeF_2-$

UF_4-ThF_4 (62-36.5-0.5-1 mole %), were experimentally determined over the temperature range 500 to 800°C. The conventional apparatus was modified for these measurements to provide a skirt around the upper portion of the cup which allowed excess salt on the outside surface of the cup to run off without interfering with the discharge through the capillary. (AEC Activity 4103.1B)

Experimental Engineering. — Operation of the first molten-salt forced-circulation loop was started in the MTR. The fission heat is about 9 kw, which gives a power density of 66 w/cm^3 for the total fuel inventory. The high-temperature end of the loop is at 1300°F, and the Reynolds number for the salt flow is 4600. More than 300 hr of operation with the reactor at power has been accumulated. The assembly of the second in-pile loop is approximately 50% complete.

Large freeze flange joints with aluminum seal rings, which had leakage rates of less than 6×10^{-11} cm^3 of helium per second while cold, were found, after being thermally cycled 12 times between 1100 and 1300°F, to have maximum leakage rates of 9.4×10^{-8} cm^3 of helium per second. The specified maximum leakage rate is 10^{-7} cm^3/sec.

A molten-salt-lubricated hydrodynamic journal bearing made of INOR-8 was operated for a scheduled 1000 hr at steady-state conditions of 1200°F, 1200 rpm, and a load of 200 lb. The bearing was in good condition after the test. A centrifugal pump has operated continuously for more than 13,000 hr with a fluoride salt at 1200°F, a shaft speed of 2700 rpm, and a flow rate of 645 gpm. A similar pump equipped with a Fulton-Sylphon bellows seal has operated continuously with NaK for more than 10,000 hr, at 1240°F, a shaft speed of 2500 rpm, and a flow rate of 1200 gpm. A small frozen-lead-sealed pump, consisting of a centrifugal pump mounted vertically over a fractional horsepower motor drive, has operated continuously in accumulating over 4500 hr of operation. The pump is circulating a fluoride salt at 1200°F.

Fifteen forced-circulation corrosion-test loops are in operation. Operation of an Inconel loop was terminated, as scheduled, after 8800 hr. Of the 15 loops in operation, 11 are INOR-8 and 4 are Inconel. Operating times of the individual loops range from 8300 hr down to 800 hr. Operation of six Inconel and three INOR-8 thermal-convection loops was terminated after a year of testing. Three Inconel loops and two INOR-8 loops were started up, and thus 26 thermal-convection loops are in operation. (AEC Activity 4103.1B)

Metallurgy. — Data on creep to 1% strain were obtained for INOR-8 rod and sheet specimens at 1250°F and stress levels of 10,000 to 20,000 psi. The data indicate that the creep strengths of rod and sheet are similar at 15,000 psi but that rod material is stronger at higher stresses and sheet material is stronger at lower stresses. The activation energy for INOR-8 creep in a fluoride salt environment was calculated to be 76,670 cal·mole^{-1}·(°K)$^{-1}$.

Triplex tubes consisting of porous nickel cores metallurgically bonded on either side with solid tubes were fabricated under various conditions to

determine the variables which affect density, permeability, and heat transfer. The final core density and the permeability were found to vary with initial powder particle size and appeared to be insensitive to sintering time and temperature.

Equipment and procedures were developed for attaching tubes to tube sheets in heat exchangers utilizing triplex tubing and double tube sheets, and a seven-tube mockup was fabricated. The high-temperature ductility of INOR-8 weld filler metal was improved at 1200°F from 17% to 30% and at 1500°F from 7% to 15% by adjusting melting practices to deoxidize and purify weld rod metal. Lowering the weld metal carbon content did not improve the ductility of INOR-8 weldments.

INOR-8 and Inconel specimens exposed to a sodium-graphite environment at 1200°F for 4000 hr were found to be slightly carburized, as indicated by mechanical properties changes and chemical analysis.

Metallurgical examination of two Inconel thermal-convection loops that circulated fluoride salts showed that impurities in the salt increased the corrosion rate. The NaF-LiF-KF mixture which had circulated for 4673 hr at 1250°F and which caused 13 mils of hot-leg attack was found after the test to contain $KF \cdot 2H_2O$. The other loop operated at 1350°F for 1000 hr with an especially purified $LiF-BeF_2-UF_4$ mixture, and the hot-leg attack, rather than being the expected 3 mils of moderate void formation, was light and to a depth of 2 mils.

A forced-circulation loop was examined that had operated for 9148 hr at 1260°F with $NaF-ZrF_4-UF_4$ that was circulated through a sodium-cooled heat exchanger. The salt circuit showed only light surface roughening and attack to a maximum depth of 1.5 mils. There was no evidence of mass transfer in the sodium circuit. Six of the presently operating forced-circulation loops have now been operating for more than one year. (AEC Activity 4103.1B)

Reactor Chemistry. — The vapor pressure of liquid UF_4 was measured between 4 and 180 mm Hg in connection with a continuing study of the thermodynamic properties of fuel mixtures. An equation for the vapor pressure was derived from the data obtained in the temperature interval 1030 to 1300°C that is in agreement with an existing expression for the sublimation pressure.

In an effort to predict corrosion rates of chromium-containing alloys in contact with molten fluorides, experiments were performed to determine the self-diffusion coefficients of chromium in Inconel. The results show that the diffusion coefficients determined for Inconel specimens previously annealed at 1150°C are 25 to 100 times lower than the corresponding coefficients obtained for unannealed or as-received specimens. The annealed specimens show considerable grain growth in comparison with the as-received specimens. Accordingly, the self-diffusion rates appear to be, at least at temperatures below 800°C, intimately related to the number of intergranular channels in the alloy specimen.

Thermal-gradient quenching experiments have made possible the location of three invariant points in the system $NaF-BeF_2-ThF_4$. The eutectic involving the solids NaF, $2NaF \cdot BeF_2$, $2NaF \cdot ThF_4$, and a liquid occurs at 509°C, and the composition is 72 mole % NaF–22 mole % BeF_2–6 mole % ThF_4. The ternary peritectic involving the decomposition of solid $3NaF \cdot 2ThF_4$ into $2NaF \cdot ThF_4$ and $NaF \cdot ThF_4$ has the composition 62 mole % NaF–3 mole % BeF_2–35 mole % ThF_4. The agreement of the decomposition temperature with that of $3NaF \cdot 2ThF_4$ in the binary system indicates the absence of appreciable BeF_2 solubility in the compounds $2NaF \cdot ThF_4$, $3NaF \cdot 2ThF_4$, and $NaF \cdot ThF_4$. The peritectic involving the three solids $2NaF \cdot BeF_2$, $2NaF \cdot ThF_4$, $NaF \cdot ThF_4$, and a liquid occurs at 415°C, and the composition is 57 mole % NaF–42 mole % BeF_2–1 mole % ThF_4.

A simple apparatus for sampling circulating fluids was tested on a molten-salt forced-circulation loop constructed of INOR-8. Fifteen samples of the $LiF-BeF_2-ThF_4-UF_4$ melt were removed during nearly 1000 hr of loop operation. The chromium content of the circulating fluid increased in 800 hr from 400 to 500 ppm and appeared to be increasing at a slow rate.

In the preparation of pure fluorides, chromous fluoride was found to remain as the only nonvolatile product when small (less than 40 g) samples of CrF_3 were heated at 1100°C in an inert atmosphere for 4 hr. The yield of CrF_2 was consistent with the equation $3CrF_3 \rightarrow 2CrF_2 + CrF_5$.

The reaction of molten SnF_2 with active metals also appears to be a useful general method for preparing pure fluorides. Molten tin separates readily from, and is virtually insoluble in, the fluorides so prepared, while excess SnF_2 is removable by volatilization. The fluorides MnF_2, ZnF_2, FeF_2, AlF_3, and UF_4 have been prepared by this process. (AEC Activity 4103.1B)

CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR

Physics. – The fast-neutron flux at the pressure vessel of the Kaiser-ACF prototype reactor (GCPR) was calculated for three different reflector thicknesses with the use of the results of moments-method calculations by NDA. The data showed clearly that a few inches variation in reflector thickness can make the difference between an acceptable and an unacceptable fast-neutron exposure to the pressure vessel. (AEC Activity 4110)

Fuel Element Fabrication and Inspection. – Procedures for the fabrication and inspection of solid and cored UO_2 pellets for in-pile fuel capsule tests are now fully developed, and the pellets are being produced as required. Solid beryllium oxide rods of low bulk density (~49% of theoretical) are also being made available as needed for use as core rods in fuel capsules. Satisfactory capsule and closure techniques were developed on the basis of results of burst tests, and fuel capsule assemblies are being completed on schedule. Materials for ETR, ORR, and LITR experiments are now available routinely.

Two lengths of extruded 3/4-in. beryllium rod, 4.5 and 7.5 ft long, were received from Nuclear Metals, Cambridge, Mass. Techniques are being developed for the inspection of this material, which is to be used for end plugs in beryllium-clad fuel elements. (AEC Activity 4110)

Materials Compatibility. — Metallographic examinations of low-alloy steels, Croloy (2.25% Mo) and Croloy (3% Mo), which were exposed to helium and graphite for 2100 hr at 1400°F showed no apparent surface changes. The T-1 steel exposed under the same conditions was decarburized to a depth of 1 mil. Similar specimens exposed for 1000 hr at 1000°F to CO_2 developed heavy oxide surface films that varied from 1 to 2.5 mils thick. There was no carburization or decarburization. (AEC Activity 4110)

Materials for Critical Experiment. — Inspection of 1250 ft of the 3/4-in.-OD, 0.020-in.-wall, type 304 stainless steel tubing purchased for the critical experiment at Hanford was completed. Most of the tubing specified with a tolerance of 0.002 in. was within the tolerance, but only one of the ten pieces specified with a tolerance of 0.001 in. was satisfactory.

The 2% boron—stainless steel sheet obtained for the fabrication of the control rods was found to be formable. The microstructure of a weld zone is being examined to determine the successfulness of the proposed joining technique.

Much of the required enriched uranium foil was completed and shipped to Hanford. The fabricator has completed about one-third of the first loading of UO_2 pellets. (AEC Activity 4110)

Radiation Effects Studies. — Construction work continued on the equipment needed for the evaluation of fuel capsules irradiated in the LITR and the ORR. Attempts to open the irradiated capsules sent to General Electric at Vallecitos for examination in the hot cells there revealed that the available handling and disassembly equipment was not suitable. New equipment is to be developed by G.E.

Irradiations were continued of SiC-Si coated graphite specimens. X-ray photographs of coated-graphite-canned ThO_2-UO_2 pellets received from the Minnesota Mining and Manufacturing Company showed that the pellets were broken during the canning process. This will not prevent the use of the pellets for evaluating the fission-gas retention properties of the coated graphite cladding.

Preliminary measurements of Chromel and Alumel wires irradiated for three weeks in the ORR indicated very little change in properties. (AEC Activity 4110)

Graphite Degassing Studies. — Preliminary experiments at temperatures up to 1000°C have indicated that the percentage of hydrogen in the gas evolved from TSF graphite increases with time, while the percentages of water and carbon dioxide decrease. Tests with AGOT graphite showed that the rate of gas evolution per unit volume decreases as the volume of graphite increases. The apparatus for tests at 1800°C has now been completed. (AEC Activity 4110)

Heat Transfer Experiments. — Preliminary runs were made in an apparatus for determining fuel-element hot-spot location and magnitude. In this apparatus a fuel element cluster is heated in a graphite channel. The preliminary data show a maximum circumferential temperature difference of 32°F with

a surface heat flux of 8200 $Btu \cdot hr^{-1} \cdot ft^{-2}$ and a Reynolds modulus of approximately 70,000. The surface temperature differences around the central tube were low, and the profile appeared to vary with axial position. (AEC Activity 4110)

Design Study of a Contaminated-Coolant Gas-Cooled Reactor. — The preliminary design of a 3095-Mw (thermal), helium-cooled, graphite-moderated reactor employing graphite-UO_2 fuel elements was completed. At design conditions, 1500°F reactor outlet gas would be circulated to eight steam generators to produce 1050°F, 1450-psi steam, which would be converted to electrical power in eight 157-Mw (electrical) turbogenerators. The overall efficiency of the nuclear power station is 36.5%. The significant activities released from the unclad graphite-UO_2 fuel appear to be less than 0.2% of those produced and would be equivalent to 0.002 curie/cm^3 in the primary helium circuit. The maintenance problems associated with the contamination level were taken into consideration. A cost analysis indicates that the capital cost of this nuclear station per electrical kilowatt would be around $220 and that the production cost of electrical power would be 7.8 mills/kwhr. (AEC Activity 4110)

Applied Mechanics. — Equations for analyzing a support grid structure made up of a rectangular pattern of beams were modified to include shear effects, along with those of bending and torsion. These equations will be used in the analysis of an arrangement of beams in which each member supports a uniformly distributed load of the same magnitude and each beam is simply supported at the ends. The individual beam cross sections are restricted only by a requirement for symmetry about the two center lines. Thus an optimum selection of beams for a specified configuration can be made. An IBM 704 code was written for the solution of the equations. In addition, an IBM 610 code was written for analyzing a square system of beams in which only the bending rigidity is included. (AEC Activity 4110)

Design of a GCR-ORR In-Pile Loop. — An in-pile test facility is being designed with which to investigate the heat transfer characteristics of gas-cooled fuel elements under conditions up to twice the peak conditions of GCR-II. This indicates that the facility should be able to accommodate single fuel-element heat loads up to approximately 40,000 $Btu \cdot ft^{-2} \cdot hr^{-1}$. For a two-lattice-position configuration, with a 20-in. test element in each leg, a total load of about 40 kw can be expected. Rod surface and gas coolant temperatures to be investigated will range from GCR-II conditions upward through a range of values of interest in advanced gas-cooled reactor concepts to limits governed by considerations of site and economics. (AEC Activity 4110)

Design Review and Power Plant Analyses. — Detailed reviews were completed of all phases of the Kaiser-ACF title I design and the reports to the AEC prepared by Kaiser and ACF. (AEC Activity 4110)

MARITIME SHIP REACTOR PROGRAM

Maritime Ship Reactor. — The Laboratory continued to furnish technical assistance to the Maritime Reactors Branch (AEC) in the program to develop

nuclear-powered merchant ships. Review of drawings and specifications for the NS "Savannah" reactor system continued, with emphasis on fuel element design and safeguards considerations.

Laboratory field inspectors continued to represent MRB at the Barberton, Ohio, plant of Babcock & Wilcox, where the primary-system components are being fabricated. Arrangements were completed for assignment of resident inspectors to the Nuclear Facilities Plant of Babcock & Wilcox during fabrication of fuel elements and other core components, which will begin in January 1959. Additional field inspections were performed on other components, including condenser tubing, primary pumps, primary stop valves and check valves, heat exchangers, and pressurizer heaters.

Deboronization studies of type 304 stainless steel tubing which initially contained 240 ppm boron and studies of the incorporation of boron into UO_2 were terminated because burnable poison is no longer included in the NS "Savannah" reactor design. Out-of-pile experiments on incorporation of small-diameter wires of various metals into UO_2 pellets to serve as temperature monitors during irradiation were completed. Nickel, gold, vanadium, and niobium did not react with UO_2 under conditions of these experiments and appear to be sufficiently promising to merit further tests in pellets under irradiation. Electroless nickel is being investigated as an alternate brazing alloy for fuel bundles. Results obtained in the fabrication of a 16-element tube bundle with the use of electroless nickel for joining indicate that this alloy has excellent brazing characteristics.

Design and procurement for the pressurized-water loop to be installed in the ORR are progressing as scheduled. Building alterations are approximately two-thirds complete. A stress analysis of the in-pile section of the loop was completed. (AEC Activity 4151)

GENERAL REACTOR RESEARCH

Fuel Element Development. — Irradiation testing of the ORR aluminum plate-type fuel element containing 20% enriched uranium incorporated as a dispersion of 37 vol % U_3O_8 in aluminum is still in progress. The test element has performed satisfactorily through eight weeks of operation.

Exploratory experiments to determine the compatibility of UN-Al dispersions have revealed considerable reaction after heat treatment for 5 hr at 600°C. Strong evidence of intermetallic U-Al compounds and slight indication of uranium oxides were observed by x-ray diffraction. The specimens warped and buckled during this treatment. It was shown that when UN is exposed to air at 400°C, it reacts to form U_3O_8.

Additional results on losses of boron—stainless steel material prepared by powder metallurgy have substantiated results reported previously. Prior results had shown that compacts prepared with a 0.25 wt % B—type 304L stainless steel master alloy exhibited losses as high as 40% when treated 1 1/4 hr at 1150°C under hydrogen. Similar experiments with a 0.3 wt % B—type 347 stainless steel master alloy revealed losses in the same order of magnitude. (AEC Activity 4203)

Power Reactor Fuel Reprocessing: BORAX-4 Flowsheet. — The alloy M-388 (99% Al—1% Ni) dissolves in solutions of NaOH, NaOH-$NaNO_3$, and HNO_3-$Hg(NO_3)_2$ at essentially the same rate as pure aluminum. Use of any one of these solutions for decladding BORAX-4 fuel promises to result in low uranium losses. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Corrosion Studies. — Titanium steam tubes exposed to Darex dissolver solutions at a metal surface temperature of about 135°C for 660 to 1040 hr showed some roughening in solutions containing very small amounts of dissolution products; one fissured area was observed in the solution containing no dissolution products. Periodic removal of dissolver scale with 10% NaOH has shown no deleterious effect.

In the vapor phase above boiling 6 M H_2SO_4, Ni-o-nel corroded at about 0.8 mil/month over 122 hr, but in the vapor phase of an active dissolver this rate increased to about 2 mils/month. Liquid-phase specimens showed about the same rate in the active dissolver as in the static tests (~1.9 to 2.4 mils/month), except that the rate dropped to 0.6 mil/month when specimens were in contact with dissolving stainless steel. Of various heat treatments tried on Ni-o-nel, heating at 1850°F for 1/2 hr followed by air quenching gave the lowest rate of attack in Thorex solutions. Rates were 0.7 and 0.4 mil/month, respectively, for 500-hr exposures in initial and final Thorex solutions. Intergranular attack amounting to about 7 mils/month maximum was observed in the initial Thorex solution at the edge of welded areas. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Darex Process. — The first-extraction-cycle Darex-Redox runs were completed in the Building 3503 Interim-23 equipment. The Hanford-supplied Darex feed was spiked with irradiated Graphite Reactor slugs. When salting strength was reduced, gamma decontamination factors of 200 to 500 were achieved, but uranium and plutonium losses increased. In one run in which silicon content was high, flooding occurred at normal flow rates; reduction of all flow rates resulted in smooth operation. The height equivalent to a theoretical stage for uranium extraction was tentatively found to be 12 ft in tests using 2-in., glass-packed columns.

Second-cycle feeds were prepared from the uranium products resulting from the runs discussed above (and from Nyflex-Redox first-cycle solvent extraction product). Results of laboratory-scale batch equilibrations, simulating the second-cycle extraction and scrubbing operations on these feeds, indicated that extractable ruthenium from the first cycle was the predominant fission product comtaminant in the uranium product from the second cycle. In these equilibrations, gross gamma decontamination factors varied from 2 to 107.

In five chloride removal runs made with 10-liter batches of APPR dissolution product, chloride was reduced to 150 to 650 ppm by adding (in two batches) only that amount of HNO_3 normally consumed in dissolution and lost to the solvent extraction feed. These runs also showed that either an updraft or downdraft condenser can be used if an air sparge is maintained during the reflux or chloride decomposition step. A sparge should not be used during the recovery of mixed acid since it contributes to the loss of recoverable chloride.

Laboratory runs demonstrated the feasibility of an alternate method similar to the one above but giving better chloride removal. In this method, the 13 M HNO_3 was added and recycled in a single batch instead of in the normal two-batch additions. However, the advantage of increased chloride removal is offset by increased equipment requirements and more complex operation.

In a single continuous dissolution run, made in a 6-in. glass dissolver with a downdraft condenser, a prototype stainless steel APPR fuel element weighing 4.64 kg was dissolved in 15 min with 2 M HCl–5 M HNO_3 feed. A dissolver product loading of 87 g/liter was produced at a dissolvent feed rate of 4.5 liters/min. The apparent dissolution rate based on the initial surface area of the element was 18.2 $mg \cdot cm^{-2} \cdot min^{-1}$.

The Hanford assistance effort was continued on the demonstration of a Redox first-cycle flowsheet in the cold glass solvent extraction equipment in Building 3503. Two additional Darex flowsheets were investigated, each having relatively high silicon contents of 0.011 and 0.016 M. The flooding rate decreased by about a factor of 2 from the approximately 600 $gal \cdot hr^{-1} \cdot ft^{-2}$ previously reported to 333 and 295 $gal \cdot hr^{-1} \cdot ft^{-2}$, respectively. The values of the height equivalent to a theoretical stage for standard and Darex flowsheets reported previously were high, due to inadequate equilibrium data. Such data have since been obtained, and all calculated values were in the range of 1 to 2 ft. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Engineering Equipment Studies. – Sand filtration equipment has been put in operation to study the removal of SiO_2, MoO_3, and other solids present in fuel dissolver solutions. Studies last month were devoted to the removal of SiO_2 from Darex solution. Tentative results indicate that filtration rate is increased by use of vacuum, higher temperatures, and filter aid. The solution at 20°C had an average filtration rate of 41, 30, and 24 $gal \cdot ft^{-2} \cdot hr^{-1}$ for 1, 2, and 3 hr, respectively. The clear filtrate contained 10 ppm Si, which produced no emulsion on being contacted with 30% TBP–Amsco. A maximum filtration rate was obtained with an addition of between 1.5 and 2 volumes of filter aid per volume of wet silica cake in the feed solution. Average filtration rates of from ~20 to more than 100 $gal \cdot ft^{-2} \cdot hr^{-1}$ can be obtained, depending on the temperature and pressure used and the amount of filter aid added.

A 0.4-in.-dia hydroclone was unsatisfactory for either partial or complete separation of silica from Darex solvent extraction feed solution. The turbulence and shear forces involved converted the undersized silica particles into a difficultly filterable gel.

Preliminary studies were begun on the suspension of oxide fuel pellets and fines during chemical decladding operations. Sparging of ferrosilicon (specific gravity, 9 to 10; average particle size, 100 μ) resulted in the suspension of 90 g/liter 1 in. above the settled bed. Since >250 g/liter of Consolidated Edison ThO_2-UO_2 solids would be required to produce a criticality hazard in noninfinitely safe parts of the equipment, no trouble is anticipated for fines >100 μ. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Hermex Process. — A single extraction of 53.13 g of a uranium-thorium mixture, simulating thorium irradiated to 4000 g of uranium per ton, by 200 ml of boiling mercury reduced the thorium-uranium ratio from 230 to 4.55. This corresponds to a uranium concentration factor of about 50. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Mechanical Processing of Fuels. — The design of the SRE fuel decladder was completed and submitted for fabrication bids. The low bid was $9000. Preliminary design of other mechanical processing and leaching equipment was begun.

Visits to Hanford and Idaho were made which assisted in the writing of criteria and specifications for a shear for spent fuel bundles. Preliminary shear studies at ORNL showed that a line contact shear essentially closes the ends of fuel-bearing tubes in a fuel assembly.

A prototype fuel element has been designed that will be substituted for the seven fuels considered for chopping and leaching studies. This basic fuel bundle (3 7/8 x 3 7/8 x 72 in., consisting of 36 stainless steel tubes 35 mils thick, and filled with porcelain rods) will be used in the experimental disassembly and shearing studies.

Short-time (~2-min) dissolutions of UO_2 in 10 M HNO_3 gave a solution containing ~24.4 g of uranium per liter. Based on the initial surface area of smooth-surfaced right-cylinder pellets, an average dissolution rate of 60 $mg \cdot cm^{-2} \cdot min^{-1}$ was obtained. The partially dissolved pellets were extremely porous and had considerably more surface area than the original pellets, which indicated that the actual reaction rate was much lower than the calculated value. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Nyflex System. — The first-extraction-cycle Nyflex-Redox runs were completed in the Building 3503 Interim-23 equipment with Hanford-supplied Nyflex feed solutions spiked with Graphite Reactor fuel. Five runs gave gross gamma decontamination factors of 20 to 50. Uranium and plutonium losses were acceptable. Later runs, in which salting strength was reduced, gave gross gamma decontamination factors of 60 to 240, but uranium and plutonium losses were unacceptable. Feed instability was observed in some of these runs. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Sulfex-Consolidated Edison Flowsheet. — Four Sulfex-Consolidated Edison laboratory decladdings and dissolvings were made with fuel samples irradiated from 100 to 4300 Mwd/metric ton. The solutions and solids obtained are now being analyzed for product losses. The results from these four runs were inconclusive regarding losses to the decladding solutions because these losses appear to have been caused by fluoride contamination. The fluoride contamination probably was introduced by formation of insoluble fluoride-containing materials during a cleanup dissolution of Kaowool end plugs in 2 M HF prior to the decladding operation. A precipitate which was insoluble in Thorex solution was recovered from each decladding solution. Uranium losses to this precipitate were less than 0.003% in all runs.

Synthetic Consolidated Edison solvent extraction feed solutions containing 0.03 M rare earths plus yttrium, 0.017 M zirconium, and 0.006 M ruthenium were decontaminated by more than a factor of 10^3.

The solubilities of H_3BO_3, $Na_2B_4O_7$, $CdSO_4$, and $Cd(NO_3)_2$ in several process solutions were determined. Boron is soluble to the extent of 0.2 to 0.4 M in 4 to 6 M H_2SO_4 and in the dissolver solution. Boric acid in concentrations up to 0.1 M had no effect on the dissolution of ThO_2-UO_2 pellets in boiling 13 M HNO_3–0.04 M NaF–0.04 M $Al(NO_3)_3$. These tests were made to evaluate further the use of soluble poisons for head-end criticality control. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Zirflex Flowsheet. – Sixteen Zirflex decladdings using NH_4F and two decladdings with HF were made on PWR sample fuel pins irradiated from 250 to 2500 Mwd/metric ton. In each case the cores were dissolved in 10 M HNO_3 after the decladding step. All analytical results are presently unavailable; however, on the basis of preliminary results, uranium and plutonium losses to the decladding solution are not appreciably affected by irradiation up to 2500 Mwd/metric ton. (AEC Activity 4301)

Fused Salt–Fluoride Volatility Processing: Hot-Cell Operations. – Hot-cell equipment is being tested for processing fully irradiated STR fuel by the volatility process. Several cold STR fuel (1-kg batches) practice runs were made to "shake down" the equipment and to develop remote operation techniques. Development is centered on the modified LiF-NaF flowsheet, that is, where the STR zirconium fuel is dissolved by hydrofluorination at 650 to 700°C in 58-42 mole % LiF-NaF and reaches the final composition of 32-23-45 mole % LiF-NaF-ZrF_4. Fluorination and volatilization of UF_6 from the fused salt is then possible at 450 to 500°C.

Positive indications of nickel deposition have been observed in the STR fuel dissolution runs. This is presumably due to either of two mechanisms: the reduction of NiF_2 in the fused salt by the hydrogen produced in the zirconium dissolution, or reduction by the zirconium metal. The chemical reactions are

$$NiF_2(soln) + H_2(g) \longrightarrow Ni(s) + 2HF(g)$$

$$2NiF_2(soln) + Zr(s) \longrightarrow 2Ni(s) + ZrF_4$$

It is believed that both reactions tend to reduce nickel corrosion during the dissolution procedure. This seems particularly true in the case of the second equation if metal contact between fuel and container establishes an electrochemical cell. The ΔF_r per fluoride equivalent for the second equation is -32 kcal at 1000°K. (AEC Activity 4302)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes by Sintering. – Since relatively low temperatures (850°C) are used to produce sinters from clay-waste mixtures, the reactions of the system require a considerable period of time for completion. One of the ways

to increase the rate of formation of sodium aluminosilicate is to use certain fluxing agents. Boric acid was selected to serve as a flux and to substitute for Conasauga shale in preparing sinters from nonradioactive neutralized Purex waste. The boric oxide content ranged from 0 to 17%. The optimum concentration of boric oxide for this clay, based on the sinter's ability to retain cesium, was 8.5%. The effectiveness of boron in this system is due to its ability to serve as a network former, like silicon but at a lower temperature. The boron hastened the formation of the aluminosilicate network by entering into the network itself. If, however, the boric oxide content is too high, the network will contain an excessive number of boron links; cesium would be expected to leach more readily under these conditions. The results show that with 17% B_2O_3 the network is weakened, as is evidenced by the higher content of alumina and silica in the leaching solution; the leaching of cesium was also found to be increased. These results support the concept that the role of boric oxide as a fluxing agent is to facilitate the formation of the network. (AEC Activity 4352)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes into Deep Wells. — The factors controlling the sorption of cesium and strontium by clay minerals are of considerable importance to the deep-well disposal program, for they determine both the distance away from the disposal well to which these ions may move and also the amounts of heat-producing fission products which will be concentrated near the bottom of the disposal well.

Several types of clay and earth material, including illite, bentonite, montmorillonite, kaolinite, halloysite, and Conasauga shale, were tested to determine their sorptive capacity for strontium, in a continuation of previous work with cesium. Slurry tests and hot column tests were used for most of this work.

Strontium removal, unlike cesium removal, was found to depend on pH. The higher the pH of the solution, the greater the amount of strontium removed. At a pH of 7, the capacity of the clays for strontium was about half that for cesium. Illite clay was found to have the highest capacity for strontium, montmorillonite was second, and halloysite had the least. Illite sorbed about 1.25 mg of strontium per 10 g of clay from a 6 M sodium-neutralized Purex solution, at a pH of about 7.

Studies of the sorption of cesium and strontium, by various clay materials, from a sodium-neutralized Purex waste solution that had been complexed with citric acid and diluted (1:9) with 1 M sodium chloride indicated a relatively low capacity for both radioisotopes. The strontium sorption was somewhat reduced by the presence of sodium ions and was reduced still more by the presence of citric acid. The sorption of cesium did not appear to be affected by the presence of citric acid, although it was reduced by the presence of the sodium.

The conclusion appears justified that competing ions like sodium and calcium can be used to control the amount of sorption of cesium and strontium. A number of other factors also must be considered, and the problem of control is complex. (AEC Activity 4352)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes into Salt Formations. — The site for preliminary field experiments with nonradioactive waste has been chosen at the Hutchinson, Kansas, mine of the Carey Salt Company. All operating mines in the northeastern quadrant of the country were inspected, and the Hutchinson mine was chosen on the basis of geological formation, geographic location, facilities available in the mine, estimated costs, and the possible site of the test with radioactive wastes. The use of an operating mine is advantageous because of the immediate availability of a test site, a competent work force with the necessary equipment to mine test cavities, and the necessary auxiliaries — shaft, hoist, power, railroad sidings, and personnel.

Field experiments are necessary because laboratory experiments cannot duplicate the conditions found in geological formations. Samples removed from a formation go through a process of stress relief which alters the physical properties of the salt. Even if the overburden pressure is reapplied, the stresses induced will not necessarily be the same unless the sample is completely elastic, which is not true for geological materials.

The field tests will determine the response of salt *in situ* to the cavities full of waste, the movement of the nuclides from the cavity, the movement of the cavity itself, the thermal distribution both inside and outside the cavity, and the production of gas from the interaction of the waste and salt.

The first series of tests will use synthetic wastes chemically and thermally similar to radioactive wastes, but they will not contain radioactive materials. At present, eight tests are contemplated that require eleven excavations. The cavities will be 7.5 x 7.5 x 10 ft deep. The facilities required for such experiments are: two cavities of approximately 300 gal capacity; graphite heaters; gas-tight covers; thermocouple wells; thermocouples; liquid-sampling devices; liquid-level measuring devices; power panel; temperature, liquid-level, and moisture recorder panels; and gas-control panels. Each test will run for approximately six months; some will run concurrently. A total test period of about two years is estimated. If a series of radioactive tracer tests is conducted at the same location the total test period for both series will be three years. The major variables in the study will be: (1) type of waste—Purex and acid aluminum nitrate (primarily nitric acid and aluminum nitrate), (2) the concentration of acid waste and the pH of the wastes, (3) geometrical configuration of the cavity, (4) volume-area relationships, (5) method of construction (hand excavated, blasted, or solution mined), and (6) rate of heat production.

A special test will be designed to determine the flow of salt under various overburden pressures and temperatures. It is envisaged that a room will be progressively widened to increase the pressure on the pillars, and these changes in pressure and deformation will be measured.

Instrument and engineering design for the experimental facility is about 90% complete. Analysis of the proposed resistance heating system, consisting of graphite electrodes and the internal resistance of the waste,

indicates that though the power distribution in the waste is not uniform, the temperature is relatively uniform through the waste cavity.

Corrosion studies of the material for the equipment necessary for the heating system and the liquid and gas collection and analysis system indicate that Hastelloy C cannot be used in acid Purex waste saturated with sodium chloride; Haynes alloy 25 and titanium are undergoing tests. (AEC Activity 4352)

Waste Disposal Research and Engineering: Process Waste Water Treatment Plant. — The plant was designed to allow the addition of various amounts and combinations of lime, soda ash, clay, returned sludge, and calcium chloride. It was desirable to determine, for each of these materials, the dosage which would remove the greatest amount of radioactive strontium and cesium. Since a trial-and-error attempt to vary five factors on an operating scale is difficult to control, further laboratory studies were necessary. To determine the importance of these five variables, separately and in combination, and the characteristics of their response surfaces, a statistical design was constructed for laboratory experimentation. Preliminary results show that soda ash and lime (less than stoichiometric dosage) affected strontium removals, while clay type, dose, and particle size affected cesium removals. All the effects were significant at the 5% level. As the lime dosage was increased to the stoichiometric requirement, strontium removals were increased. Soda ash dosages greater than stoichiometric requirements increased strontium removals. Grundite was 11% more effective, on the average, than Conasauga shale in the removal of cesium. Also, the removal of cesium increased as dose increased and particle size decreased. (AEC Activity 4352)

Radiation Damage: Efficiency for Cleavage of Polymethyl Methacrylate and Polyisobutylene by Reactor Neutrons. — In a program of comparing gamma-ray and reactor irradiation of polymers, the cleavage reactions of polymethyl methacrylate and polyisobutylene were studied. On comparing the scission rates, as measured by viscosity, for the two reactions in-reactor and in a 1.2×10^6 r/hr gamma source, a correlation with hydrogen content was found. It was assumed that the reactor effect is a superposition of a gamma-ray effect relatively independent of chemical composition and a neutron effect linear with hydrogen content. This leads to a neutron scission rate per unit hydrogen content relative to a 1.2×10^6 r/hr gamma-ray scission rate. The energy absorbed per unit hydrogen content by reactor neutrons has been measured by calorimetry, and this result was also compared with the energy absorbed by a 1.2×10^6 r/hr source. The calorimetry ratio was twice the cleavage ratio, indicating a 50% efficiency of reactor neutrons for the two cleavage reactions. (AEC Activity 4540)

PROGRAM 5000 — PHYSICAL RESEARCH

ISOTOPE PRODUCTION

Radioisotope and Stable Isotope Production. — Operation of the Fission Product Pilot Plant continued, and a total of approximately 12,000 curies of Cs^{137} has been separated.

Calutron separation of the isotopes of lutetium was completed; ion currents equivalent to 34.3 g of lutetium were collected. Separations of the isotopes of gadolinium and magnesium are under way.

The exploratory attempt in the 24-in.-radius tanks to deposit the isotopes of silicon on copper target backing for cyclotron studies was completed. Ion currents equivalent to 5.8 g of total silicon were collected on these targets (special copper pockets), which were turned over to cyclotron personnel.

Charge materials were provided for isotope separation in the following forms: $SiBr_4$ (liquid), Mg metal (lumps), $LuCl_3$ (fused), $GdCl_3$ (fused), and Ca metal (chips). Lutetium and gadolinium charge materials are being recovered and purified for use in future collections.

The stable-isotope chemistry group recovered and refined 13 new lots of isotopes of three elements. Among these were K^{39}, K^{40}, K^{41}, Ca^{46}, Ca^{48}, Yb^{168}. The samples of Ca^{48} and Yb^{168}, along with Hf^{179}, Ag^{107}, Ag^{109}, and Se^{74}, were prepared for inventory following mass analysis.

The material collected in the 167 position during the recent ytterbium isotope collection was further refined; the products are now being analyzed.

A refinement method has been devised which is capable of further purifying lead isotopes from inventory stock so that the impurity level is in the neighborhood of 10^{-2} to 10^{-3}%. The method is primarily based on electrodeposition and crystallization from nitric acid solutions.

Radioisotope Research and Development. — Laboratory-scale experiments continued on re-evaluation of the flowsheet for processing Purex waste in the Fission Product Pilot Plant. It was found that phosphate, which may be present in waste from the Thorex Pilot Plant, will cause cerium to precipitate during the iron precipitation step. Approximately 20% of the cerium is precipitated at a phosphate concentration of 0.005 M, and 73% is precipitated at 0.01 M.

It was found that ammonium persulfate cannot be used in sufficient concentrations to maintain cerium in the Ce^{4+} state under the reducing conditions present in a radiation field because the sulfate ions resulting from the reduction of persulfate cause the formation of double sulfate rare-earth salts. During current processing in the Fission Product Pilot Plant, 0.004 M $KMnO_4$ was used to oxidize cerium. The excess manganese will be separated from the cerium by an oxalate precipitation.

The fission yield of Ce^{144} was determined by dissolving a weighed amount of natural uranium having a known irradiation history. The actual yield determined by this method agreed closely with the calculated yield.

The separation of Y^{91} from the rare-earth fraction in short-cooled waste is necessary prior to the separation of Pm^{147} on ion exchange columns. Four methods of separating Pm^{147} are being investigated:

(1) batch extraction with di-2-ethylhexylphosphoric acid, (2) continuous countercurrent mixer-settler extraction with tributyl phosphate, (3) partial complexing of Y^{91} by ethylenediaminetetraacetate or citrate during an oxalate precipitation, and (4) partial complexing with ethylenediaminetetraacetate during ion exchange adsorption.

The ion exchange column separation equipment in Building 3028 was redesigned for greatly increased production of Pm^{147} of high purity. Two heated tantalum columns, 3 and 2 in. in diameter, and one glass column, 1 in. in diameter, will be used.

The final decontamination procedure for technetium was tested by processing 70 liters of concentrate from the Fission Product Pilot Plant. Approximately 2 g of technetium decontaminated by a factor of 10^6 was recovered. The starting solution contained 3.0 curies/liter of Sr^{90}. (AEC Activity 5100)

Stable Isotope Development. — A voltage regulator circuit designed to replace the conventional Thermohm-type charge-oven heater power control during use of high-temperature M-16 or M-18 ion sources is functioning satisfactorily. All four of the calutrons in Building 9731 and two calutrons in the Special Separations Program which require precise heater power regulation have been equipped with this auxiliary control.

Separated isotopes of silicon were deposited on copper by two methods: (1) Si^+ ions produced in the calutron and having a 90° angle of incidence impinged on a water-cooled copper target; (2) material, consisting of silicon and copper, sputtered from the first target was allowed to deposit on a second target area located parallel to the incoming ion beam. The silicon remaining at the point of ion impact adhered firmly to the copper; the sputtered material was less adherent and tended to flake off the copper surface in small, thin foils. The targets are being investigated to determine whether either of these methods might be applied to the preparation of targets containing certain separated isotopes.

Beam scanning techniques were applied as an in-process control during the separation of gadolinium isotopes. Scans indicated that the purity of Gd^{156} lay within the range 95 to 97%; the actual mass analysis of the Gd^{156} was 97.1%.

An investigation was made of the focusing properties (in the Z direction) of the 190° magnet pole faces shaped to produce a radially inhomogeneous field. An ion source having a series of vertical slits was used as an object. No Z-wise distortion of the image in the 190° position was noted within the investigated 4-in. region centered over the median plane of the system. A source deficiency, namely, excessive angular divergence of the emitted ion beam, was experienced. (AEC Activity 5100)

PHYSICS

High-Voltage Experimental Program: Total Cross Sections in the Kilovolt Region by Time-of-Flight. — During a recent total cross section run,

Sc^{45}, V^{51}, Fe^{57}, Co^{59}, Cu^{63}, Zn^{66}, Zn^{68}, Ge^{70}, Ge^{72}, Ge^{74}, and Ge^{76} were studied in the neutron energy region from 3 to 30 kev. A number of new resonances were observed, isotopic assignments were made, and previously unresolved levels were split. Energies at the peak cross sections of some of the most prominent of these resonances are given in the following table:

Sample	E_o(kev)
Sc^{45}	3.25, 4.26, 6.59, 8.0, 9, 12, 22, 24, 30, 32
V^{51}	6.5
Fe^{57}	3.9, 6.1
Co^{59}	4.9, 8.0, 10, 17, 22, 23, 26, 30
Cu^{63}	5.28, 7.7, 10, 20, 21, 23
Zn^{66}	10, 14, 18
Zn^{68}	18
Ge^{70}	3.3, 5.6, 6.2, 7, 8, 10, 13
Ge^{72}	4.9, 9, 12
Ge^{74}	4.88, 20
Ge^{76}	4.9, 22, 25, 29

Further analysis of the data is in progress. (AEC Activity 5220)

High-Voltage Experimental Program: Activation Cross Sections at 195 kev. — An RdTh-D_2O neutron source has been used to measure activation at 195 kev with about a 50-kev energy spread. Most of the isotopes measured previously at 25 kev (Phys. Rev. 107, 504, 1957) were studied, a 3 x 3 in. NaI (Tl) crystal being used for absolute gamma counting as before. The cross sections are based on an indium activation in spherical shell geometry, the neutron source strength being determined in a large graphite-sphere neutron detector (Nuclear Instruments 1, 335, 1957). Preliminary values are tabulated below:

Target Nuclide	σ_{act} (millibarns)	Target Nuclide	σ_{act} (millibarns)
Na^{23}	0.7	As^{75}	150
V^{51}	7	Zr^{94}	42
Mn^{55}	11	Zr^{96}	7
Cu^{63}	42	Ru^{96}	140
Cu^{65}	14	Ru^{102}	190
Ga^{71}	53	Ru^{104}	28
Ge^{74}	19	Pd^{108}	77

Target Nuclide	σ_{act} (millibarns)	Target Nuclide	σ_{act} (millibarns)
Ag^{107}	290	Pr^{141}	38
In^{115} (54-min)	200	Sm^{152}	150
Sb^{121}	240	W^{186}	100
I^{127}	170	Au^{197}	320
Ba^{138}	3	Hg^{202}	32
Ce^{142}	22	U^{238}	120

(AEC Activity 5220)

Classification of Low-Lying Nuclear Energy Levels. — The gamma-ray spectrum of Rh^{106} has been investigated with a scintillation spectrometer. The Coulomb-excited 2^+ level at 1.13 Mev in Pd^{106} was found to be populated. The intensity of the 1.13-Mev gamma ray from this level was determined as 2.3 when the intensity of the 0.514-Mev gamma ray was normalized to 100. To differentiate the photopeak of the 1.13-Mev gamma ray from the sum peak of the 0.514- and 0.624-Mev gamma rays, the spectrum was measured for two different source-to-detector distances (14 and 40 cm). The present data, combined with data from earlier investigations, gave a value of 2.1 ± 0.6 for the ratio of the cascade gamma rays to the crossover gamma rays from the 1.13-Mev level and a value of 1.2 ± 0.4 for the ratio of the reduced B(E2) values of the 0.617- and 0.514-Mev gamma rays. The present studies also revealed several new gamma rays. Their energies (and intensities) are: 0.71 (0.17), 1.19 (0.2), 1.49 (0.16), 2.83 (0.01), and 2.95 (0.01) Mev. A decay scheme has been proposed which incorporates the data from the total spectrum and also from the spectra made in coincidence with the 0.514- and 0.624-Mev gamma rays. (AEC Activity 5220)

The 86-Inch Cyclotron: Nuclear Physics. — Several minor changes were made in the dE/dx, E particle counting system, and the pulse multiplier was rebuilt. These changes made the operation of the system simpler and more reliable. It is now being used for studies of (p,d) and (p,α) reactions.

Deuteron spectra from (p,d) reactions were measured for 17 targets throughout the periodic table, and a few angular distributions were also measured. The spectra are peaked near maximum energy and fall off rapidly with decreasing deuteron energy, even well above the Coulomb barrier for the deuterons. Within the very gross structure there appear many peaks and valleys broader than the individual level structure. The peaks have similar angular distributions and show similarities among neighboring elements when the data are plotted as counting rate vs the Q of the reaction. Similarities with respect to the excitation of the residual nucleus may also be present. The cross sections decrease with increasing angle, falling typically by a factor of 10 to 20 from 30° to 120°.

The study of (p,α) spectra and excitation functions was extended. The excitation functions for fluorine and aluminum targets fall off much less steeply with decreasing proton energy than do those for other targets.

The study of total elastic and nonelastic cross sections for protons on uranium isotopes was continued with the dE/dx, E counter system. It was found that deuterons contribute a large part of what was thought to be (p,p') scattering in a preliminary measurement. (AEC Activity 5220)

The 86-Inch Cyclotron: Applied Physics. — The isotopes F^{18}, Mn^{52}, Ge^{68}, As^{74}, and Bi^{205} were produced in service irradiations for outside customers. A gallium target was used to produce Ge^{68}; the maximum current was 500 μa. A 0.020-in. depression was milled in a flat-plate copper target and filled with gallium metal; the gallium was liquid during bombardment.

Attempts were made to increase the internal mass-3 beam current and to ascertain its origin. In one such attempt, HD was used as the feed gas to the arc, but the highest current for steady operation (50 μa) is actually obtained with H_2. Both CO_2 and CH_4 were used as the feed gas to determine whether the peak could be ascribed to C^{4+}; however, there was no peak present with CO_2, and the ratio of the mass-3 current to the proton current was the same for CH_4 as for H_2. Work on this problem continues. (AEC Activity 5220)

The 86-Inch Cyclotron Operation. — The target instrumentation was moved to a new location, which is shielded from the external beam system by earth and building footings. It is now possible to use the maximum available deflected beam without incurring a health hazard to personnel. The 20-channel analyzer, amplifiers, and other electronic equipment used with external-beam experiments were moved from their old location in the room above the beam pipe to a shed outside the cyclotron building, which has been remodeled into a counting room. The temperature regulation of the heating system in the new location is better than it was in the old location; the 2° fluctuations now observed seem not to affect the performance of the equipment.

Two runs were made to gain a better understanding of the effects of the parameters that define the effective beam radius. Second-harmonic coils are used to move the effective beam center; the "down coil" controls the effective radius. The system is now calibrated for actual radius vs energy, for down-coil current vs energy, and for the effects of these parameters on beam current. For isotope production purposes the position of maximum energy for a current of 1000 μa was determined. (AEC Activity 5220)

Heavy-Particle Physics. — The elastic scattering of nitrogen from thin carbon foils (200 $\mu g/cm^2$) is being investigated. Two CsI scintillation counters in coincidence are used to detect the scattered nitrogen and recoil carbon atoms, respectively. It is possible to discriminate against scattering from impurities, as well as against inelastic scattering and

transfer reactions. The angular region investigated is from 36 to 138° in the barycentric system. The differential cross section falls rapidly from 36 to 60°, levels off slightly around 60°, and has two relatively sharp minima at 92 and 118° and two maxima at 104 and 132°. The cross section at both maxima is about seven times as large as the cross section at the minimum between them. (AEC Activity 5220)

Electronuclear Machines. — Cyclotron Analogue II is a fixed-frequency electron cyclotron to be operated as a model of the proposed 850-Mev proton cyclotron. It will have an eight-sector spiral magnetic field and is being designed to accelerate electrons to an energy of 450 kev.

Digital computer calculations for the design of the sector coils are nearing completion. This work involves locating the correct conductors appropriately to produce a magnetic field with the proper spiral and increase of flutter (azimuthal variation of the magnetic field) with radius to maintain the axial focusing frequency of the accelerating electrons within narrow limits. The coils are first located by computing the magnetic field of a multiplicity of straight conductor segments. The derived shape is then converted to a series of tangent circular arcs. Another computer program calculates the magnetic field of finite-cross-section circular-arc conductors to within three parts in 10^5. Approximate formulas are used to fit the required flutter to within a few per cent; detailed adjustment of the coil shape to give constant axial focusing frequency is based on the results of a point-by-point orbit integration in which exact equations of motion are used.

The voltage-sensing circuitry required for providing accurate regulation of the r-f power supply is being built. Also, work on the high-current power supply for the sector coils has begun. This unit, which must provide 550 amp at about 40 v, is a conventional rectifier unit with a series current regulator consisting of a large number of paralleled power transistors with a common control circuit.

The sector coils consist of four turns of heavy conductor carrying approximately 550 amp. The accuracy requirements for the magnetic field dictate that the conductors must be accurately located within ± 0.001 in. Serious consideration is being given to the use of a tape-programed milling machine to produce both the conductor and the mounting base. Drawings of a full-scale test sector coil (duplicating the complexity of the final assembly) are being prepared for submission to an interested vendor. (AEC Activity 5240)

The 48-Inch Cyclotron. — This project was officially terminated; the scope of the original program was considerably enlarged upon the approval of the proposed Relativistic Isochronous Cyclotron. (AEC Activity 5240)

Relativistic Isochronous Cyclotron. — The proposed Oak Ridge Relativistic Isochronous Cyclotron (ORIC) was approved, and funds for construction have been made available. This fixed-frequency cyclotron is designed to accelerate protons to 75 Mev and nitrogen ions to 100 Mev. The threefold azimuthally varying field and a variable r-f system will provide great

flexibility in operation. Catalytic Construction Company was selected for the architect-engineering phase of the work. Completion of the $3,000,000 project is scheduled for late in 1960.

Special Separations. — The second charge used in the calutron separation of U^{233} was exhausted in three runs which resulted in an estimated collection of 6.9 g. The total collection thus far is 14.35 g.

Three additional runs concluded the collection of U^{238} in which UCl_4 was used as the charge material and the M-16 as the source; the total estimated collection is 79.0 g, at a process efficiency of approximately 5.0%. Sixteen additional runs with the double-arc source, M-12, have resulted in collection of 1000.96 g. The process efficiency on normal runs was doubled (8.0% to 16.0%) by an increase of the 90° baffle opening from +8°, -4° to +12°, -12° and an increase of the receiver slot from 0.093 to 0.145 in.

A total of 12 kg of UCl_4 was prepared for use as charge material in the separation of U^{238}. Collector recovery has not been completed. Approximately 94% of the U^{235} initially on hand has been recovered and removed from the special separations operation. The carbons from the source and collector have been leached, and the resulting solution has been evaporated and sampled. A final balance will follow the cleaning of the calutron tank.

A total of 3.4 g of U^{233} has been processed as product, and a sample has been sent to KAPL for assay. A second collector is being processed. Five hundred seventy grams of UCl_3 remain on hand for the continued separation of U^{233}.

Three standard samples were prepared as a blend of U^{234}, U^{235}, U^{236}, and U^{238}. There were approximately 25 shipments from the research pool.

Twelve kilograms of depleted uranium metal for the Civilian Application Program is being processed. The delivery of foil and rods is expected in January. (AEC Activity 5250)

CHEMISTRY

Druhm Process. — Two experimental reduction runs were made with gas-cooled copper UF_6 nozzles and graphite liners. There was no corrosion of the nozzle during either run, but uranium metal recovery was poor, due to cracks in the graphite liners. (AEC Activity 5310)

Raw Materials Research and Development. — Dinonylnaphthalenesulfonic acid (0.1 M) and diamyl amylphosphonate (0.1 M), when used in combination, showed coefficients of >100 for extraction of uranyl and thorium ions from 0.5 M sulfate solutions at pH 1 and 1.5, whereas coefficients for these reagents when used separately were <0.5. A somewhat weaker synergistic effect was obtained in extractions of vanadium(IV) and iron(III).

Results of batch studies of the kinetics of uranium extraction from sulfate solutions show that the rate of extraction with amines is about one order of magnitude faster than with di(2-ethylhexyl)phosphoric acid. When the aqueous phase was perchlorate instead of sulfate, the rate with di(2-ethylhexyl) increased and was nearly the same as with amines. Tests over a wide range of turbulence and interfacial area show that the rate of extraction is directly dependent on area and virtually independent of turbulence, which is not usually the case for diffusion-controlled extractions but is in line with an ion exchange mechanism.

Countercurrent tests in a modified Purex first-cycle flowsheet with dibutyl phenylphosphonate (DBPP), which has been reported to be significantly more stable to irradiation than tributyl phosphate (TBP), indicated potential use of lowered aqueous acidity and salting strength in a solvent extraction process for irradiated uranium. Separation factors from fission product rare earths and ruthenium were higher than with TBP. The higher uranium extraction power of DBPP permitted lowering of nitric acid concentrations to 0.5 M in feed and 0.01 M in scrub for extraction with 1 M DBPP in xylene—Amsco 125-82 diluent, resulting in a decrease to 0.25 M HNO_3 in the aqueous waste. A primary disadvantage of the higher extraction power is the larger volume of 0.01 M HNO_3 strip solution required (O/A = 1/2 for 1 M DBPP).

Extraction coefficients for neptunium(IV) with 0.01 M tri-n-octylphosphine oxide (TOPO) in Amsco 125-82 from nitric acid solution reached maxima of 40 and 25 at 1 and 8 M HNO_3, with an intermediate minimum of 15 near 4 M. The coefficients dropped sharply below 1 M. The coefficients increased with increased sodium nitrate concentration at constant total acidity and with decreasing acidity at constant total nitrate concentration, to 250 at 1 M HNO_3 and 400 at 0.5 M HNO_3, both in 6 M total nitrate. A tenfold higher concentration (0.1 M) of the highly branched tris(2-ethylhexyl)phosphine oxide was required to give extraction coefficients from nitric acid as high as those with 0.01 M TOPO.

Different uranyl sulfate extraction rates obtained with amine sulfates in violent shaking and in slow stirring (ORNL-2595) were measured through a nearly quiescent interface of approximately constant area. Acid extractions by 0.1 M amines in benzene, and uranyl sulfate extractions by the corresponding amine sulfates obeyed first-order rate laws, $kt = \log X_e/(X_e-X)$, where X_e is the amount transferred at equilibrium, and X is the amount transferred at time t. The rate constant k was directly proportional to stirrer speed in extraction of sulfuric acid with tri-n-octylamine from 0.05 M solution (0.00268 to 0.0260 min^{-1} at 10 to 100 rpm) and nearly so in extraction of uranyl sulfate from 0.001 M UO_2SO_4 solution. Diffusion through the interfacial layer complex apparently determines the rate and (in conformity to Fick's diffusion law) is proportional to the concentration gradient. Extraction of sulfuric acid was faster than of hydrochloric acid, while uranyl sulfate extraction was slower than either. With other amines, acid extraction rates were approximately proportional to the base strengths of the amines in benzene, primary $>$ secondary $>$ tertiary. (AEC Activity 5310)

Chemical Engineering Research. — In the search for a method of quantitatively analyzing the protection against corrosion afforded a porous metal surface by a transpirational inert fluid, preliminary experiments using nickel specimens mounted on an impeller in a ferric chloride solution showed (for reaction rate in terms of concentration at the interface)

$$R = 850C^{0.6},$$

where

R = reaction rate when the reciprocal of the impeller speed is extrapolated to zero, in./year,
C = bulk ferric chloride concentration, moles/liter.

(AEC Activity 5310)

Equipment Decontamination. — Corrosion scale was removed from short lengths of HRT stainless steel pipe by several different solutions, namely, chromous sulfate, alkaline periodate, alkaline hypophosphite, and Turco 4501 followed by Turco 4502. Only the chromous sulfate solution completely dissolved the scale. The other treatments removed the scale as small solid particles. Treatment of short lengths of pipe from the HRT with 0.5 M $CrSO_4$–0.5 M H_2SO_4 for 2 hr at 85°C dissolved the scale, thus giving gamma decontamination factors of 40 to 60. The remaining gamma activities, primarily Ru and Nb, were removed by acid or basic hydrogen peroxide, alkaline periodate, or Turco solutions for an over-all decontamination factor of approximately 10^3. (AEC Activity 5310)

Extraction Reagent Performance. — Three additional amines, triisooctylamine, Primene JM-T, and Alamine, have been irradiated to 400 whr/liter with 1-Mev electrons in new solvent scouting tests. Respective yields, G (moles per 100 ev of energy absorbed), for gas formation were 2.9, 2.9, and 3.1.

Dibutylphosphoric acid (DBPA) and di(2-ethylhexyl)phosphoric acid (D2EHPA) have been irradiated at 300 whr/liter to determine the reproducibility of anomalous results previously observed at irradiations of 1200 to 2000 whr/liter in which the expected yields of dibasic and tribasic acids were inverted. At the lower doses, the yields, G, from DBPA and D2EHPA were, respectively, dibasic acid, 1.58 and 0.70; tribasic acid (H_3PO_4), 0.52 and 0.52; total target decomposition, 2.32 and 1.54; target converted to polymer, 0.22 and 0.32. These data show that extensive decomposition of primary radiolysis products occurred at the very high dose levels, where as much as 80% of the target was destroyed, and that the anomalous results reflected the presence of secondary products.

PMH (2,2,4,6,6-pentamethylheptane), a possible major component of Amsco 125-82, has been irradiated to the 89-whr/liter level after equilibration with 4 M HNO_3. The presence of HNO_3 reduces the yield, G, of double-bond formation to ~2 to 3, as compared with the corresponding yield of 7.5 from the dry and 5.5 from the water-saturated PMH. Yields of polymer, hydrogen, and methane are not greatly affected by the presence of HNO_3. (AEC Activity 5310)

Ion Exchange Technology. — A fixed-bed ion exchange run using 20- to 30-mesh Duolite S-30 to remove Cs^{137}-Ba^{137} (at a concentration of 0.5 curie/liter) from a synthetic ORNL waste (0.3 M $NaNO_3$—0.1 M NaOH) showed that the over-all height of a transfer unit, based on the solution phase, was 0.3 in. at a superficial flow rate of 7.4 $ml \cdot min^{-1} \cdot cm^{-2}$. (AEC Activity 5310)

Organic Chemistry. — In the evaluation of organic compounds as reactor fluids, perfluoroheptane was irradiated in the LITR, and the irradiated material was analyzed by vapor chromatography. The gas samples contained air (which may have been introduced during sampling), much CF_4, and some C_2F_6. The liquid sample contained at least five fragments; perfluoropropylene has been definitely identified.

The rearrangements of threo- and erythro-1,2-diphenyl-1-m-tolylethylene glycol (I) and 1,1-diphenyl-2-m-tolylethylene glycol (II) in concentrated sulfuric acid at 0°C have been examined. The yields of the two ketones produced were determined by the isotope dilution method, and the fates of various C^{14} labels of I and II were established. From the foregoing data, the contributions of several paths to the rearrangements of I and II were calculated, and it was shown from these that the m-tolyl/phenyl migration ratio in the rearrangement of the conjugate acid of diphenyl-m-tolylacetaldehyde is not less than 2. The present results support in every detail the mechanism previously proposed for the rearrangements of triaryl glycols and triarylacetaldehydes.

The photochemical reaction

$$Br_2 + HCOOH \xrightarrow{h\nu} 2HBr + CO_2$$

was investigated by the methods reported previously (ORNL-2636). The deuterium rate isotope effect with formic-d acid, DCOOH, at 45°C was found to have a value of k_H/k_D = ca. 3.9. The corresponding C^{13} isotope effect with $HC^{13}OOH$ was shown to have a value of k_{12}/k_{13} = 1.023 (J. Am. Chem. Soc. 79, 4944, 1957). (AEC Activity 5330)

Analytical Chemistry Research. — In conjunction with a study of the effect of temperature on the efficiency with which metals are extracted by tri-n-octylphosphine oxide (TOPO) in cyclohexane, measurements were made of the solubility of TOPO in cyclohexane as a function of temperature. At 6.5 ± 0.1°C, the freezing point of cyclohexane, the solubility of TOPO is 8.142 g per 100 ml; at 25.00 ± 0.05°C, the solubility is over four times as large, 35.61 g per 100 ml. As the temperature is increased further to 35°C, the solubility increases only slightly to 36.08 g per 100 ml, apparently approaching a limiting value. The plot of the logarithm of the mole fraction of TOPO vs the reciprocal of the temperature was found to be linear only over the rather narrow temperature range from 11 to 25°C. An approximate value of 12.1 kcal/mole was calculated for the heat of fusion of TOPO.

METALLURGY

Fundamental Physicometallurgical Research. — The lattice parameter of thorium was determined in the temperature range 4.2 to 150°K. Both as-worked and annealed thorium filings were used with an internal standard of aluminum powder. The a_o vs temperature plot for each specimen showed no deviation from a smooth curve. Under the conditions of this experiment, a low-temperature transformation, which might be expected from Bridgman's high-pressure experiments, was not observed.

Investigations of zirconium-gallium alloys indicate that the beta phase decomposes by the eutectoid reaction

$$\beta(\sim 1.3 \text{ at. \% Ga}) \rightleftharpoons \alpha(\sim 0.4 \text{ at. \% Ga}) + Zr_3Ga$$

at temperatures near 850°C. The depression of the allotropic transition temperature of zirconium by gallium additions is believed to indicate a large size-factor effect in this system, since, otherwise, elevation of the temperature would be expected with a trivalent solute. Further indications of the size-factor effect are the reduced beta solutility, a large heat and excess entropy from the temperature dependence of the beta solubility, and an unusually large hardening effect of gallium in these alloys.

The heat capacity of a zirconium-10 at. % indium alloy has been measured over the temperature range 1.2 to 4.5°K. Since the maximum solubility of indium in α-zirconium is less than 10%, the indium was put into solution in β-zirconium and the alloy quenched to room temperature, yielding a metastable α' structure. The density of states and the Debye temperature are in qualitative agreement with those extrapolated from alpha-annealed indium alloys, indicating the feasibility of preparing alloys with solutes of limited solubility in this manner for examination of the effects of alloying on the density of states and the Debye temperature. (AEC Activity 5420)

Microstresses in Metal Crystals. — In order to improve the quality of diffuse scattering measurements, it has been decided that they should be made at liquid-nitrogen temperature to reduce temperature diffuse scattering, and that they should be made in vacuum to eliminate air scattering. The design of a special specimen holder to accomplish this has been completed, and work on its construction and testing is in progress.

Future measurements of the Bragg maxima of thin surface oxide films grown on metals will be made with a new monochromator and optimum diffractometer geometry. The monochromator has been designed, and its construction is nearly completed. (AEC Activity 5420)

Ceramics Research. — Spheres of thoria from 0.125 to 0.006 in. (+100 mesh) in diameter were prepared by a recently developed method. They were fired at 1700 to 2000°C and tested for abrasion resistance by an empirical

method. This test consists in suspending 1/2-g samples of spheres screened, usually to -35 +50 mesh, in a vertical stream of air. The air is contained in a 3/4-in. glass tube and moves ~9 fps. At 1/2-hr intervals the weight of -35 +50 mesh spheres remaining is determined.

Under these conditions spheres of straight ThO_2 fired at 1750°C lost ~12% in weight per hour. Firing at 2000°C did not increase the abrasion resistance notably but caused the spheres to sinter together. (AEC Activity 5420)

Fundamental Investigations of Radiation Damage in Solids: Thermal-Neutron Irradiation of Germanium. — The energy position and electrical role of fast-neutron- or gamma-ray-introduced energy levels in germanium are of considerable interest. The effect of thermal-neutron irradiation on n-type germanium is to introduce an excess of p-type impurities (Ga) over n-type impurities (As). A type of Fermi level spectrometry has been suggested, in that conversion to p-type material by gallium introduction following thermal-neutron irradiation would carry the Fermi level across any fast-neutron- or gamma-ray-introduced energy levels. The gallium introduction occurs with a half life of 11.4 days, and the Hall coefficient and conductivity of the sample are monitored as the carrier concentration changes.

Preliminary experiments performed in the biological thermal-neutron treatment chamber of the thermal column of the Graphite Reactor have indicated a thermal-neutron flux of 1.4×10^9 neutrons·cm^{-2}·sec^{-1} and a resonance or fast (>0.1 Mev) neutron flux in excess of 1.2×10^7 neutrons·cm^{-2}·sec^{-1}. The latter figure is considerably in excess of that predicted for this particular experimental facility from the published In-Cd ratio of 10^5. (AEC Activity 5420)

Fundamental Investigations of Radiation Damage in Solids: Irradiation Effects and Short-Range Order in Silica and Quartz. — The crystalline order in the region of one of the defects (J. Appl. Phys. 27, 1376, 1956) of the quartz structure produced by irradiation of silica and quartz crystals has been examined by electron spin resonance. In a single crystal its g tensor has eigenvalues $g_1 = 2.0003$, $g_2 = 2.0006$, $g_3 = 2.0018$. On the assumptions that the defect has the electronic spin S = 1/2 and that the nuclear spin I = 0 (Si^{29}, for which I = 1/2, is 4.7% abundant), the shape of the envelope of the single-crystal lines was calculated for random orientation. Good agreement of the calculated line shape with that in gamma-irradiated Corning silica was found. The line shape in reactor-irradiated Corning silica and in a heavily irradiated single crystal was not in agreement. The line shape in a "waterless" silica (Corning 7943) was found to be similar to the shape in heavily irradiated silica and quartz crystal. Other indications of a structural difference are: (1) the optical absorption edge in the "waterless" is ~1.0 ev below the edge in the "normal" silica and (2) the density of the "waterless" silica is 0.5% higher. The agreement of calculated and observed line shape in the gamma-irradiated "normal" silica indicates that the crystalline order around the defect in the silica is similar to that in the single crystal. It is suggested that around each defect in the "normal" silica there is an ordered

region which has a diameter of ~30 Å. The similarity of the line shape in the heavily irradiated silica and single crystals to the line shape in the "waterless" silica suggests that the short-range order is the same. In this case, axial symmetry of the defect could account for the observed line shape. (AEC Activity 5420)

PROGRAM 6000 — BIOLOGY AND MEDICINE

BIOPHYSICS

Waste Disposal Research: Soil Disposal of Intermediate-Level Radioactive Wastes. — Knowledge of the mechanism of fixation of fission products to soils is important in understanding the operation of present-day soil disposal systems and in developing improved methods of soil disposal. Cesium was selected for the initial study due to the hazards associated with certain of its radioisotopes and the large quantities of these radioisotopes produced in the fission process.

Various clay minerals exhibit different types of cesium sorption curves, depending on the nature of their exchange sites. Clays having exchange sites with no specificity, such as the bentonites and kaolinite, exhibit cesium sorption curves which can be described by the law of mass action with a single equilibrium constant. Illite has a portion of its exchange sites highly specific for cesium, and the sorbed cesium is held more tightly the less the amount present. In the case of vermiculite, an increase in the cesium concentration results in a decreased percentage of the cesium sorbed by the clay until a critical concentration is reached. At approximately 10^{-2} meq Cs per meq cation exchange capacity (CEC) in 0.1 N NaCl there is a sharp increase in the effectiveness of the vermiculite to remove cesium from solution. A maximum removal is obtained at 0.5 meq Cs per meq CEC and the percentage of cesium sorbed again decreases with increasing cesium concentration. If the vermiculite is previously treated with potassium, the exchange capacity is reduced from ~50 to 10 meq per 100 g, and the sorption curve obtained is similar to that of illite. X-ray diffraction data by Tamura show that upon potassium treatment the spacing decreases from 14 Å to 10 Å. This indicates a structural change from vermiculite to illite. It would be expected that cesium would cause a similar structural collapse. (AEC Activity 6440)

PROGRAM 8000 — WORK FOR OTHERS

Professional Health Physics Training Program. — Two members of the Health Physics Division conducted the Third International Course in Health Physics jointly sponsored by the World Health Organization, the Indian Government, and the USAEC. The course extended over a five-week period and was held at the Department of Atomic Energy in Bombay, India. The 25 participants represented the following countries: India, Thailand, Australia, China, Japan, the Philippines, and Indonesia. (AEC Activity 8780)

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

December 1958

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