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## STATUS AND PROGRESS REPORT

## DECEMBER 1957

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# OAK RIDGE NATIONAL LABORATORY

# STATUS AND PROGRESS REPORT

December 1957

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December 1957

This status and progress report summarizes somewhat less than one-half the activities of the Laboratory. A few of the activities are reported every month, but most of them are reported on a bimonthly schedule. Program 4700 is reported separately.

## PROGRAM 2000 — SPECIAL NUCLEAR MATERIALS

In-Line Instrumentation — A digital interface-level controller was designed and fabricated. The instrument was operated by means of the difference in the electrical conductivities of the two phases.

A prototype gamma-activity monitor was designed and constructed. It operates satisfactorily in the Thorex Pilot Plant extraction-column product stream when the gamma activity is $\sim 10^5$ counts/min/ml. Background effects are minimized in this instrument because the sample is a continuous stream that falls through the gamma spectrometer cell without touching the cell surfaces.

A turbine flow-rate meter, installed in the Metal Recovery Plant acid addition line, operated satisfactorily during the first week of use. (AEC Activity 2724)

## PROGRAM 3000 — WEAPONS

Special Separations — One of the tanks in Building 9204-3 has been used in the high-resolution-calutron experimental investigations. A number of runs in which the charge was $UCl_3$ were made in order to determine performance characteristics of the equipment.

The high-resolution equipment was used to separate about 120 mg of $U^{236}$ from 18.25% feed material. Approximately 70 mg of the product contained 97.8% $U^{236}$, and the other 50 mg contained about 96% $U^{236}$.

A separation of $U^{234}$ from 13.5% feed material is in progress. Both this and the $U^{236}$ collections were delayed by water leaks which developed in the cooling lines within the diffusion pumps. In each instance, dismantling of the pumps was required prior to resumption of separation activities. (AEC Activity 3610)



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## HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test — Leaching tests on the fuel recombiner—iodine absorber were successfully completed, with no leaching indicated, during the first week in December, and the unit was installed in the reactor. The reactor piping was flushed with a small amount of heavy water to remove any light water still present, and the reactor was then charged with heavy water.

With the exception of the roof pan seals, leak testing and repairs necessary to establish the gas-tightness of the shield are nearly complete. The north wall was filled with water and inspected, and the cooling water, refrigeration, instrument air, and auxiliary steam systems were successfully sealed and placed in operation.

On December 14 the reactor shield was evacuated to 1/2 atm absolute pressure, and the reactor was brought up to 1700 psi and 280°C for a final operational check. After an interruption on the following day for removal of an obstruction from the inlet line of the fuel-circulating-pump purge pump, additional heavy water was added and operation was continued. The hydraulic behavior of the low-pressure system was studied with the new fuel recombiner—iodine extractor installed and control circuit and instrument adjustments were made. Reactor behavior was judged satisfactory on December 20, and the system was cooled to 120°C without draining. By pumping water through the reactor steam piping, the heavy water was subsequently cooled to 25°C to simulate the conditions necessary for low-temperature criticality experiments.

Enriched uranium was added to the HRT beginning on December 24; the initial critical experiment was completed on December 27. The total fuel loading was 2058 g of $U^{235}$, and the reactor was critical at 29°C with a fuel concentration of 3.13 g of $U^{235}$ per kilogram of $D_2O$. Performance of the mechanical equipment was completely satisfactory, and the experiment was carried out without incident. (AEC Activity 4103.3)

HRT Chemical Pilot Plant — Preparations for radioactive operation were continued. Maintenance in the cell was completed except for repair of leaks in electrical conduits. Leaks in the process piping and equipment were found and repaired until the leak rate, measured with a $Kr^{85}$ tracer technique, was reduced to less than 2 cc of gas per day at a 15-psi differential. (AEC Activity 4103.1)

Uranyl Sulfate Blanket Processing — Two runs were made in P1 loop with plutonium sulfate in uranyl sulfate solution at 250°C. In the first run 1.2 to 1.7 mg of $Pu^{4+}$ per liter was added to 1.2 m $UO_2SO_4$; in the second run 67 mg of $Pu^{6+}$ per liter was added to 1.4 m $UO_2SO_4$. In both runs the concentration of dissolved oxygen in the solution was sufficient to oxidize plutonium to the hexavalent state. Essentially all the plutonium remained in solution; a small amount was adsorbed on the walls. Essentially none of the plutonium or the loop corrosion products were removed in the hydroclone underflow pot. (AEC Activity 4103.1)

Thorium Oxide Slurry Development — Oxide satisfactory for slurry engineering studies is being prepared at a rate of 10 kg/day; thorium oxalate batch-precipitated at 10°C is fired at 1600°C, and the oxide product is classified by sedimentation to a maximum particle size of 5 μ. Improvements in the precipitation step (i.e., better agitation and longer digestion times) produced a more uniform product, reduced clinker formation, and increased the yield of usable oxide products.

In studies to develop alternate methods of slurry oxide preparation, thorium oxalate produced by continuous precipitation in a vortex cavity mixer has yielded an oxide product with a size distribution more favorable than that from other methods of continuous precipitation. Slurries of thorium oxide dispersed in $C_6H_6$ and $CH_3OH$ were burned in an oxyacetylene torch to give fine-particle (~1 μ) products which were not trapped in the spray column—vacuum filter collection system.

A slurry of 1600°C-fired pumped oxide (dried and reslurried) in $D_2O$ (700 g of thorium per kilogram of $D_2O$) was irradiated in the LITR for 212 hr at 300°C without apparent damage. In a slurry of thorium oxide (Drifilm-coated), containing 500 g of thorium per liter, in diphenyl, stirrer operation stopped after six days of irradiation in the LITR at 300°C. An out-of-pile control experiment with similar slurry was operated for 425 hr without difficulty.

Gas production experiments in the ORNL Graphite Reactor with a thorium-uranium oxide ($U^{235}/Th = 0.028$) fired at both 650 and 900°C showed that the gas production and recombination rates were linear functions of slurry concentration and independent of the calcination temperature (G value = 0.6).

In out-of-pile gas recombination studies a slurry of thorium-uranium oxide prepared from the coprecipitated oxalates calcined at 1600°C had a negligible activity at 250°C in the absence of catalyst. The addition of $MoO_3$, in increments of 0.012 m, and activation with hydrogen gave $H_2$-$O_2$ reaction rates at 250 to 300°C which were far greater than those required for reactor use ($\gg$10 moles of $H_2$ per hour per liter at $P_{H_2}$ = 500 psi) and which increased with $MoO_3$ concentration to 0.048 m. The rate in the slurry containing the 0.048 m $MoO_3$ was 86 moles of $H_2$ per hour per liter of slurry at 250°C. (AEC Activity 4103.1)

HRP Chemistry: Fuel Studies — Laboratory measurements were made of the rates of reaction of deuterium with oxygen in the presence of simulated HRT fuel solutions in heavy water. The test solutions were, at room temperature, 0.046 M in hexavalent uranium, 0.062 M in sulfate, and either 0.00144, 0.000653, or 0.000299 M in cupric ion. Tests were performed at 250, 275, and 300°C in order to permit the results to be extrapolated and interpolated to provide estimates of the required copper concentrations for various conditions of HRT operation. The recombination rates in the deuterium system were found to be 40 to 70% of the previously measured rates at the same temperatures and for the same hydrogen pressures in the light-hydrogen system. These results generally confirm those of previous studies of the deuterium-oxygen reaction in light-water systems at 250°C and in both light- and heavy-water systems at lower temperatures. (AEC Activity 4103.1)

HRP Analytical Chemistry — A direct, flame photometric procedure was developed, for the determination of aluminum in concentrations as low as 10 ppm in slurries of thorium oxide and in other materials, with a coefficient of variation of about 10%. The tolerance limits for the presence of a number of interfering elements were found to be as follows:

| Element | Tolerance (mg/ml) |
|---|---|
| U(VI), Cr(VI), Th, K, Ca, Mg, Ba, Fe(III) | 1.0 |
| Na, Li, Ti | 0.04 |
| Mo, W, Cu, Ni | 0.02 |

A method was developed for the determination of titanium which is being utilized in measuring the rate of corrosion and erosion of the titanium components of test loops in which large quantities of thorium oxide are being circulated. A modification of the colorimetric method, based on the use of Tiron (disodium-1,2-dihydroxybenzene-3,5-disulfonate), was found to be suitable for this purpose.

In order to control the composition of the HRT fuel, the excess amount of sulfuric acid which is present in the uranyl sulfate solution must be known. It has been found that this free-acid content can be determined in the presence of hydrolyzable substances by an enthalpimetric method of titration. (AEC Activity 4103.1)

Slurry Testing and Evaluation — Run 200-A-14 has continued for over 3500 hr with no apparent change in the slurry physical properties or appearance in the particles other than a slight rounding off of the corners. Samples of this 1600°C-fired oxide withdrawn after 3000 hr showed mean particle diameters of about 1.7 μ, compared with an initial size of about 2.5 μ, thus indicating a very slow degradation. However, the attack rate on the stainless steel loop has not changed since the first 24 hr of operation, remaining at 1.2 mils/year based on the iron pickup of the slurry. (AEC Activity 4103.1)

Slurry Blanket System Development — Run SM-4 has operated for over 2400 hr with a sulfated slurry made from 800°C-fired oxide, the last 1200 hr having been at a charged concentration of 600 to 700 g of thorium per kilogram of $H_2O$ (1 metric ton of thorium in the loop). Repeated measurements of the gamma-ray transmission through the slurry indicate that a region of higher-than-average thorium concentration exists in the upper hemisphere at all flows and concentrations tried. The high concentration indicated is believed to be due largely to a pileup on the top of the core vessel. The distribution pattern is not affected by the flow in the range of 200 to 350 gpm at temperatures as low as 150°C nor by the flow in the range of 300 to 350 gpm at temperatures of 190 to 200°C. Flows as low as 200 gpm at 190°C bring about a noticeable increase in the thorium concentration in the upper hemisphere.

Microscopic examination of the slurry has shown no further growth of particles, and the material appears to circulate well.

Some difficulty has been encountered in maintaining pressure control at the higher concentration because of dense slurry accumulations in the pressurizer interfering with the heat transfer from the pressurizer wall to the liquid. (AEC Activity 4103.1)

Blanket Engineering Research — Laminar heat-transfer measurements with aqueous thorium oxide slurries indicate that the corresponding heat transfer coefficients are less than the conventional Newtonian-fluid coefficients by a factor of

$$\frac{1}{\left[1 + (g_c \tau_y D/24 \eta V)\right]^{1/3}}.$$

Turbulent heat-transfer coefficients are the same as those given by the Sieder-Tate relation provided that the coefficient of rigidity is substituted for the viscosity. The onset of turbulence is given by the Hedstrom criterion, with the transition region existing for Reynolds numbers up to 4 times the critical (similar to the behavior of Newtonian fluids).

The room-temperature yield stresses of slurries containing 1000 g of thorium per kilogram of $H_2O$ and prepared from a variety of oxides calcined at 1600°C were in the range 0.04 to 0.1 lb/ft$^2$. (AEC Activity 4103.1)

Core and Blanket Vessel Development — Design of a 30-in.-dia low-pressure blanket vessel model is about 90% complete. It has been decided to develop a jet mixer for the blanket vessel, which will increase the apparent circulation rate at some cost in pressure drop. A jet is being purchased for preliminary experimentation. Equipment to test this jet has been constructed, as well as equipment to determine the angle of repose of settled thoria in the blanket vessel. (AEC Activity 4103.1)

Centrifugal Circulating Equipment — The test of the 400A-1 pump in fuel solution saturated with gas was terminated after 1585 hr and the pump was removed from the test loop for installation in the HRT mockup. The pump and motor did not gas bind, but there was localized corrosion and/or erosion attack on the lower wear ring and diffuser which might have resulted from the presence of gas bubbles. This run completed 13,185 hr of maintenance-free operation of the pump. Inspection disclosed that the thermal barrier seal weld had cracked and that the Stellite-12 overlay on the thrust bearing shoes had deteriorated badly.

A 1033-hr run was completed on the 400A-2 pump with the internal cooling system arranged so that a stagnant region existed between the thermal barrier and the lower radial bearing. The thrust bearing and lower radial bearing were badly worn indicating that this is not a satisfactory coolant circuit.

The 300A-1 pump has been removed from the HRT mockup after 4400 hr of maintenance-free operation with uranyl sulfate solutions. This pump will be installed in a $ThO_2$ slurry loop which is under construction.

Preparations are being made for testing the effects of radiation on the insulation of stators for canned motor pumps. One stator was installed in the HRT cell near the fuel circulating pump. Two others will be irradiated in a cobalt source. (AEC Activity 4103.1)

Miscellaneous Equipment — Construction of a facility for thermal-cycle testing of heat exchangers and other equipment was completed. The facility was operated successfully through several test cycles. A few minor adjustments in instrumentation and steam separators are required for reliable automatic operation.

Fabrication of an all-titanium letdown heat exchanger for the HRT mockup was completed. The Crane Company completed fabrication of the 3-1/2-in. and 20-in. titanium-lined pipe. A leak developed in the 20-in. pipe during a hydrostatic test and is being repaired.

An electrically heated Dowtherm boiler and a length of 3-1/2-in. titanium 110AT pipe were received for construction of a boiling-solution pressurizer. The pipe had large surface defects; a second length is being supplied. (AEC Activity 4103.1)

Heat Removal Equipment — Fabrication of the type 347 stainless steel heat exchanger for the HRT mockup was completed, and the unit passed hydrostatic and helium leak tests.

No failures have occurred in five small tube bundles after being thermal-cycled for 2000 hr in boiler water containing chloride and oxygen. (AEC Activity 4103.1)

Gas Handling Equipment — The high-pressure recombiner loop has been rebuilt, with Inconel being used at points previously subject to stress-corrosion cracking. A run to demonstrate the long-term reliability of the device has been in progress for 200 hr. Titanium specimens installed in locations where grain growth and recrystallization had occurred in previous tests were observed to remain at loop temperature. Grain growth and recrystallization normally occur at temperatures considerably higher than the temperatures in the loop.

A small titanium system in which uranyl sulfate solution is boiled at 1500 psi has been operated for 450 hr. (AEC Activity 4103.1)

Fuel Systems Development — The core solution system (HRT mockup) completed a 2245-hr run with no major mechanical troubles. With a solution composition of 0.06 M $UO_2SO_4$, 0.02 M $H_2SO_4$, and 0.005 M $CuSO_4$ plus minor impurities, precipitation of uranium occurred between the pressurizer and the main loop with a purge of 14 gph of condensate into the pressurizer. Under gas pressurization (no purge) and under steam pressurization with a purge rate of 2 to 3 gph the precipitate redissolved.

The iodine removal tests have been completed. A 6-in.-dia by 6-in.-deep bed of silver-plated wire mesh removed the iodine with an efficiency of 99.6% at 120°C. An 18-in.-deep bed loaded with 20 g of iodine, tested in the loop for 650 hr, showed negligible loss of iodine and silver.

On December 8, 1957, the loop was shut down for installation of a titanium letdown heat exchanger, replacement of the 300A Westinghouse pump with a 400-A model, and reinstallation of the 80-kw loop heat exchanger. (AEC Activity 4103.1)

Fuel Pump Development — No feed pump diaphragm or check valve failures have occurred since February 1957. Heads now being tested have operated for 8521, 7389, 5800, 5800, 1991, and 1240 hr, respectively. Purge pump heads have operated 2844, 1600, and 1600 hr, respectively. Several check valves have operated in excess of 10,000 hr. A double-diaphragm head is pumping 2.3 gpm of fuel solution. An experimental head screened with 20-μ Poroloy is ready for test.

The turbine pump completed its initial run in water successfully. Pumping of uranyl-copper sulfate solution was interrupted because of copper plating-out on two impellers. This difficulty was attributed to improper heat treatments conducted by the manufacturer; additional impellers are on order.

The Stellite-piston pump fitted with Haynes-25 rings was used for pumping uranyl sulfate for one month. Excessive wear of cylinder and rings was observed. Testing of this pump has been terminated.

The slurry check-valve-material test loop was operated with a 250-g/liter charge of 1600°C-fired $ThO_2$. The life of type 347 stainless steel and of Stellite trim was very short. Kennametal K-501 trim was operated successfully for 164 hr, at which time the loop became plugged. The system is being rebuilt to eliminate the latter difficulty.

A purchase order has been placed with Pressure Products Industries, who will build a reactor-prototype oxygen compressor that will deliver 2 scfm of gas at 2500 psi. (AEC Activity 4103.1)

Homogeneous Reactor Fuel Processing — Silvered Yorkmesh wire packed to a density of 29 lb/ft$^3$ was more effective than silvered alundum for removing iodine from a steam-oxygen-iodine stream at 120°C in the HRT mockup loop. Laboratory tests in which iodine absorbed on silvered Yorkmesh was subjected to various extreme conditions envisioned as possible during reactor operation, showed that the iodine was not displaced either mechanically or chemically. The incorporation of a silvered Yorkmesh bed before the platinum recombiner in the HRT was recommended.

Laboratory studies using simulated HRT fission- and corrosion-product solids have shown the irreversible deposition of such solids from $UO_2SO_4$ solutions onto stainless steel at temperatures up to 150°C to be no more than

the insignificant amount of 40 μg of solids per square centimeter, with most values being about 10% of that amount. The studies are being extended to 300°C. Hydroclones on corrosion test loops at Y-12 have collected corrosion-product solids from those loops at rates ranging from 5 to 100% of the estimated daily production rates, the average value being about 20%. (AEC Activity 4103.1)

Gaseous-Fission-Product Disposal — The HRT charcoal beds A and B were checked by passing oxygen containing $Kr^{85}$ through them and were found to be satisfactory for use with the gas flows expected from the reactor. Various molecular sieves have shown efficiencies ranging from 14 to 36% of that of charcoal for removing traces of krypton from a stream of dry oxygen at 25°C. Equilibrium adsorption isotherms for xenon on Linde molecular sieve 5A and charcoal used in the HRT adsorbers showed xenon to be adsorbed 12 and 11 times more strongly, respectively, than krypton. (AEC Activity 4103.1A)

## CIVILIAN-POWER ADVANCED-TYPE GAS-COOLED REACTOR (GCR)

Gas-Cooled Reactor: Physics — Calculations of fuel burnup indicate that, for high moderator temperature, core life depends less strongly on initial conversion ratio than had been anticipated, at least for initial multiplication factors of less than about 1.1. As a result, it appears desirable to extend core life by using considerably higher enrichment than is required just to accommodate the stainless steel cladding. For a given fuel enrichment, capital costs diminish with decreasing fuel-element spacing (increasing power density), but fuel costs rise (decreasing multiplication factor and core life). Minimum over-all costs appear to be realized with a lattice pitch of about 8 in. and fuel enrichment of around 2%.

It appears probable that for these conditions average fuel exposure of 8000 Mwd/ton (1000 kg) or greater can be achieved. Since this implies fuel residence times in the reactor of eight years or more, it follows that the benefits of staggered loading can be realized without continuous on-stream loading machines and that the fuel inventory held up outside the reactor will be relatively small.

Calculations show, for a fixed reactor power of 700 Mw, a helium inlet temperature of 450°F, and a peak design fuel surface temperature of 1300°F (exclusive of hot-spot allowances), that sizing the fuel channel diameters to match the radial power distribution can raise the mean helium outlet temperature 300°F for a radial peak-to-average power ratio of 1.5. If full advantage is taken of this possibility, the mixed-mean gas outlet temperature is within 30°F of what it would be with a flat flux distribution for the same pumping power. (AEC Activity 4119)

Gas-Cooled Reactor: Design — Stress calculations were made for the main helium ducts, and the duct layouts were determined. Bellows expansion joints were specified for both hot and cold ducts. The reactor support structure is to consist of a honeycomb grid of girders surmounted by a circular steel plate.

This structure is partially cantilevered from a series of vertical support members that carry the load directly through the pressure shell to the foundation so that the shell carries only its own weight.

A study of steam generators led to the selection of four straight-through boilers, 20 ft in diameter and 60 ft high. The blower drive motors will be constant-speed a-c horizontal motors mounted completely inside the helium pressure system in order to eliminate the problem of seal leakage. Reactor control at part load will be accomplished by bypassing part of the helium flow around the reactor. This approach is considered to be acceptable in a base load plant, and it was selected for its inherent simplicity and reliability. (AEC Activity 4119)

Gas-Cooled Reactor: Experimental Engineering — Equipment was designed for an isothermal static-pot test of the compatibility of graphite, fuel element cladding materials, and helium in a stainless steel vessel at 1400°F. A more refined materials-compatibility test system that will provide thermal convection of helium with a temperature differential of several hundred degrees is being designed. (AEC Activity 4119)

Gas-Cooled Reactor: Metallurgy — Methods for preparing $UO_2$, for forming the desired shapes, and for sintering the shaped material to the desired density and tolerance were determined. The $UO_2$ is prepared from a low-temperature stabilization of ammonium diuranate. Specimens for irradiation testing are being prepared from $UO_2$ of the type to be used for the actual reactor.

Creep-rupture tests are under way in order to study the effect of section thickness on the mechanical properties of type 304L stainless steel. These tests are being conducted at 1200°F in an argon atmosphere on 0.060-, 0.020-, and 0.010-in.-thick sheet specimens. It appears that elongation during the first stage of creep is a function of section thickness, being greater for the thinner sections. Creep-rupture tests of various stainless steels in air and in argon at 1500°F indicate that air has a marked strengthening effect on type 304 stainless steel. The equipment required for a study of the effect of external pressures on fuel elements was designed, and fabrication is nearly completed. (AEC Activity 4119)

Gas-Cooled Reactor: Radiation Damage — Design work was completed on equipment for studying $UO_2$ fission-gas release, and the apparatus is to be installed in the LITR by January 15. Preliminary designs and cost estimates were prepared for modification of a hot cell as required for examination of irradiated $UO_2$ pellets. (AEC Activity 4119)

## MOLTEN-SALT REACTOR (MSR) PROGRAM

Molten-Salt Reactor: Design Studies — Work proceeded on the design of a large (~275-Mw, electrical) power plant that will utilize molten-salt fuel and breeding blanket mixtures. The design is being prepared in sufficient detail to permit cost analyses. On the basis of engineering and nuclear considerations

the salt mixture $LiF-BeF_2-ThF_4-UF_4$ (62.4-37-0.5-0.1 mole %) was selected as the fuel and the mixture $LiF-BeF_2-ThF_4$ (71-16-13 mole %) as the blanket.

The general design of the remote maintenance system was established. The radioactive portions of the plant will be in a cylindrical cell approximately 60 ft in diameter. A remotely operated crane that can be positioned above any piece of equipment in the cell will be located at the top of the cell. The crane will be capable of moving the equipment to an adjoining hot cell. A bridge below the crane will provide one or more remotely operated manipulators.

Layouts of low-power (10 to 50 Mw) test reactors were prepared for study. In these reactors the conditions of the high-power homogeneous reactor are simulated with cores of multiple fuel tubes through graphite. (AEC Activity 4103.1B)

Molten-Salt Reactor: Experimental Engineering — A method of fabricating pipe joints for the MSR system was tested. The joint consists of a flange designed to reduce the metal temperature to well below the freezing point of the salt at a point approximately 2 in. from the pipe wall and thus freeze the salt between the flange faces to prevent leakage. Gas leakage is minimized by a standard ring joint used at the outer edge of the flange as a gas seal. During 300 hr of operation the flange underwent 50 thermal cycles between 1150 and 1300°F and 50 thermal cycles between 1350 and 1500°F with no salt leakage. The helium leakage rate was approximately $1.2 \times 10^{-6}$ cc/sec. (AEC Activity 4103.1B)

Molten-Salt Reactor: Chemistry — Thermal-gradient quenching studies indicate that the liquidus temperatures of the following compositions are below 550°C:

| Component (mole %) | | |
|---|---|---|
| LiF | $BeF_2$ | $ThF_4$ |
| 74 | 12 | 14 |
| 64 | 18 | 18 |
| 68 | 10 | 22 |

Since the liquidus temperature should be affected only slightly by substitution of $UF_4$ for a small fraction of the $ThF_4$, it appears that useful breeder blankets and breeder fuels are available within these composition limits.

The solubility of $PuF_3$ at 600°C in $NaF-BeF_2$ mixtures containing 36 and 50 mole % $BeF_2$ appears to be about 140% of that for the $NaF-BeF_2$ mixture containing 43 mole % $BeF_2$. The solid phase in equilibrium with each melt was shown to be $PuF_3$. Attempts to establish the presence of plutonium alloyed with the nickel of the equipment used in these studies showed only traces (0.2 mg of Pu in a 25-g sample of nickel) which could readily be attributed to incomplete removal of salt from the sample surface.

The solubility of helium in the LiF-NaF-KF eutectic was found to be $11.3 \pm 0.7$, $17.5 \pm 0.2$, and $23.0 \pm 0.7 \times 10^{-8}$ mole of helium per cubic cen-

timeter of salt per atmosphere at 600, 700, and 800°C, respectively. These values are approximately one-half those for helium solubility in the $NaF-ZrF_4$ (54-46 mole %) mixture. The small difference observed for these quite different salt systems suggests that only relatively small variations in noble-gas solubility with composition of the fluoride melt are to be expected.

The 250-lb production equipment was operated for two weeks to produce some 600 lb of $LiF-BeF_2$ mixture. Operation was, in general, trouble-free, and the material produced apparently is of satisfactory purity. Monitoring of the operation by safety and health physics personnel failed to disclose serious hazards, but several practicable recommendations for future operation of the facility were made. (AEC Activity 4103.1B)

Molten Salt Reactor: Metallurgy -- Twenty-four of the 29 thermal-convection loops designed to provide materials compatibility data were operated. Eleven of the tests were completed, and the components were examined metallographically. The blanket salts with relatively high concentrations (7 mole % or greater) of $ThF_4$ showed less than 1 mil of attack, and thus it appears that $ThF_4$ is an even less serious oxidizing agent than $UF_4$ in these systems. Fuels containing 1 mole % $UF_4$ and coolants that were circulated in Inconel loops under the same conditions gave negligible attack. A zirconium-base fuel containing 4% $UF_4$ showed 2 mils of attack at 1250°F in 1000 hr of operation.

Studies involving circular-groove tests and weldments in restraint indicated satisfactory weldability of three heats of INOR-8 from three separate sources (Westinghouse, Haynes Stellite, ORNL). A possibility existed that at MSR temperatures creep would not be a factor in the design; however, relaxation tests of INOR-8 at 1200°F for more than 50 hr indicate that creep will be an important factor in the design of systems utilizing INOR-8. (AEC Activity 4103.1B)

Molten Salt Reactor: Advanced Design -- For a number of experiments, such as the production of higher transuranic elements, a high thermal-neutron flux is desirable. In theory, any arbitrarily high thermal-neutron flux is obtainable from a nuclear reactor if there is no limit on power and power density. In practice, costs connected with initial construction, burnup, and reprocessing put an upper limit on the available total power, and heat transfer considerations limit the power density.

The highest thermal flux obtainable from a given reactor power is being investigated by starting from the model of an idealized "reflector-moderated" reactor, which consists of a spherical shell embedded in an infinite moderator. The shell is assumed to contain the fissionable material, and its thickness is small compared with the radius but is large compared with the diffusion length in the shell material. The shell emits fission neutrons which slow down to thermal energies in the moderator, and some of the thermal neutrons return to the shell to cause new fissions. The moderator inside the shell acts as an internal thermal column in which the thermal neutrons pile up.

In such an idealized reactor with beryllium or beryllium oxide as a moderator, the maximum thermal flux would be about $10^{-3}$ neutron/$cm^2$ per fission neutron emitted in the shell, which corresponds to 130 Mw for a flux of $10^{16}$ neutrons$\cdot cm^{-2} \cdot sec^{-1}$. For heavy-water or graphite moderators the power requirements for the same flux are higher. (AEC Activity 4103.1B)

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR-1) — The Laboratory continued to furnish technical support to the Army Reactors Branch (AEC) on the 10-Mw pressurized-water reactor being tested at Fort Belvoir, Virginia, by Alco Products, Inc. The six-month test operation and training program, which was started on July 25, continued without major difficulties. Accumulated thermal power production now exceeds 3 Mw-years, or approximately 20% of the anticipated core life.

Improved fuel elements and control materials are being developed for core II of APPR-1. Low-silicon chromium was procured for use in fabricating control-rod absorber sections of $Eu_2O_3$ dispersed in elemental stainless steel. Several APPR-1 fuel plates were prepared in which the $B_4C$ concentration was increased from 0.14 to 0.21 wt % and in which type 347 stainless steel was substituted for the type 304L stainless steel as cladding. No difficulties with these changes were observed, but metallographic examination has not been completed.

Postirradiation examination of specimens of APPR-1 control material (3% B in Fe) revealed incipient damage by failure of the bond between core and cladding. The failures were anticipated from the results of previous examinations of samples which had received considerably less irradiation. Quantitative analyses of the burnup of boron atoms are being made. (AEC Activity 4601)

Gas-Cooled Reactor Development — A preliminary design of the gas-cooled reactor experiment was completed by Aerojet-General Corp., and members of the Laboratory staff participated in a critical review of the over-all program.

A re-evaluation of the cost and time schedule of the gas-cooled in-pile loop for operation in the ORR is nearly complete. The two compressors for circulating the loop coolant were received and inspected. The design of the control panel was completed, and many of the instruments have been received. A 16-in.-dia aluminum standpipe, which will prevent lowering of the reactor pool level in case of accidental rupture of the loop, was fabricated and installed in the ORR pool.

A mockup of the in-pile section of the loop was fabricated for a study of the flow pattern in the region adjacent to the test sample section. The heat losses from the loop were determined experimentally. (AEC Activity 4603)

Maritime Ship Reactor — The Laboratory continued to provide technical support to the AEC Maritime Reactors Branch (MRB) on the program for construction of the first nuclear-powered merchant ship, the "NS Savannah". Drawings and specifications for various components of the pressurized-water reactor and propulsion equipment being furnished by Babcock and Wilcox Company were reviewed, and recommendations were submitted to the MRB. A limited program for materials development support was established in the Laboratory's Metallurgy Division. (AEC Activity 4151)

GENERAL REACTOR RESEARCH

Basic Reactor Research: Experimental and Theoretical — Additional critical experiments were performed with the 32-cm-dia sphere in which the solution used was uranium oxyfluoride enriched to 93% in the $U^{235}$ isotope. The lining used to protect the aluminum shell from corrosion was replaced by one which has a negligible effect as a thermal-neutron absorber. Consequently, a slight change in volume, due largely to mechanical repairing of the entrance made to effect the change, was incurred. The volume increased from 17.02 to 17.06 liters. The corresponding change in critical mass, with an effectively infinite water reflector present, was from 855 to 829 g of $U^{235}$. In the unreflected case the mass change was from 2.129 to 2.198 kg of $U^{235}$. The reactivity temperature coefficient was determined as $-1.47 \times 10^{-4}$ per °C in the reflected system and $-2.27 \times 10^{-4}$ per °C in the unreflected system, based on a delayed neutron yield of $64 \times 10^{-4}$ (absolute).

Two additional experiments were performed in which the 32-cm-dia sphere was completely reflected with water and had 0.254- and 0.381-mm (10 and 15 mils) thicknesses of cadmium cemented to the aluminum shell. The required critical mass in the former case was 1.283 kg of $U^{235}$; the latter required only 10.7¢ additional reactivity. (AEC Activity 4202)

Fuel Element Development — The objective of the present program has been to develop a satisfactory fuel material for aluminum plate-type elements which contains uranium with a 20% enrichment of the $U^{235}$ isotope. The 20% enrichment limitation presents fabrication difficulties because of the higher concentration of uranium required. Investigations are currently being conducted on 48 wt % U—Al alloys and on dispersion-type fuels containing uranium oxides or uranium carbides.

Difficulties experienced in localized clad thinning have been markedly minimized with the selection of type 5154 aluminum frame material and the 48% U—Al alloy modified with a 3% Si addition. This combination results in a composite plate in which the average localized clad thinning is only 15% compared with a 65% thinning previously observed in experimenting with type 6061 aluminum and the unmodified 48% U—Al alloy. Successful fabrication of these composites is possible by alcladding the type 5154 aluminum prior to roll bonding with the type 1100 cladding material. No unusual rejection rate has been observed during this development.

Experiments with composites containing a 43 vol % dispersion of $U_3O_8$ in aluminum clad with aluminum strongly indicated the possibility of eliminating flux annealing from the process. This would eliminate the pitting problem resulting from flux annealing and might also bring about an improvement in the corrosion resistance of alclad fuel plates.

Heat treatment of experimental compacts containing dispersions of 50 wt % $UC_2$ in aluminum resulted in $CH_4$ and UC reaction products. Subsequent thermodynamic calculations revealed that the reaction $UC_2 + 2H_2 \rightarrow UC + CH_4$ is possible. Additional data showed that approximately 1% moisture can be removed from $UC_2$ powder while it is being heated to 600°C. It is postulated that $3H_2O + 2Al \rightarrow 3H_2 + Al_2O_3$ during the elevated temperature treatments, which provides the hydrogen source for reaction with $UC_2$. (AEC Activity 4203)

Thorex Process — Two pilot-plant runs were completed with highly irradiated (3000 and 3500 g of $U^{233}$ per ton) thorium decayed for 32 and 52 days. Continuous two-cycle operation, based on the ORNL Thorex co-decontamination flowsheet, was satisfactorily maintained for 240 and 330 hr. Potassium iodide to the dissolver and sodium bisulfite in the first- and second-cycle feeds were maintained by continuous metering. Thorium and uranium solvent extraction losses averaged 3.3 and 0.4%, respectively. Average over-all gross gamma decontamination factors were $1.9 \times 10^4$ for thorium and $1.3 \times 10^7$ for uranium. Product activity specifications were exceeded for the thorium product by 150 for gross gamma emitters and for the uranium product by 400 for ruthenium.

Throughout the runs equipment performance was good. The Fulflo filter elements were changed daily owing to the rapid rise in radiation intensity, which exceeded 100 r/hr after 24 hr.

In laboratory studies, sulfite added to the Thorex feed to increase the ruthenium decontamination (by destroying nitrites) reacted with nitrates at elevated temperatures to form NO gas. Used solvent was more efficiently decontaminated with a lime slurry than by the present carbonate treatment.

Decalin was not a satisfactory diluent for TBP in 2-in.-dia pulsed-column studies owing to the long (5.6-ft) HETS (height equivalent to a theoretical stage) and to a reduced flooding rate (about 75% of that with Amsco, the diluent now used).

A modified 25-TBP flowsheet was satisfactory for recovering $U^{233}$ from stored $Pa^{233}$-bearing aqueous waste. (AEC Activity 4301)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes by Sintering — An adiabatic self-sintering experiment is proposed that is based on the use of a high-level waste solution, mixed with shale, limestone, and soda ash, sintered in a furnace equipped with outside heaters operated at a temperature slightly lower than the sintering temperature in order to reduce the heat losses. The experimental facility (mixer, furnace, and off-gas system) has been designed. It will be located in two shielded underground vessels at the former site of pilot pits Nos. 1 and 2. Engineering design has been completed, and pretesting of the component parts has started.

The mechanisms responsible for the fixation of fission products in the ceramic sinter are of interest. Analysis for crystallinity by x-ray diffraction techniques has shown the presence of nephelite ($Na_2O \cdot Al_2O_3 \cdot 2SiO_2$) and other components not yet identified. Diffraction maximums at 8.6, 3.70, 2.52, 2.13, and 2.05 Å have been obtained. It is possible that some of the diffraction maximums assigned to nephelite are due in part to the unknown components. Since $Cs^{137}$ is one of the critical nuclides in the reactor waste complex, its retention in the sinter at temperatures less than the maximum sintering temperature is important. By using Hope solution and a cesium carrier, an equal weight of Conasauga shale has been found to take up 1.8 times as much cesium as montmorillonite does and 5.7 times as much cesium as kaolinite does by sorption at room temperature. (AEC Activity 4305)

Waste Disposal Research and Engineering: Disposal of Radioactive Wastes into Geologic Structures — The first step in the deep-well disposal project consisted in determining what types of waste can be disposed of in deep wells. Deep wells cannot handle fresh acid waste containing large amounts of suspended solids or dissolved solids which will precipitate out when the solution is neutralized. The wastes for the well must first be aged, neutralized, and filtered. A determination has been made of the probable composition of typical fresh wastes, and laboratory procedures have been developed for making nonradioactive, synthetic waste samples for preliminary laboratory work. Some progress has been made in methods of neutralization (with calcium rather than sodium as suggested by I. R. Higgins) which will result in an easily filterable precipitate.

Discussions with the U. S. Geological Survey have resulted in a tentative agreement to use synthetic aquifers rather than field samples in the first experiments on chemical compatibility, with the hope of reducing the number of variables. It is planned to begin with a column containing pure quartz sand and then to add, one at a time, measured amounts of feldspar, calcite, clay, and iron-oxide-coated sand grains.

The study of the solubility of rock salt in acid aluminum nitrate and Purex-type wastes in determining the feasibility of the disposal of radioactive waste in salt formation is being continued. Results for analytical-grade salt and rock salt from the Kleer Mine in Grand Saline, Texas, are identical and vary from 50 to 80% of the solubility of NaCl in distilled water, depending upon the concentration of the solvent. The solubility of the major impurities in rock salt, $CaCl_2$, $MgCl_2$, and $CaSO_4$ is also being investigated. The interaction between the salt and the waste-type solutions produced a gas which is presently being identified; chlorine is one of its constituents.

The concentration of the wastes in the test cavity sufficient to enable natural conduction to dissipate the heat generated by the decaying fission products is being calculated. The assumptions are that the liquid in the cavity is well mixed, that the surrounding salt is infinite in extent, and that steady-state conditions prevail. Not included in the assumptions are the loss of the heat required to raise the temperature of the waste and the surrounding salt to the steady-state temperature, and the decreasing amount of heat generated per unit time as the fission products decay. (AEC Activity 4305)

Waste Disposal Research and Engineering: Dispersal of Radionuclides in the Environment — ORNL process wastes originate from equipment cooling water, decontamination-pad drains, floor drains, storage canals, evaporator condensate, and laboratory sink drains. They are characterized by volumes ranging from 100 to 200 million gal/year and by activities ranging from less than 50 to several hundred beta counts per minute per milliliter; $Sr^{89}$ and $Sr^{90}$ account for 20% of the gross activity, the total rare earths account for 46%, and $Cs^{137}$ accounts for 30%.

The result of the Laboratory's expanded research and development programs was an increase in the levels of $Sr^{89}$ and $Sr^{90}$ in the process waste stream and the Clinch River. A conventional straight-line type of lime-soda softening plant was designed to reduce these levels. Plant construction was completed during August 1957 at a cost of about $265,000. The plant has a capacity of 500,000 gal/day and employs gravimetric chemical feeding, flash mixing, coagulation, and sedimentation. An 800,000-gal equalization basin is provided to minimize fluctuations in chemical composition of the influent waste.

Up to the present time only lime and soda ash have been used in the treatment process. Operating experience has shown that the plant is capable of removing 85 to 90% of $Sr^{89}$ and $Sr^{90}$ and total rare earths. This waste treatment plant is unique in that it is the first to be designed and operated primarily for the removal of radiostrontium from large volumes of low level waste.

To determine the effectiveness of the White Oak Creek—Clinch River System in diluting and dispersing the wastes from ORNL, tracer level tests were run on July 4—7 and July 9—13. The TVA was conducting reaeration studies in the Clinch River during this period and maintained a steady flow in the river of 3000 $ft^3$/sec for the first test and 6000 $ft^3$/sec for the second test. Five curies of $I^{131}$ was injected into White Oak Creek at its confluence with the Clinch River for each test. Samples were taken continuously during the course of the test at Clinch River miles 19.6, 17.5, 15.5, and 13.1. Scintillation counters were used to measure the concentration of radioiodine flowing past the sampling station. Through a cooperative arrangement with the U. S. Geological Survey, the flow through this reach of the river was measured during the test. The velocity and temperature profiles at the sampling stations were also determined by the U.S. Geological Survey.

Additional downstream samples were collected at Centers Ferry, Clinch River mile 4.4, and at the Kingston Steam Plant, Emory River intake. (AEC Activity 4305)

Radiation Damage: Advanced Engineering and Development — Tests have been run to determine the effectiveness of a proposed adsorption trap for radioiodine in the off-gas system from experiments in the ORR. A simulated adsorption trap was set up with the linear air velocity equal to that expected in the ORR experiment off-gas system. The trap design simulated in this test provides for 1000 $ft^3$/min of air passing through an adsorption trap 2.75 ft in diameter by 1 ft deep. The simulated trap was 1/2 in. in diameter by 12 in.

long and had an air flow of 7 liters/min. Iodine-131 was generated in its elemental form and introduced into the air stream. The concentrations of radioiodine and "dead" iodine were very small compared with those expected if experiments having a fission power totaling 120 kw were to fail simultaneously. However, tests at the Chemical Processing Plant in Idaho have been run at 30 times the expected loading. The amount of iodine escaping from the trap and the distribution of the iodine in the trap were determined by radioassay. The adsorption materials used were activated charcoal and Linde molecular sieve material No. 13X. The charcoal trap retained 99.95% of the iodine, with 90% of the activity remaining in the first 3/8 in. of the bed. The distribution of iodine within the bed was unchanged after 20 hr at full air velocity. The molecular sieve material also retained 99.95% of the iodine, but only 55% of the activity remained on the first 3/8 in. of the bed. The activity moved downstream slightly during the 20-hr air flow test. These tests indicate that it is possible to remove iodine from the off-gas stream from experiments in the ORR by means of a charcoal adsorber bed of convenient size.

It has been shown experimentally that atoms in a solid may be displaced from their lattice sites by very energetic gamma rays (0.5 Mev or greater). (AEC Activity 4540)

## PROGRAM 5000 — PHYSICAL RESEARCH

### ISOTOPE PRODUCTION

Radioisotope Production — Six Hanford slugs were processed to produce a large supply of 8-day $I^{131}$ so that the equipment could be shut down for repairs. It is estimated that between 700 and 1000 curies were produced. Repairs to the equipment have been started, and the equipment is expected to be back in operation by the middle of January.

Approximately 75 g of purified sulfur, irradiated in the LITR, is being processed by distillation every three weeks on a routine basis. The resulting 14.3-day $P^{32}$ is being produced to supplement Graphite-Reactor-bombarded sulfur and also to further develop the LITR process which will be used exclusively in the near future.

Two runs were made in the short-lived fission-product equipment. In the first run Amsco-125 was used as the diluent instead of carbon tetrachloride. Extraction of rare earths from the 14 N nitric acid solution was again inefficient. Also, contact with concentrated nitric acid greatly increased the degradation of the TBP, causing appreciable amounts of phosphate in the aqueous phase; corrosion-product concentrations were also increased by this operation. This run was continued to the point at which dilution of the product solution was necessary, then was stopped because of precipitate formation.

In the second run, five extractions from 6 N nitric acid were made, and the fission-product solution was reduced in volume to 200 ml and was processed in a manipulator cell. The precipitate which formed upon dilution carried none of the activity but seemed to be a mixture of uranium compounds — phosphates and probably some oxide form. Following removal of the precipitate, the rare earths were precipitated with oxalate (lanthanum carrier), and ion exchange separations were begun. Barium-140 and $Sr^{89}$ products were prepared, but the rare-earth separation was not good. Most of the problems in the final purification of the short-lived fission products are due to the uranium left in the solution after the solvent extraction operation. Even with extraction efficiencies of greater than 99%, enough uranium remains — when the solution is reduced in volume — to cause precipitation.

Radioisotope Development — Phase II equipment installation in the Fission Products Pilot Plant was completed. Six window units, three model 8 manipulators, and the G-M model E manipulator were installed. Equipment testing is in progress.

The rate of gas formation from SRO acidic process waste was measured at 10 and 40°C. The average age of the waste during the 10°C test was 200 days, and during the 40°C test was 212 days. The average rate of gas formation was 1.0 cc/hr/gal at 10°C and 3.07 cc/hr/gal at 40°C.

The precipitation of tetravalent cerium, on a laboratory tracer scale, as the molybdate, pyrophosphate, or arsenate resulted in the recovery of up to 98.5% of the cerium essentially free from other rare-earth elements. The molybdate precipitation method can be adapted to recover cerium directly from Redox-type raw waste.

Air containing 10 μg of $I_2$ per cubic foot was passed through a 4-in.-dia packed tower to determine the effect of varying the packing height, caustic flow rate, and air flow rate on the removal of iodine from the air. The effect of air flow rate on the decontamination achieved was negligible over the range investigated. Increasing decontamination factors, which varied from 53 for 3 in. of column packing (1/2-in. beryl saddles) to 280 for 25 in. of column packing, were achieved with increasing packing height and caustic flow rates.

Investigations are continuing on the chemical process for purification of $Pm^{147}$. Equilibrium studies were made with 0.25 M lactate solution at varying pH and with Dowex 50 resin to determine the distribution of europium, promethium, and americium between solution and resin. The best conditions for separating promethium and europium are 0.25 M lactate solution at pH 4.5. (AEC Activity 5100)

Stable Isotope Separation — During the present carbon separation approximately 720 mg of $C^{13}$ has been collected. Production rates have been reduced in order to produce 90% $C^{13}$ from 1.1% $C^{13}$ feed material.

The first calutron separation of erbium isotopes was terminated on November 21, 1957. Starting with approximately 2 kg of 99% erbium oxide, an estimated 118 g of separated isotopes was collected during 3683 tank hours.

Separation of the six isotopes of selenium is in progress. More than 5 kg of $SeO_2$ charge material has been prepared by reacting elemental selenium with hot 8 N nitric acid; the selenium ions produced are collected in copper or aluminum receiving pockets. Monitored currents indicate a collection of 18 g thus far.

Chemical processing has been completed on new lots of $Ti^{46}$ and $Ti^{49}$ and on a complete series of tin (two samples each of all ten isotopes). The following lots of isotopes have been assayed and are ready for distribution: $Cr^{53,54}$, $Cd^{111}$, $Ge^{70}$, $Re^{185,187}$, $Ti^{46,47}$, and $Tl^{203,205}$.

The isotopes of erbium being recovered from the graphite collector pockets are being chemically refined. The isotopic material which was collected at the mass-172 position has been recovered and analyzed spectrochemically. It was found to contain approximately 2000 ppm of ytterbium. Inasmuch as ytterbium has a known isotope at mass 172, it will have to be removed by ion exchange techniques before the material can be studied for the possible natural existence of $Er^{172}$.

The following conversions of inventory forms were made: $Li^6$ metal to $Li^6F$; $Li^7_2CO_3$ to $Li^7_2SO_4$; $Li^7Cl$ to $Li^7$ metal; $Rb^{87}Cl$ to $Rb^{87}F$; $Si^{29}O_2$ and $Sn^{112}O_2$ to the elements. Isotopic targets of a number of elements were prepared by electroplating the elements from various appropriate solutions onto platinum disks for (p,n) studies in the ORNL High Voltage Laboratory.

Two inventory lots of $Mo^{95}O_3$ were combined into one homogeneous sample for the Harwell cross-section program by dissolving the samples with sulfuric and nitric acids, precipitating the combined molybdenum with α-benzoin oxime, and then igniting back to oxide at 500°C.

Stable Isotope Development — A high-temperature ion source, designated as the M-18, is being assembled. This source, essentially a larger model of the M-16 used in the high-resolution calutron, is expected to supersede the M-14 presently used in the separation of rare earths and other elements requiring high oven temperatures.

The high-resolution calutron equipment has been subjected to a series of runs using normal uranium as the charge material. The beam envelope was determined by scanning the beams across 0.040-in. slots. In later runs, ions were collected. Milligram quantities of $U^{235}$ were enriched from 0.7% feed to a maximum of 93.9% product, which represents an enhancement of approximately 2100. The production rate was 2 ma.

Development work is in progress to synthesize quantities of vanadyl trichloride ($VOCl_3$) to be used as charge material for a forthcoming separation of vanadium isotopes. The procedure currently being investigated involves

the reduction of $V_2O_5$ with hydrogen at dull-red heat (600—650°C) and subsequent chlorination of the lower oxide at a lower temperature (400—450°C). A previous calutron collection utilized vanadium trifluoride ($VF_3$) as the charge, but this compound is no longer available and is extremely difficult to prepare. (AEC Activity 5100)

PHYSICS

High Voltage Program — The large flat-response $4\pi$ neutron detector (R. L. Macklin, Nucl. Instr. 1 001—004, 1957) has been used in conjunction with the ORNL 5.5-Mv Van de Graaff singly and doubly charged alpha-particle beams to begin a few $(\alpha,n)$ reaction cross-section measurements. The $Be^9(\alpha,n)$ reaction, utilized extensively in radioactive neutron sources, has been investigated from threshold to 8 Mev. The $Li^7(\alpha,n)B^{10}$ cross sections indicate two small resonances at lower energies than the major one known from the inverse $B^{10}(n,\alpha)$ reaction (1.9 Mev). These correspond to small departures from the 1/v law for $BF_3$ detectors below 1 Mev.

Neutron transmissions have been measured with a resolution of about 3% over the energy range 2 to 20 kev for the following nuclides: $C^{13}$, $N^{14}$, $Al^{27}$, $Cl^{35}$, $Ti^{48}$, $Ti^{49}$, $V^{51}$, $Fe^{56}$, $Fe^{57}$, $Cu^{65}$, $Rb^{85}$, $Rb^{87}$, $Sr^{86}$, $Zr^{90}$, and Sm.

Coulomb excitation of higher states in several even-even nuclei has been detected. For $Te^{122}$, in addition to the first $2^+$ state at 564 kev, which is strongly coulomb excited, a very weak excitation of the second $2^+$ state was found at 1256 kev. This second $2^+$ state has also been observed to be excited in the beta decay of $Sb^{122}$. From angular correlation measurements on the cascade gamma rays following beta decay, the mixing ratio E2/M1 of the upper-cascade gamma ray has been measured as 10. The crossover E2 gamma ray has a matrix element about equal to that for a single-particle transition, whereas the E2 matrix element of the upper-cascade gamma ray is approximately 100 times larger than the single-particle estimate. The E2 matrix element for the decay of the lower cascade gamma ray is 36 times larger than the single-particle estimate. From the value of E2/M1 the size of the M1 matrix element for the upper-cascade gamma ray was found to be 1/100 of the single-particle estimate.

Tungsten-186 is a permanently deformed nucleus which exhibits excited states fitting a rotational sequence. The first $2^+$ state is at 122 kev and is strongly coulomb excited. Its E2 matrix element is 113 times larger than the single-particle estimate. In addition, a state at 730 kev was also observed to be coulomb excited. This may be the first vibrational state. The matrix element for E2 excitation of this state is 5 times the single-particle estimate. (AEC Activity 5220)

The 86-Inch Cyclotron: Nuclear Physics — Angular distributions of alpha particles from $(p,\alpha)$ reactions induced by 23-Mev protons were measured. They are peaked in the forward direction for all alpha-particle energies. Anomalous inelastic proton scattering was studied with magnetic analysis at 45 deg.

At this angle there is no interference from carbon or oxygen peaks; thus it is possible to study essentially all elements by making suspensions in polystyrene of chemical compounds containing only the element of interest and carbon, oxygen, and hydrogen. The effect was traced through the rare earths (where its behavior was found to be somewhat less regular than in other parts of the periodic table) and in other regions where metal foil targets are unavailable. (AEC Activity 5220)

The 86-Inch Cyclotron: Applied Physics — The isotopes $Ti^{44}$, $As^{74}$, $Sr^{85}$, and $Pb^{202}$ were produced in service irradiations for nonproject customers. A test loop for corrosion studies was bombarded in the external beam for the ORNL Chemistry Division. (AEC Activity 5220)

Heavy-Particle Physics — Measurements were continued on the light-particle spectra from the nitrogen bombardment of oxygen. The value of a in the level density formula $w = C \exp 2 (aE)^{1/2}$ was found to be $1.3 \pm 0.2$ from the reaction $O^{16}(N^{14},\alpha)Al^{26}$; this value appears to be independent of the angle of observation. Excitation functions were measured for the production of $Ti^{45}$, $Sc^{43}$, $Sc^{44}$, and $K^{38}$ from the nitrogen bombardment of sulfur. (AEC Activity 5220)

Electronuclear Machines — The design of the 8-sector coils for Cyclotron Analogue II is about 95% complete; the Analogue is an electron model of the proposed 900-Mev azimuthally-varying-field (AVF) proton cyclotron. A code was written for the digital computer to determine the degree of approximation required when computing, precisely, the field of the sector coils. A study was made and a computer code was written for finding the shape of a resonant chamber which will accelerate the electrons with nearly uniform voltage at all radii. The resonant chamber must be 90 deg in azimuthal width and must operate at 234 Mc/sec. The shape of a resonator which has a voltage standing wave along the accelerating lip that is constant within 25% was determined experimentally. (AEC Activity 5240)

Mass Spectrometry and Related Techniques — The positive and negative mass spectra of chlorine gas were studied for pressures ranging from 0.001 to 50 mm. Radical ions, $Cl_3^+$ and $Cl_3^-$, were observed and studied as a function of pressure. Experimental evidence indicates that these ions arise from the ionization of the $Cl_3$ radical produced by the light reactions $Cl_2 \rightleftarrows 2Cl$ and $Cl + Cl_2 \rightleftarrows Cl_3$. (AEC Activity 5250)

## CHEMISTRY

Raw Materials Chemistry — As previously reported (ORNL-2166), thorium can be recovered from Blind River uranium-barren ion exchange (IX) effluents by the Amex (amine extraction) process. Chloride concentrations resulting from chloride IX elution do not significantly affect the Amex thorium process, but nitrate concentrations resulting from nitrate IX elution seriously depress the thorium extraction. The interference can be avoided by amine extraction of the nitrate, and the resulting recovery of the relatively expensive nitrate

for recycle appears to be economically attractive. The nitrate can be extracted either before the thorium extraction or simultaneously with it.

As reported previously (ORNL-2426), the preferential extraction of strontium from $NaNO_3$ solution by di(2-ethylhexyl)phosphate (D2EHP) was independent of pH between 9 and 12. The strontium extraction coefficient (o/a) was ~10 with 0.1 M D2EHP from 0.5 M $NaNO_3$ at pH >7, and dropped to <1 at pH <3. Extraction was enhanced, however, at pH's between 4 and 7, with the extraction coefficient approaching 200 at pH ~5. The high extraction is explained on the hypothesis of a chelate acid salt (analogous to the uranyl complex $UO_2X_4H_2$ formed with the same reagent, ORNL-2166) stable near pH 5 but destroyed by hydrolysis at low pH or by neutralization at high pH. (AEC Activity 5310)

High-Temperature and Structural Chemistry — The specific electrical conductance of solutions of sodium metal in fused sodium iodide was found to vary similarly to solutions in the fused bromide, namely, to increase with the sodium metal concentration. The equivalent conductance of the metal decreases rapidly from (extrapolated) values of more than 12,000 $ohm^{-1}.cm^2$ at infinite dilution, or more than 9000 at 0.2 mole % metal, to 3400 and 3700 $ohm^{-1}.cm^2$ at saturation concentrations of 2.25 and 6.3 mole % at 700 and 800°C, respectively. At 900°C the equivalent conductance drops to a minimum of 4850 at 8 mole % before reaching the value of 5500 at the solubility limit of 12.1 mole % metal. The solubility values from these measurements are slightly lower than those reported here earlier, which were probably somewhat affected by the difficulty of achieving complete phase separation.

Zinc and cadmium, similarly to magnesium, were found to suppress the transition of calcite into aragonite that occurs when cp calcium carbonate is ground in a mortar. (AEC Activity 5330)

Organic Chemistry — Continued investigation of thoria slurries in molten diphenyl revealed that, in addition to the dispersion effected by the adsorption of a polar organic molecule (carboxylic acids, aryl sulfonic acids) from solution, disperson can result from reaction on the thoria surface. Dri-Film (an organo-silicon chloride) reacts with moist thoria to form a protective, wettable (by diphenyl) surface. The dispersons formed by either treatment were unaffected by static tests at 255°C for as long as two weeks. Dynamic testing will be handled by other divisions of the Laboratory. (AEC Activity 5330)

Chemistry of Fused Salts — The self-diffusion coefficients of the nitrate ion in molten $LiNO_3$ and $AgNO_3$ were determined. The results may be expressed in the form of a rate equation $D = Ae^{-\Delta H/RT}$, where D is the self-diffusion coefficient and $\Delta H$ is the heat of activation. In $LiNO_3$, A and $\Delta H$ are 1.95 x $10^{-3}$ $cm^2/sec$ and 6.3 kcal, and in $AgNO_3$ they are 0.31 x $10^{-3}$ $cm^2/sec$ and 3.8 kcal, respectively. The $\Delta H$ value for the $NO_3^-$ in $LiNO_3$ is appreciably higher than that for the $Li^+$ (5.5 kcal), whereas $\Delta H$ values for the cations and anions of all the other nitrates measured ($NaNO_3$, $KNO_3$, $CsNO_3$, and $AgNO_3$) are approximately equal within experimental error.

A "mobility" of the cation may be defined as being equal to $D_+/(D_+ + D_-)$. These "mobilities" in $LiNO_3$, $NaNO_3$, $KNO_3$, $CsNO_3$, and $AgNO_3$ are 0.73, 0.61, 0.53, 0.50, and 0.64, respectively. (AEC Activity 5330)

Analytical Chemistry Research — Since trioctylphosphine oxide (TOPO), dissolved in an inert organic solvent, is known to extract certain elements only from acidic solutions, experiments were conducted to evaluate the extractibility of the acids HCl, $HNO_3$, $H_2SO_4$, $HClO_4$, and $H_3PO_4$. Equivolumes (10 ml) of 0.1 M TOPO in cyclohexane and of a particular acid were equilibrated, from which the amount of acid in the TOPO phase was calculated. The limiting molar ratios of acid to TOPO were found to be 2 for HCl or $HNO_3$, 1 for $HClO_4$ or $H_2SO_4$, and 0.5 for $H_3PO_4$. An attempt to utilize absorption spectra as a means of evaluating acid extraction met with failure, since the spectra were essentially the same throughout the range examined. The only absorbance that was observed occurs in the near-ultraviolet region at about 320 mμ.

The extraction characteristics of TOPO have been applied to the separation of uranium and bismuth in a single equilibration. If a solution containing hexavalent uranium and bismuth is made 5 M with $HNO_3$ or HCl, uranium can be completely extracted therefrom by the use of a 0.05 M solution of TOPO in cyclohexane, while bismuth remains in the aqueous phase. Under these conditions trace quantities of uranium, 0.05 μg/ml, can be effectively separated from a solution containing as much as 50 mg of bismuth per milliliter, with a coefficient of variation of about 10%.

A colorimetric method was developed for the direct determination of zirconium in TOPO-cyclohexane extracts. Pyrocatechol violet, the chromogenic reagent, forms a colored complex with zirconium which exhibits maximum absorbance at a wavelength of 660 mμ. At this wavelength the molar absorbancy index is 38,500. The absorbancy of the complex adheres to Beer's law for concentrations of zirconium up to at least 4 μg/ml. Since up to 20 mg of zirconium can be extracted by TOPO-cyclohexane from either acidic chloride or nitrate solutions in a single equilibration, the method can be applied to the separation and determination of zirconium from solutions of these types. The coefficient of variation of this method is about 3%.

## METALLURGY

Fundamental Physico-Metallurgical Research — To study the stability of an aluminum rod ⟨111⟩ orientation, a rod containing an 88% ⟨111⟩—12% ⟨001⟩ texture was cold-swaged to give reductions in area of 60, 80, 90, and 96%. The axis distribution charts showed that the relative volumes of the components did not change for reductions of 60, 80, and 90%, but the amount of ⟨001⟩ texture increased slightly for the 96% reduction. The sharpness of the ⟨111⟩ texture was greatest in the 60%-reduced specimen and decreased with increasing deformation. A marked spreading toward the ⟨112⟩ orientation from ⟨111⟩ was observed in the 96%-reduced specimen.

The specific heat of alpha-annealed crystal-bar zirconium in the liquid-helium temperature range has been found to be $c = 2.736 \times 10^4 T + 1.943 \times 8 \times 10^{10} (T/282.5)^3$ ergs/deg. These experimental values have been corrected for the heat capacity of the specimen heater and the graphite thermometer, obtained from an independent measurement.

The specific heat of zirconium quenched from the beta phase is $c = 2.769 \times 10^4 T + 1.943 \times 8 \times 10^{10} (T/286.6)^3$ ergs/deg . Thus quenching through the allotropic transformation increases the density of states of the electron and shifts the vibrational spectrum of the atoms presumably because of the disorder introduced into the crystal by the transformation.

Preliminary vapor pressures of a body-centered cubic zirconium-cadmium alloy containing 11.3 at. % Cd revealed positive deviations from Raoult's law in contrast with the negative deviations observed in the alloy containing 7.5 at. % Cd. Since the partial pressure of zirconium is negligibly small over these alloys, the cadmium vapor pressures give the $\beta/(\beta + \text{gas})$ phase boundaries for 1 atm external pressure. The values are 7.5 at. % Cd at 1006°C and 11.3 at. % Cd at 931°C. (AEC Activity 5420)

Fundamental Investigations of Radiation Damage in Solids — Polycrystalline specimens of the semiconducting intermetallic compound InAs have been irradiated in the ORNL Graphite Reactor. The samples were shielded from thermal-energy neutrons by indium and cadmium foils. The carrier concentration and conductivity of n-type material increased under fast-neutron irradiation. A total exposure of $2 \times 10^{17}$ fast neutrons resulted in the addition of approximately $2 \times 10^{17}$ current carriers as determined by Hall coefficient measurements. The conductivity of p-type material decreased rapidly to a conductivity minimum and then increased in a manner analogous to that of n-type material. Hall coefficient measurements following radioactive decay will be employed to ascertain whether the p-type material was converted to n-type as would be indicated by the conductivity changes.

Measurements of the thermal conductivity, K, of vitreous silica have been made at low temperatures before and after fast-neutron bombardment. The observed increase of K upon bombardment is in agreement with the prediction of Klemens'. Over the exposure range used, K increases approximately linearly with dose, whereas the density appears to have become nearly saturated, suggesting that the two properties are not directly related. A possible explanation for the K increase upon bombardment is that the neutron bombardment relieves macroscopic strains. Alternatively, it is possible that bombardment causes an increase in local order in the vitreous material. (AEC Activity 5430)

## BIOPHYSICS

Ecological Studies — Preliminary analyses of insect samples taken on White Oak Lake bed vegetation during the summer of 1957 showed some change in species composition compared with 1956 samples. Total numbers of insects and biomass estimates were smaller in 1957, probably as a result of meteorological phenomena. Biomass of herbivorous insects averaged about 200 mg per 10-sweep sample. Estimates of $Cs^{137}$ in herbivores ranged from $3.8 \times 10^{-5}$ to $6.0 \times 10^{-4}$ μc per 200-mg sample. Most samples of predators contained no significant amounts of radioactivity. (AEC Activity 6440)

Waste Disposal Research: Soil Disposal of Intermediate-Level Radioactive Wastes — By geologic and hydrologic field tests, the U. S. Geological Survey has intensively studied the 4-acre site in Melton Valley to determine parameters that control the movement and containment of water in the Conasauga formation. The results show that the formation yields less than 1 gpm per foot of drawdown in the pumped well and that movement along the strike of the rocks is greater than in the direction of the dip.

The rate of seepage from pit 2 has increased each year; however, the rate of seepage from pit 3 decreased in 1956, despite a consistently higher pit stage. This is believed to have been due to the raising of the water table around pit 3, resulting in lower water table gradients. Temperature changes are observed to affect the rate of seepage. There is nothing in the record of the pits that suggests a marked change in the permeability of the soil.

Although $Cs^{137}$ has not reached observation wells in quantities sufficient to detect, it has been detected in trees and the litter in the immediate environs. This material may be airborne, and a statistical analysis of the activity collected by standard gum paper techniques showed that the amount of airborne activity in the pit area was 2-1/2 times greater than that at similar off-site stations.

From laboratory column studies the total capacity of the shale for cesium was ascertained to be 0.34 mc/g. Breakthrough capacity is at least dependent upon the rate of flow, the depth of the column, particle size, and equilibrium conditions. Based upon the average breakthrough capacity of three columns, each cubic yard of shale can treat about $6 \times 10^4$ gal of ORNL waste. (AEC Activity 6440)

## PROGRAM 8000 — WORK FOR OTHERS

Professional Health Physics Training Program — Three members of the Health Physics Division conducted the Second International Course in Health Physics jointly sponsored by the World Health Organization, the Belgian Government, and the U.S. Atomic Energy Commission. The course extended over a five-week period and was held at the Centre D'Etudes Nucleaire, Mol, Belgium. The 26 participants represented the following countries: Austria, Belgium, Bulgaria, Egypt, France, Germany (West), Greece, Iran, Iraq, Italy, Lebanon, Netherlands, Poland, Portugal, Russia, Switzerland, Turkey, and Yugoslavia. (AEC Activity 8780)

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## VISITORS

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