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STATUS AND PROGRESS REPORT

OCTOBER 1954

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

October, 1954

W. H Sullivan

and

A. Savolainen

Date Issued: NOV 5 1954

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October, 1954

The subject material of this report represents approximately half of the Laboratory's program, which is covered with some exceptions on a bimonthly schedule.

PROGRAM 2000 - SOURCE AND FISSIONABLE MATERIALS

U^{235} Recovery - In a third trial run on the proposed first-cycle solvent extraction flowsheet in 3/4-in.-dia. pulsed columns, a gross beta decontamination factor of 2.7×10^4 was obtained, a fivefold increase over that obtained in the previous run (ORNL-1781). The principal contaminants were ruthenium and rare earths. The increased decontamination is attributed to the use of 0.75 M instead of 1.0 M aluminum nitrate as scrub solution.

In batch countercurrent tests on the second solvent extraction cycle a gross beta decontamination factor of 1.7×10^3 was obtained. The second cycle feed used was a mixture of first cycle product which had had an average decontamination factor of 6.3×10^3, giving an overall decontamination factor of about 1×10^7. The chief contaminant in the second cycle product was zirconium. An overall factor of about 4×10^7 is needed to meet the product activity specifications when 300-day-irradiated 90-day-cooled U-Al alloy is processed. The additional decontamination required can probably be gained by adsorption of zirconium from the second cycle uranium product solution on a silica gel bed, a decontamination technique successfully applied to the Purex process.

Ruthenium extraction from the 25 process dissolved alloy feed [1.8 M $Al(NO_3)_3$, 0.12 M HNO_3] by 6% TBP in Amsco diluent is undesirably high, the distribution coefficient (O/A) being 0.2. In batch equilibration studies, the addition of 0.06 M Fe^{++} and 0.5 M urea to the feed reduced the ruthenium distribution coefficient to 0.01; however, the additives promoted gassing, which might interfere with solvent-extraction column operability. It was shown also that the Ru distribution coefficient (O/A) was a function of the acid concentration, with a maximum value of 0.34 in 0.03 M HNO_3 and 0.00014 in both 2.5 M HNO_3 and in 1.23 M acid-deficient systems. The extracted ruthenium was effectively scrubbed from the extract with 3 M HNO_3 [Ru D.C. (O/A) = 0.01] in 15 min; with the 1 M $Al(NO_3)_3$ scrub originally specified in the flowsheet the ruthenium distribution coefficient (O/A) was 0.21. This scrubbing reaction is apparently time-dependent. (AEC Activity 2560)

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PROGRAM 3000 - WEAPONS

Special Electromagnetic Separations - Relatively pure Pu^{240} and Pu^{241} were obtained in the fifth calutron separation of plutonium isotopes. Sixty grams of plutonium were vaporized in a single-arc calutron developed especially for the efficient processing of small batches. Conversion of the vapor to well-focused Pu^{+} ions was 6% efficient. The isotopic composition of the feed was 84.5% Pu^{239}, 12.7% Pu^{240}, 1.71% Pu^{241}, and 0.22% Pu^{242}. Although the product assays (atom percent) are not complete, they will probably be similar to the previous run: 80% Pu^{240}, 60% Pu^{241}, and 99% Pu^{239}. (AEC Activity 3601)

PROGRAM 4000 - REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Design - A revised flowsheet for the low pressure systems of the Homogeneous Reactor Test was issued. Final drawings and specifications were issued for the entrainment separator, catalytic recombiner, recombiner condenser, dump tanks, and high pressure valves. Design was started on tools and fixtures for remote maintenance of equipment inside the cell, and a preliminary flowsheet for the auxiliary and emergency cooling system was developed. Detailed piping design was started. (AEC Activity 4109.1)

Homogeneous Reactor Test (HRT) - The concrete work for the HRT control room was completed and steel was erected for the shielding tank foundation. The design of the tank top-plug structure was revised on the basis of a 30-psig internal pressure. This provides a safety factor of two above the pressure calculated to result from the sudden release of all the liquid in the reactor. Specifications for the top plugs were sent out for bids. Design drawings and specifications were completed for the two chemical separations cells which will adjoin the south side of the reactor tank.

A scale model, representing the assembled reactor with its connecting process piping, was completed and is being studied from the standpoing of improving piping and equipment layouts. The aluminum mock-up of the reactor core tank was found to collapse at an external pressure of 260 psi, the calculated value. This first model was not as strong structurally as will be the actual Zircaloy-2 core vessel. As a result of screen embrittlement, perforated plate will be used instead in the diffuser section of the core tank.

A total of $964,000 has been committed in contracts and orders for reactor parts. This represents more than 90% of all the materials that will be needed for the construction of the HRT facility. (AEC Activity 4109.11)

HRP Blanket Engineering Development - The HRT mock-up design for the slurry blanket has reached the point where a large fraction of the components can be purchased. Tests with a small model pressure vessel indicate that sedimentation of slurry solids in the pressurizer can be controlled by injecting slurry into the vessel.

High temperature loop tests appear to show an important effect of sulfate in inhibiting attack by the slurry on stainless steel in the presence of oxygen. These tests and room temperature laboratory tests also show that sulfate promotes an increase in slurry viscosity. Sodium or thorium silicate reduces the viscosity at room temperature. (AEC Activity 4109.31)

HRP Chemistry--Fuel Studies - Continued work on the pressure-volume-temperature-composition relationships in the two-liquid phase region of the system UO_3-SO_3-H_2O has produced data for the volumes of each of the three phases as a function of temperature and fraction filling for two stoichiometric system compositions.

Solubility studies in the four component system NiO-UO_3-SO_3-H_2O have shown that the surface representing saturation in the NiO-UO_3 rich portion of the composition space isotherm is essentially a plane surface at 25°C, 100°C, 175°C, and 250°C, respectively. The compositions of two new complex solid phases formed at 250°C have been partially fixed as $NiO \cdot UO_3 \cdot SO_3 \cdot xH_2O$ and $NiO \cdot 2.3\text{-}2.5\ UO_3 \cdot SO_3 \cdot 6H_2O$.

Solubility studies in the CuO rich portion of the system CuO-SO_3-H_2O at 25°C have failed to yield uniquely interpretable data. Work on this system and the system CuO-UO_3-SO_3-H_2O will therefore be confined to temperatures above 100°C where equilibrium is more readily obtainable.

A series of static corrosion tests with a uranyl chromate solution at 265°C has indicated that corrosion of carbon steel is less than that with uranyl sulfate solution by a factor of 25 to 70. On stainless steel type 304, the chromate was less corrosive than the sulfate by a factor of 30 if no oxygen was added to the system. In the presence of oxygen the two solutions did not show a measurable difference, both giving extremely low corrosion rates. (AEC Activity 4109.31)

HR Blanket Studies--Chemical Development - In out-of-pile studies pure thorium oxide slurries acted as catalysts for combining hydrogen and oxygen gas. Approximately 0.05 mole of hydrogen per liter of slurry (530 g of thorium per kilogram of water) was combined with oxygen at 300°C and a hydrogen partial pressure of 500 psi (oxygen pressure 250 psi). The presence of 4 to 8% uranium as the oxide in the thorium oxide slurry doubled the combination rate. The addition of 0.1 m cupric oxide to the thorium oxide slurry increased the combination rate to 1.7 moles of hydrogen per liter of slurry per hour. This rate is calculated as being sufficient to maintain at 2000 psi a slurry blanket at 300°C which is dissipating 6 kw of fission heat per liter of slurry, assuming a $\underline{G}$ value of 0.8 (molecules of H_2 produced per 100 ev of fission energy absorbed).

In in-pile irradiation studies, the recombination rate of hydrogen and oxygen in the presence of mixed thorium oxide--uranium oxide slurry was approximately the same as that obtained in out-of-pile studies. The irradiated slurry contained 530 g of thorium, as the oxide, per kilogram of water and 8% U^{235} as the oxide. The irradiation was carried out for six days in the ORNL graphite reactor at a flux of 7×10^{11} $n/cm^2 \cdot sec$, which corresponds to a fission heat generation of 1 kw per liter of slurry. With the gas phase at 200° to 250°C and the slurry phase at 290° to 300°C, radiolytic gas pressures in excess of steam varied between 1000 and 2000 psi and inversely with the gas phase temperature. Analysis of the data indicated a G value of approximately 0.8.

A concentrated thorium oxide slurry (prepared from thorium formate), which had become plastic upon being pumped at 250°C and cooled to room temperature, became fluid again upon addition of 0.01 m sodium silicate. Sodium carbonate, sodium pyrophosphate, and sodium hexametaphosphate were also found to be good dispersing agents for thorium oxide slurries. Particle size analyses and X-ray diffraction studies on the pumped material and the effectiveness of deflocculents in restoring fluidity to the pumped slurry suggest that the increase in viscosity during the pumping results primarily from a degradation of the slurry particles and subsequent flocculation of the fragments.

X-ray diffraction patterns indicated that thorium hydroxide both as precipitated from thorium formate solution and after being dried at 105°C was amorphous, but electron diffraction patterns showed a crystalline structure in each case. Autoclaving the moist hydroxide in water at 250°C or drying it at 500°C converted it to material shown by both X-ray and electron diffraction patterns to be crystalline but did not increase its abrasiveness. (AEC Activity 4109.31)

HRP Miscellaneous Equipment - A stainless steel bellows valve operator with a 1/2 in. stroke was designed and tested. It was operated a total of 110,000 cycles before developing a minute air leak. An additional 116,000 cycles were required to make it unusable. A loop for testing the performance and useful life of dump valves and trims is under construction.

Results of experimental operation of the simulated HRT entrainment separator suggest that wire mesh by itself may not be sufficient, and that other methods of removing entrainment will have to be tried. (AEC Activity 4109.4)

HRP Core and Blanket Systems Development Studies - The Newport News Shipbuilding and Dry Dock Co. has developed methods for rolling, forming, forging and welding Zircaloy-2 in preparation for fabrication of the HRT core vessel. They have procured plate in 3/8- and 5/16-inch thicknesses for the vessel, and their forming and welding procedures have been approved.

Hemispherical heads for construction of the pressure vessel are on order with Lukens Steel Company. To date they have successfully made one head. Two

have been made but the bonds between the stainless steel cladding and the carbon steel base material were unsatisfactory.

Tests of one aluminum prototype of the core vessel are complete, and two bellows were tested for use as an expansion joint between Zircaloy-2 and steel. One bellows, manufactured by Zallea Bros., is satisfactory. (AEC Activity 4109.4)

Gas Handling Equipment - The electrical control and power circuits of the high-pressure recombiner loop were completed during the past month. The electrolytic cells were operated at atmospheric pressure without trouble.

The ORNL gas circulator was calibrated at high frequencies. A series of 30 start and stop tests were also performed. It has been found that the circulator will not operate satisfactorily for extended periods of time when circulating completely non-condensible gas because the bearings begin to run dry. (AEC Activity 4109.4)

HR Fuel Circulating Equipment - A contract was negotiated with Westinghouse to supply two 400 gpm pumps and two 230 gpm pumps for the HRT. One of each of the above pumps will be all stainless steel; the others will have titanium replacements for those parts which are subject to high velocity and/or turbulence at temperatures above 212°F.

Adjustment of various instruments and mechanisms on the 4000 gpm loop has resulted in smoother operation. The loop operates at 1000 psi and heats to 250°C in about 2 hours. The system was operated intermittently to accumulate information on thermal cycling.

The Pulsafeeder pump loop, which had been operated using Ames ThO_2 slurry, was recharged with Lindsay ThO_2. In subsequent operation the valve erosion rates were greatly increased.

The ORNL 5 gpm pump, operating on an endurance test to determine life expectancy of the various parts, has run for a total of about 4500 hours, circulating water at a temperature of 250°C. (AEC Activity 4109.5)

Heat Removal Equipment - Tube joint welding development for the 50 Mw main fuel heat exchanger, employing manual techniques, is nearing completion. Investigation of tube testing methods is continuing.

Information has been obtained on the costs of tube sheet machining, tube joint preparation and welding, stainless steel tubes, steam drum fabrication, and on other construction and testing costs relating to the 300 Mw heat exchanger.

Design of the HRT fuel and blanket heat exchangers, which are duplicate units, is nearing completion. The only major change has been the addition of a perforated baffle plate across the entire water surface to permit satisfactory operation at heat loads up to approximately 10 Mw. (AEC Activity 4109.5)

Homogeneous Reactor Instrumentation - A report presenting the HRT control circuit and setting forth a proposed operational procedure has been issued.

The conceptual design for the actuators of the HRT 1/2π valves was completed and released on an alternate bid proposal.

Layout of the control and auxiliary rooms has been completed, together with the detailed design of supporting frames and cabinets. The general design criteria for sensing elements inside the main shield have been issued.

Purchase orders have been completed for 60% of all instrument items. (AEC Activity 4109.6)

HR Fuel Reprocessing - In dynamic solubility tests using a circulating loop of 10 liters capacity, the solubility of cerous sulfate at 280°C in simulated homogeneous reactor fuel solution (0.02 m uranyl sulfate, 0.01 N sulfuric acid) was about 0.006 g/kg H_2O. In static experiments (10 to 1000 ml) the value was 0.004 g/kg H_2O.

Rare earth sulfates, containing Nd^{147} as a tracer, that had been coprecipitated with iron and chromium hydrous oxides at 280°C, did not redissolve when the simulated homogeneous reactor fuel was cooled rapidly to room temperature. Removal of the rare earth sulfates from a simulated homogeneous reactor fuel solution by centrifugation at room temperature may therefore be possible.

Iodine was removed from a simulated uranyl sulfate fuel solution by heating the solution to 275° to 300°C and contacting the gas phase (oxygen) above the solution with a metallic silver wire. Iodine removals as high as 80% were obtained, and the amount removed was independent of the initial oxidation state of the iodine.

In further liquid-solid separation studies with 1/2- and 1/4-in.-dia. hydroclones, 90 to 95% of water-suspended ThO_2 particles from 0.7 to 8 μ in diameter were recovered in the underflow at feed flow rates of 1 and 0.2 gpm, respectively. The rate of 0.2 gpm with the 1/4-in. hydroclone is near the proposed rate for continuous processing of the fuel of the HRT.

A study of the fuel and chemical processing costs for a three-reactor power station indicated that unit costs, including inventory charges, would be $12.94 per megawatt-day of heat or 2.3 mills per kilowatt-hour of electricity produced. The process considered included removal of the insoluble high cross section fission products from the uranyl sulfate core solution by liquid-solid centrifugal separators and solvent-extraction processing of the combined core solution and blanket slurry in a 200-kg/day Thorex plant.

Calculations of the hazards due to radioactivity produced during operation of homogeneous reactors indicate that I^{131}, Ba^{140}-La^{140}, and total rare

earths are biologically the most dangerous radioisotopes. The relatively lesser importance of Sr^{89} and Sr^{90}-Y^{90} shown by this study, in contrast to their importance indicated by other studies, results from attention to the short-term hazard aspects of a leak only and does not take into account the long-term considerations of fission product dispersal. (AEC Activity 4109.81)

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR) - Fuel and burnable-poison requirements for the ORNL design for the APPR were investigated by three methods; discrepancies of only a few percent were found and these can be related to limitations in one or another of the methods. The methods included a modified two-group hand calculation; a three-group, three region diffusion theory method, coded for the ORACLE; and a 30-group, nine-region diffusion theory, coded for the UNIVAC. (AEC Activity 4250)

AIRCRAFT NUCLEAR PROPULSION PROJECT

Aircraft Reactor Experiment - The leak which necessitated the dumping of the sodium from the system was repaired, and some modifications were made as the result of events that occurred during the dumping operation. On October 16 the sodium was recharged to the system at 600°F, circulated, and dumped four times to check thoroughly the operability of the system. Analyses of the sodium at 600° and at 1000°F showed the oxygen content to be satisfactorily low (of the order of 0.025%), and therefore, the present loading will be used. The copper gauze detectors were monitored during these operations, and no evidence of a sodium leak was found. With helium in the fuel system the temperatures in the fuel and sodium systems were then gradually raised (about 10°F/hr) to 1300°F. After the 1300°F temperature was reached, the helium pressure was reduced to 0.5 psig and krypton was added to bring the system pressure to 5 psig. After 30 min, the helium in the annulus system was checked repeatedly, and no evidence of a leak was found.

The fuel carrier ($NaZrF_5$) was then loaded in the fill tank at 1200°F, and the fuel system was evacuated to raise the fluoride into the system. It was possible to follow the progress of the fluoride through the system by watching the thermocouple indications because the system was initially at 1300°F and the fluoride was at 1200°F. There is assurance that no gas is entrained in the fuel system because no liquid level change occurs when the pressure over the fuel pump is changed, and the temperature differentials across the six parallel fuel passages in the reactor change simultaneously when a temperature perturbation is put into the system. All the pumps are working satisfactorily.

Fluoride samples were taken after 6 hr of circulation in the fuel system and after 20 hr to determine the corrosion products as a measure of the cleanliness of the fluoride system. Analyses showed the system to be clean, and therefore it will not be necessary to change from the original fill to a new batch of carrier. Enrichment of the fuel carrier will be started after 75 to 100 hr of satisfactory operation of the system. (AEC Activity 4401.1)

Circulating-Fuel Reactor Experiment (CFRE) - Multigroup calculations and critical experiments are sufficiently far along to show that a 21-in.-dia. core represents a good design for the CFRE, based on fuel concentration, shield weight, and hydrodynamic considerations.

Experience with the core-flow test rig indicated that quite a number of important improvements and refinements should be made. A new core profile has been evolved. It differs from that used to date in that the core diameter is 21 in. instead of 18 in. and the contour is modified somewhat to give a more favorable static pressure gradient along the walls. An improved pump-volute and core-inlet region was designed and is being built. A better technique for fabricating core shells was developed and will be used for the new set of shells.

The new model of the central swirl chamber type of expansion tank and pump system was completed and tested. It primes nicely and functions well over a wide range of liquid levels and pump speeds. (AEC Activity 4401.1)

ANP Experimental Engineering--Basic Developments - A heat exchanger header experimental assembly for observing the effects of small, controlled leaks of $NaF-ZrF_4-UF_4$ into NaK was operated twice. The tests indicated that small leaks are self-healing.

The large-scale loop for testing ARE components was disassembled and examined following 2000 hr of operation. The metallographic results, though incomplete, indicate that the lifetime of the Inconel in the ARE should be more than adequate. The loop has been reassembled, and it is to be tested for an additional 1000 hr at 1350°F with the gas-seal pump operating at 1500 rpm to produce 40-gpm flow (ARE design conditions). An anti-plugging device installed on the gas nozzle of the pump operated satisfactorily, and therefore a duplicate was installed on the ARE fuel pump. A modification of the pressure-measuring devices that allows pressure readings at subatmospheric pressures was developed, tested, and installed.

Because of leaks through the welds in the Inconel tubing, no large-scale, high-turbulence, high-temperature-difference, molten-fluoride test units have operated at design conditions for more than 24 consecutive hours. A concerted study of this problem revealed that difficulties with weld design and welding technique were probably the causes of the leaks, and work is under way to overcome these difficulties.

A third sodium-beryllium-Inconel mass transfer test unit is operating with high sodium turbulence and a large temperature difference (1300°F maximum, 1000°F minimum sodium temperature and minimum Reynolds Number greater than 100,000). Over 600 hr of the intended total of 1000 hr of operation has been completed. The results will add to information obtained from the two previous tests (one at 1200°F maximum temperature for 1000 hr and one at 1300°F maximum for 468 hr).

Vertical-shaft sump-type centrifugal pumps for pumping molten fluorides and liquid metals at temperatures up to 1500°F have been developed to the point of semi-standardization and are being made in the Y-12 shops as needed. Maximum performance tests of these pumps at 3600 rpm give flow rates of 1 to 6 gpm at heads of 100 to 85 ft of fluid, without gassing.

Natural gas burners have been fabricated and operated with 400-kw heat release in less than 0.5 ft^3. These burners were developed as an alternate to electric power for providing a concentrated heat source. (AEC Activity 4401.1)

ANP Experimental Engineering--In-Pile Components - A horizontal shaft sump pump was operated 1000 hr at 1400°F (maximum) with turbulent flow of the pumped fluid ($NaF-ZrF_4$) and a temperature difference of 100°F in the system. Sections of the system are being examined for corrosion. Another similar unit was operated 500 hr at 1400°F (isothermal). Air motor drives were used for both these pumps. Excessive carbon deposits (from oil leakage) were found in the hot zones of both pumps. One pump showed considerable shaft wear from the frozen fluoride salt mixture.

Sufficient data were obtained from water tests of a plastic model of the proposed regenerative turbine-type pump for in-pile use to make it possible to freeze the design. Studies were made of gassing and degassing, impeller clearance, internal labyrinth leakage, filling, priming, and performance (head vs flow). (AEC Activity 4401.1)

ANP Reactor Chemistry - The ARE fuel solvent was successfully transferred to ARE fill tank No. 2. Eleven batches (totalling nearly 2750 lb) of the $NaZrF_5$ mixture were required to fill the tank to the level which activated the highest electrical contact. Transfer of these eleven batches of molten material at 1300°F by application of helium pressure required approximately 42 hr. The operation was adjudged completely satisfactory.

A portion (nearly one-half) of the material was transferred from the tank to the ARE fuel circuit and is being circulated at about 30 gpm and a temperature of about 1250°F. Three samples were successfully withdrawn from the circulating fluid for analysis. Analytical data show the circulating liquid to contain about 85 ppm of Cr, about 15 ppm of Ni, and less than 10 ppm of Fe. It appears that cleanliness of the system, purity of the fluid, and adequacy of the handling techniques has been demonstrated beyond reasonable doubt.

Further evidence of the tendency for UF_3 to disproportionate in alkali fluoride mixtures was derived from an experiment in which excess UF_3 was equilibrated for 2-3/4 hr at 800°C in NaF-KF-LiF eutectic; the filtrate from this mixture contained 23.2 wt % uranium, but only 16.9 wt % U^{3+}.

The presence of UF_4 in melts which are subjected to electrolysis between a carbon anode and a nickel cathode gives rise to a relatively inefficient process wherein the reduction of U^{4+} to U^{3+} at the cathode is counteracted by the oxidation of U^{3+} to U^{4+} at the anode. Although electrolysis is of great value in reducing structural metal ions at the cathode, reduction of UF_4 to UF_3 in fair quantity will require a different cell geometry.

The high solubility of CrF_3 in the NaF-LiF-KF mixture indicates that the cold-leg films of alkali fluochromate previously observed in heat transfer and other experiments should be moderately easy to avoid. Equilibration of CrF_3 with NaF-LiF-KF (11.5-46.5-42.0 mole %) for 5 hr at 600°C indicates a solubility of 2.0 wt % CrF_3. The solid phase in equilibrium with the melt is a complex fluochromate. Disproportionation of CrF_2 in this alkali-fluoride mixture certainly occurs at 600° and at 800°C; additional data are needed before an equilibrium constant for the reaction

$$3CrF_2 \rightleftharpoons 2CrF_3 + Cr$$

can be evaluated. (AEC Activity 4401.5)

ANP Critical Experiments - Measurements of neutron flux and fission-rate distributions within the first of the current series of critical assemblies of the reflector-moderated circulating-fuel reactors were completed. The second array differs significantly from the first in that it consists of an essentially spherical central beryllium island surrounded by a fuel shell and a beryllium reflector. The island is about 10 in. in diameter and the fuel annulus is approximately 4.5 in. thick; the latter has a volume of 50.4 liters. The fuel, as in the preceding experiments, consists of laminae of Teflon, $(CF_2)_n$, and 93.2% enriched U^{235} metal sheets 0.004 in. thick. The reflector is effectively infinite.

The assembly was critical when the fuel region was filled to about 80% of capacity with 5.9 kg of U^{235} at a density of 0.147 g of U^{235} per cubic centimeter. Completing the loading to the design value determined by multigroup calculations would have resulted in an estimated excess reactivity of 7.5%. (AEC Activity 4401.2)

ANP Radiation Damage - Three capsules containing the fuel mixture $NaF-ZrF_4-UF_4$ (50-46-4 mole %), which were irradiated for 300 hr in the MTR at 2500 watts/cm^3 and at an operating temperature of 1620°F, exhibited corrosion that agreed with the results of previous tests (2 to 5 mils depth of attack). Additional capsules containing UF_3-bearing fuel, which in the past has shown no corrosion under irradiation, are being examined.

A radioassay method was developed for determining iron contamination during sampling of the fluoride fuel from capsules irradiated in the MTR. Corrected iron concentrations in the fuel were low and therefore consistent with the slight corrosion previously observed metallographically. Chemical analyses showed higher iron concentrations because of iron contamination from the drills used to obtain the samples. (AEC Activity 4401.4)

ANP Corrosion Research - Sufficient evidence has been accumulated from static and thermal-convection-loop tests to establish that diffusion of chromium is not the rate-controlling process in the corrosion of Inconel by fluoride mixtures. The thermal-convection loop results which bear out this conclusion are that (1) attack is centered in one place in the hot leg, (2) the diffusion gradient steepens after the original impurity attack has occurred so that during the last transverse step the diffusion of chromium cannot possibly be the rate-determining factor, (3) there is little effect of temperature on diffusion in the range 1300 to 1650°F, and (4) in experiments with mixtures with greater amounts of impurities, there was increased corrosion.

Molybdenum, which appears to have excellent resistance to fluoride mixtures in static tests, was finally tested in a thermal-convection loop constructed from type 310 stainless-steel-clad molybdenum. The loop, which circulated a mixture of $NaF-ZrF_4-UF_4$ (50-46-4 mole %) at a hot-leg temperature of 1500°F, failed after 250 hr of operation because of a failure in a molybdenum weld which caused a weld failure in the steel cladding.

Hastelloy B loops have now been operated for 500 hr and for 1000 hr at hot-leg temperatures of 1500°F with $NaF-ZrF_4-UF_4$ (50-46-4 mole %). The attack of approximately 0.5 to 1.5 mils found after 500 hr had not increased after 1000 hr, in contrast to the 5 to 9 mils of attack on Inconel in 500 hr and the 9 to 13 mils of attack on Inconel in 1000 hr. After operation of a Hastelloy B loop at 1650°F for 500 hr with $NaF-ZrF_4-UF_4$ (50-46-4 mole %), attack to a depth of 2 mils was found, and, also, there was a thin deposit in the hot leg; the attack on Inconel would have been over 13 mils. (AEC Activity 4401.5)

ANP Metallurgy--Mechanical Properties - Hastelloy B is being studied for possible use as a reactor material. Despite its excellent corrosion resistance, Hastelloy B is not yet satisfactory for reactor use for several reasons. First, it has poor oxidation resistance in air. In a CFRE type of application, however, it would see air only at the radiators where it probably could be protected by vapor deposition of chromium, aluminum, or silicon. Second, the ductility of Hastelloy B in the temperature range of interest (1300 to 1650°F) is low, but it can be improved by reducing the molybdenum content from 27 to 20%. Third, seamless tubing cannot, at present, be obtained because the impurities brought into the alloy with the nickel make it hot-short. It is thought that it might be possible to produce seamless tubing with purer material. Fourth, weld cracking has been encountered, but this difficulty can be overcome by reducing the molybdenum content.

It is thought that the developmental studies will have provided sufficient information by the first of the year for a decision to be made as to whether modified Hastelloy B can be produced in the shapes, quantities, and qualities needed for reactor application. Since the most rigid test of the material is in the heat exchanger system, several heat exchangers are to be fabricated and tested. For these units, three materials will be used: commercial Hastelloy B, modified Hastelloy B, and another nickel-molybdenum alloy. (AEC Activity 4401.5)

ANP Heat Transfer Research - In the hydrodynamic studies of the flow in the core of the reflector-moderated reactor, the photographic technique for determining velocity profiles was tested, photographs of particles flowing through the core at a Reynolds number of 10,000 were obtained, and regions of flow separation were outlined for several Reynolds numbers (up to 40,000) for a special entrance section.

A heat transfer analysis is under way of fuel flow past a plate (or strut) where both hydrodynamic and thermal boundaries are being established. Regions of flow separation and flow in curved channels are also being studied. (AEC Activity 4401.5)

ANP Physical Properties Research - Recent measurements, of the enthalpy and heat capacity of NaF-KF-LiF (11.5-42.0-46.5 mole %), made at the National Bureau of Standards and the Naval Research Laboratory, by using an ice calorimeter, confirm work done at Oak Ridge National Laboratory about two years ago; their data deviated from the ORNL results by less than 6% over the temperature range 450° to 1000°C.

In a program of refining viscosity measurements, the error band was reduced from ± 30% to ± 10% because of improvements in handling techniques and in fuel purity. High-density calibrating fluids are being used in the calibration process.

The density of liquid rubidium was determined, and found to vary with temperature over the range 40° to 410°C according to the function $\rho(g/cm^3) = 1.52 - 0.00054(T-39)$, where T is the temperature in °C.

The thermal conductivities of several solid zirconium-bearing fluoride mixtures were measured. The values obtained for the solids were significantly lower than the corresponding liquid values. Electrical conductivities were measured for several fluoride mixtures and for the heat transfer salts NaOH and KNO_3. (AEC Activity 4401.5)

ANP Analytical Chemistry Studies - A new method for the determination of U(III) in the presence of U(IV) in proposed ARE fuels was developed. A solution of 0.02 N methylene blue that is 3 N with respect to HCl will oxidize U(III) to U(IV) with no evolution of hydrogen, a phenomenon that has been encountered with most oxidizing agents that have been tried for this

reaction. The excess oxidant is determined by titrating with Cr(II). The methylene blue reagent was tested successfully on various forms of U(III), including UF_3, $NaZrF_5$-UF_3, and NaF-KF-LiF-UF_3-UF_4 mixtures. Uranium trichloride appears to react too rapidly with acidic solutions of methylene blue and liberates some hydrogen.

A method for the determination of uranium metal in proposed ARE fuels was also developed. The sample of fluoride mixture is heated in an atmosphere of hydrogen at 250°C for 1 hr to convert the uranium to uranium hydride. After cooling, the sample is heated in an atmosphere of carbon dioxide at 400°C for 2 hr to decompose UH_3. The volume of hydrogen liberated is measured over a solution of potassium hydroxide as a function of the uranium metal present. Uranium trifluoride was found to reduce carbon dioxide to carbon monoxide at 400°C almost quantitatively, so a trap of iodine pentoxide and sulfuric acid was incorporated to reconvert CO to CO_2. The method is not applicable to small concentrations of uranium metal because of the limitations of measuring small volumes of hydrogen.

Tests were completed to prove the reliability of the isotopic dilution method of uranium analysis for ANP fuels. Tests of the analytical procedure without uranium present yielded a value of only 0.14 μg of uranium per analysis for samples of the order of 1/2 mg. Standard uranium mixtures varied 0.37% from theoretical on the average. When the method was applied to a fuel that had been homogenized by grinding, the uranium values were reproducible to within 7 parts per thousand. (AEC Activity 4401.5)

ANP Shielding Analysis - The problem of the energy absorption of incident gamma radiation on a slab of material as a function of the thickness of the material was programmed for the ORACLE. The Monte Carlo method was used, and initial studies indicate satisfactory results. The study was undertaken to aid in the calculation of heating in thermal shields. (AEC Activity 4401.7)

ANP Shielding--Lid Tank Shielding Facility (LTSF) - A value of 1.01 ± 0.10 barns/atom was obtained for the effective fast-neutron removal cross section of lithium from thermal-neutron measurements made in a medium of oil. A 27.2 cm thickness of lithium (encased in stainless steel) was placed against the source plate for the measurement.

Radiation decomposition in samples of a solution of UF_6 in C_7F_{16} was found to be too great for use of the liquid as the fuel in the LTSF mockups of a circulating-fuel reflector-moderated reactor, and it is now planned to use solid UO_2 in the form of MTR-type fuel plates. A continuous series of plates will be mounted on a belt which will be moved from the neutron window, where the UO_2 will be irradiated, to the heat exchanger region and back to the window. The belt will move at any of four speeds, the complete transit being made in a minimum of 1/2 sec and a maximum of 4 sec. The UO_2 plates will be tested for rigidity under operating conditions in a special rig outside the LTSF. (AEC Activity 4401.7)

ANP Shielding--Tower Shielding Facility (TSF) - In an Air Force project for determination of the immediate effects on behavior of massive neutron radiation doses, a group of 25 monkeys was irradiated at the TSF. The reactor was operated at full power (about 500 kw) for short periods at the edge of the large water tank, which was raised to just above ground level. The results of the exposures will be reported by the Air Force operations group. In addition to the irradiation, all dosimetry was carried out by ORNL. (AEC Activity 4401.7)

GENERAL REACTOR RESEARCH

General Reactor Radiation Damage - A shielded x-ray diffractometer for use in the examination of irradiated samples was placed in operation. The diffractometer unit is a modified General Electric type SPG spectrogoniometer in which the counting circuit has been replaced by a shielded proportional counter equipped with a discriminator circuit.

Samples irradiated with 1×10^{20} and 2×10^{20} neutrons/cm^2 and measuring up to 10.4 r/hr on contact have been subjected to x-ray diffraction measurements. Zircon maintains some degree of crystallinity after an irradiation of 1×10^{20} neutrons/cm^2, but loses its crystallinity after an irradiation of 2×10^{20} neutrons/cm^2, while a hafnium-free ZrO_2 undergoes changes but still maintains a high degree of crystallinity at the higher irradiation level. (AEC Activity 4540)

Dissolution Studies - Further column studies in the laboratory continuous dissolver (using caustic) showed that the uranium-bearing solid product contains variable amounts of UAl_3 and UAl_2 in addition to UO_2; in batch dissolvings the only product is UO_2.

Batch caustic dissolvings of both cast and extruded 5.37% uranium--94.63% aluminum alloy indicated that the rates of dissolution are the same; the specific reaction rate constant is about 9×10^{-5} liter/min/sq cm. Substitution of stainless steel for glass in the dissolving section of the laboratory column did not significantly affect the rate of slug dissolution.

In batch dissolutions of unirradiated slugs with fission products added to a level of $\sim 10^8$ β and 10^8 γ c/m/ml, the ratio of fission product activity on the UO_2 precipitate to that in the supernatant was: TRE (total rare earth) β, $1.8 \times 10^{+5}$/l; Sr β, 5.6×10^3/l; Nb β, 9.1×10^2/l; Zr β, >60/l; Ru β, 1.7/l; Cs β, 7.1×10^{-3}/l. It is apparent that a very good preliminary removal of fission products from the liquid waste was effected.

Only 0.01% of the fission-product iodine in 300-day-irradiated, 90-day-cooled uranium-aluminum alloy slugs can be tolerated in the dissolver off-gas. Results of further experiments confirmed preliminary indications (ORNL-1781) that the mercury catalyst in the acid dissolvent will not complex the iodine sufficiently to prevent its escape in the off-gas.

With a condenser on the dissolver to reflux the off-gas, and with simulated acid dissolver solutions containing one and four times the concentration expected in the U^{235} process, only 0.68 and 0.49%, respectively, of the iodine escaped. In one experiment without reflux, 50% of the iodine appeared in the off-gas. (AEC Activity 4581)

Sanitary Engineering and Development - Methods for the recovery of Cs^{137} and Nb^{95} from soils were developed. The best recoveries (of Cs^{137}) were obtained with 9M H_2SO_4 and of Nb^{95} with 0.55 M $H_2C_2O_4$ + 1 M HCl. The recovery of Ru^{106} was somewhat erratic and new methods are being studied.

The use of Tennessee ball clay as a liner material for earth pits was evaluated with permeability values in the range of 10^{-8} to 10^{-10} cm/sec, the values depending on the placement procedure employed.

The evaporation study employed to determine the water-tightness of the tamped clay-asphaltic membrane pit liter was completed and the data are being analyzed. Waste movement from the unlined experimental pit was detected by use of the auger holes which encircle the pit. Thus far it appears that a waste fringe diffusion-dilution effect is being observed in the augerholes, while the main body of the pollution stream has not been intercepted.

Explorations in the four-acre area mentioned in the Status and Progress report for June 1954 were containued. The four NX core holes 200 ft in depth were completed; analyses of the shale at each of the test wells are being made; permeameter tests (with water and with a typical waste solution) at various depths in the weathered shale are in progress; and a contract was let for a 300-feet cable-tool (percussion) test well to be completed during November. The purpose is to begin in December, 1954 the detailed testing of the borings in this area and the formulation of design criteria for a large pilot-scale pit for high-level reactor process wastes. (AEC Activity 4583)

PROGRAM 5000 - PHYSICAL RESEARCH

ISOTOPE PRODUCTION

Stable Isotope Production - Separation of calcium isotopes (masses 40, 42, 43, 44, 46, 48) which was started in August, still continues. Cyclotron targets of Ca^{46} and Ca^{48} will be prepared for the production of urgently needed radioactive isotopes, particularly Ca^{47}. The processing of Ca is performed in Alpha channels with the large two-arc ion source. The production rate of 125 milliamperes per hour of ion reception exceeds that obtained in previous calcium separations.

Collection of the isotopes of iron (masses 54, 56, 57, 58) is nearing completion. Approximately 1.3 grams of Fe^{58} have been collected and emphasis is now being placed upon collection of approximately one gram of extremely pure (99.99+%) Fe^{56}. The attainment of such purity requires that this be a "second pass" collection and the "charge" is being prepared from material enriched to 99.0% Fe^{56}, of which there are several kilograms in the inventory.

Stable isotope chemistry operations were all consolidated into the same building (9731) in which the isotopes are separated. New lots of enriched K^{39}, Cr^{54}, Ni^{61}, and Cu^{63} were added to the inventory. Cr^{54} was especially prepared for use as a cyclotron target.

In the program of separating rare earths by liquid-liquid extraction nearly 1 kilogram of Dy_2O_3 was enriched to the 85-95% level.

Seventy shipments were made consisting of 47 different isotopes of 25 elements. (AEC Activity 5121)

PHYSICS

Nuclear Measurements for Reactor Design - Thin, uniform samples of ThF_4, depleted UF_4 and Au were prepared on aluminum by vacuum evaporation, weighed, and the alpha activities of the U and Th samples determined as a check of the weights. Sets of samples were exposed together in the shield of the ORNL Graphite reactor to a beam from Hole 10, using gadolinium (0.2 ev) and cadmium (0.4 and 0.8 ev) filters. The induced beta activities were observed through a 2 mil aluminum alpha shield with a 2π beta proportional counter. The counter efficiency was determined from standards, ultimately derived from beta-gamma coincidence counting.

Using the most recent values for the gold resonances and correcting for thermal neutrons transmitted by the gadolinium filter, the values 280 b (0.2 ev), 275 b (0.4 ev), and 283 b (0.8 ev) are found for uranium, and 77.5 b (0.2 ev), 69.5 b (0.4 ev) and 66.1 b (0.8 ev) for thorium for the pile beam spectrum. Subtracting the 1/v components in the usual manner gives average values of 278 b and 67.3 b for the uranium and thorium integrals. These last two values compare favorably with recent measurements from velocity selector data: on uranium at Argonne National Laboratory (290 b) and Brookhaven National Laboratory (260 b); adding the integrals of the seven thorium resonances reported from Brookhaven gives 64 b. The uncertainty in the final values is estimated at $\pm$ 7% largely due to uncertainty in the resonance integral of gold. The ratio of thorium to uranium activations is probably better known. (AEC Activity 5200)

New Instrument for Medical and Biological Assay of Gamma Emitters - Scintillation counting for the determination of radio-iodine and other gamma ray emitting substances for biological work has always been extremely difficult

with equipment commercially available because of the inclusion of scattered radiation in the measurements. This scattered radiation can easily lead to an error of a factor of two and makes precision dosimetry very difficult. A one chassis unit has been built which consists of a complete linear amplifier of the non-overload variety, a single channel analyzer, and a count integrator together with a pre-set timer and the necessary high voltage supply for the photomultiplier. This unit will provide accurate measurements of the undegraded primary radiation from gamma ray emitters in biological samples and should considerably improve medical experiments. The count integrator may also be used as a count-rate-meter although the loss of speed or accuracy which is present in a count-rate-meter is undesirable for medical applications. (AEC Activity 5200)

Scintillation Spectrometry - Efficiency measurements which have been proceeding on NaI crystals have at last yielded satisfactory agreement between the "calculated efficiency-peak to total" measurements of the peak efficiency and "direct beta-gamma coincidence" measurements of the peak efficiency. The principal cause of disagreement has been due to the considerable back scattering from even a very large lead shield (inside dimensions of 2 ft. by 2 ft. by 2 ft.). With the present efficiency measurements, absolute gamma ray intensities of 3% to 5% accuracy can be obtained. (AEC Activity 5200)

High Voltage Program - The excitation of the 0.8-sec metastable state associated with fast neutron irradiation of Zr^{90} in the pile was measured for neutron energies up to 4.0 Mev by use of the 5.5 Mev Van de Graaff generator. The T(p,n) reaction was used to produce monoenergetic neutrons. The induced γ-ray activity was detected by an anthracene scintillation counter. An automatic tuning sequence was used to pulse the neutron source and to turn on and off the scintillation counter. The threshold for excitation was found to occur at 2.3 Mev. Since 2.3 Mev is the excitation energy of the γ-ray given off in the decay of the metastable state this is strong evidence that the state is being excited by inelastic neutron scattering rather than by an (n,p) or (n,α) reaction. The excitation cross section rises rapidly above threshold and continues to increase up to the highest neutron energy. The absolute cross section has not yet been determined.

The results of some fission cross section measurements are summarized as follows:

Neutrons from the V(p,n) reaction were used to measure the fission cross section ratio of U^{233} to U^{235} at energies from 10 to 400 kv. These measurements overlap and are in agreement with previous Oak Ridge results, which extended down to 320 kv, with T(p,n) neutrons, and with those which extended down to 140 kv, using Li(p,n) neutrons. At 30 kv, the measured ratio is 1.40 $\pm$ 0.05 in contrast to 1.14 as listed in report BNL-170, based on some early work reported in LA-188. Consequently, these results are widely different from BNL-170 in the energy decade 10 to 100 kv.

A further refinement was made in the measurement of the fission cross section ratio of U^{238} to U^{235}, by establishing the correction for neutrons scattered inelastically by the fission chamber walls. For neutrons of 2.8 Mev energy this correction was estimated to be 2.8%, based on assumption of inelastic scattering cross sections at this energy of 2.2, 1.1, and 0.65 barns for Cd, brass, and Al, respectively. The calculation is rather insensitive to the kind of energy spectrum assumed for the inelastically scattered neutrons. The difference in the result is only 10% for a Maxwellian distribution having an average temperature of 330 kv, contrasted to a distribution taken as constant from 10 kv to 2.8 Mev, and zero below 10 kv.

This correction was verified experimentally by adding mass to the counter such that the ratio of scattering effect with and without the added mass is accurately known. Six points, having statistics reliable to 1/2%, were taken between 2 and 4 Mev, with and without the added mass. The results established the correction to be 2.8 $\pm$ 0.4% at 2.8 Mev, and to be nearly constant over the energy region 2 to 4 Mev.

The final cross section curve obtained for U^{238} is similar in slope with energy to that in AECU-2040, but about 9% higher. It is higher than the most recent Los Alamos results (LA-1495) above 2.5 Mev. Although the curve in LA-1495 cannot be read closely enough to permit a detailed comparison, at about 2.5 Mev the results seem to be in agreement. (AEC Activity 5200)

The 86-Inch Cyclotron, Applied Physics - A new record beam current of 3.8 milliamperes at 18 Mev was established on the 86-Inch Cyclotron and this result was confirmed calorimetrically. It was also possible to achieve extended periods of operation above 3 ma at the same energy. The energy reduction from the usual 22 Mev to 18 Mev was accomplished by introducing a first-harmonic inhomogeneity in the magnetic field, using half-coils on the magnet poles. The high beam current markedly increases the isotope production rates of radioisotopes from (p,n) reactions, for which the thick-target yield is nearly independent of energy in the region near 20 Mev. Average currents obtained recently on several targets were as follows: silver for Cd^{109}, 29 hours at 1950 μa; copper for Zn^{65}, 5 hours at 1900 μa; chromium for Mn^{54}, 55 hours at 1900 μa; and calutron-separated Cr^{54}, 11 hours at 2000 μa. (AEC Activity 5200)

The 86-Inch Cyclotron, Operation - The 86" cyclotron was operated on a three-shift, five-day basis during this period in order to meet commitments to other divisions before shutting the machine down. Several of the runs exceeded 25 hours with an average proton beam of approximately two milliamperes at 18 Mev.

Magnetic deflector channel tests were completed insofar as is possible without actual installation in the cyclotron. A test model of the septum, which is composed of 0.062 in. O.D. water-cooled aluminum tubes, successfully withstood power dissipation tests in a 50-kw beam, that is, 2.5 ma of protons at

20 Mev. On October 22, the machine was shut down and the dees and liner were removed in preparation for the installation of a beam deflector system. The shutdown is expected to last about four to six months. (AEC Activity 5200)

The 63-Inch Cyclotron - Carbon-11 (20.5 min) and nitrogen-13 (10.1 min) were identified chemically as reaction products from the nitrogen bombardment of aluminum. A double-focusing analyzing magnet for the detection and identification of heavy reaction products was designed.

A proportional counter-scintillation counter telescope was constructed for the identification of light reaction products from nitrogen-induced reactions. This instrument makes it possible to identify and measure the energy of protons, deuterons, and alpha particles without confusion. It makes use of the property that, for light particles in the energy range encountered, $E(dE/dx)$ is nearly constant. Preliminary results on the angular distribution of alpha particles from the reaction $N + C \longrightarrow F^{18} + 2\alpha$ were obtained. (AEC Activity 5200)

Spectroscopy Research Laboratory - The complete Raman vibrational spectrum of HCOF was observed allowing assignment of the weak CH out of plane bending vibration to 1244 wave numbers. Fabrication of a temperature-controlled absorption cell for infrared studies of H, D, and T in water is almost completed. Investigation of the accuracy of absolute wavelengths directly computed from echellograms measured with a specially designed coordinate comparator indicates probable errors of about 0.005 A; introduction of the correction for the cosine variation of the dispersion reduces this to about 0.002 A. Extensions to the molecular energy levels of polonium, Po_2, were made during this period. Cooperative work among the University of Tennessee, ORNL Health Physics Division, and this laboratory was begun on the spectrochemistry of important radioactive reactor products potentially assimilated by humans. Stark effect studies on hydrogen in the hollow cathode discharge reveal potential field gradients of about 3500 volts per centimeter; precision of measurement of field gradients is about 100 volts per centimeter. A pressure scanning Fabry-Perot interferometer was tested; spectrum resolution exceeds 500,000 but difficulties were experienced with the recording manometer. X-ray crystal structure data were tabulated for six more compounds ($2Na_2CO_3 \cdot UO_2CO_3$, $UO_2(HCDO)_2 \cdot H_2O$, $2K_2CO_3 \cdot UO_2CO_3$, $UO_2H\ AsO_4$, $NaUO_2\ ASO_4 \cdot 3H_2O$, and $2UO_2SO_4 \cdot 7H_2O$) bringing to 35 the number of new compounds studied. Significant corrections to ASTM data due to line extinctions in NiF_2 were observed. (AEC Activity 5200)

Mass Spectrometer Laboratory - A sample of approximately 4-1/2 micrograms of Tc^{99} in solution, plus any Tc^{98} residing in 1 kilogram of molybdenite ore, was supplied by G. E. Boyd for investigating the Tc^{98} content. No Tc^{98} was found and an upper limit was set at 10^{-12} grams per gram of molybdenite.

The analysis of sea water for uranium has encompassed the analysis of five standards and one determination on water from the Gulf of Mexico. This analysis gave 3.5×10^{-9} grams of uranium per gram of sea water.

A method of leak hunting in the ARE fuel system was developed in which a mass spectrometer is used to detect possible traces of krypton in an atmosphere of helium.

The General Electric Mass Spectrometer was modified so that one part of krypton in 10^6 parts of helium could be detected. Two resistors are employed in the ion current detecting circuit; the current due to the helium ions develop a potential across a 10^8 ohm resistor and the current due to the krypton ions develop a potential across a 10^{11} ohm resistor. Approximately one hundred mixtures of helium and krypton were examined in calibrating the mass spectrometer.

Twenty-nine samples were analyzed for the Stable Isotope Division and 65 gas samples were analyzed for other divisions. (AEC Activity 5200)

Research and Development, Stable Isotopes - Special preparations of targets and target materials included: (1) electrodeposition of Cr^{54} metal on a silver cyclotron target for Mn^{54} production (in cooperation with the Isotope Chemistry Department); (2) preparation of $Te^{125}O_2$ and $Te^{130}O_2$ from elemental Te for cyclotron bombardments for I^{125} and I^{130} production; (3) coating of tantalum sheet with normal elemental boron for evaluation of coating thickness and uniformity; (4) reduction of $Si^{28}O_2$ to elemental silicon for purification by zone melting. A metal spray gun is being adapted to the preparation of various types of isotope-coated targets.

A newly developed fractional crystallization recovery procedure for separation of $BaCl_2$ from LiCl, after the thermochemical preparation of metallic Li^7, eliminates previous losses of isotopic material through coprecipitation with $BaSO_4$. (AEC Activity 5200)

CHEMISTRY

Inorganic Solution Chemistry - Earlier work on the anion exchange behavior of Mo(VI), W(VI) and U(VI) in HCl solutions was expanded to HCl-HF mixtures and conditions were found for successful separation of these elements. Separation of small amounts of these elements from each other can be carried out with ca. 5 cm. columns by adsorbing all three elements from ca. 9 M HCl, eluting U(VI) with 0.5 M HCl - 1 M HF, then eluting W(VI) with 9 M HCl - 1 M HF and Mo(VI) with 1 M HCl. While the separation of Fe(III) from these elements is difficult in HCl media alone, satisfactory separations can be achieved with HCl-HF mixtures.

Separations involving Ge(IV), As(III) and As(V) were investigated. Since As(V) shows only relatively little adsorption throughout the whole HCl range

studied (up to 12 M HCl) and since Ge(IV) shows rapidly increasing adsorption above ca. 6 M HCl, these two elements can readily be separated from each other at high HCl concentrations. The separation of As(III) and Ge(IV) is not so simple, since these ions adsorb similarly at those HCl concentrations where elution could be effected.

Comparison of the adsorbability of a number of elements from HCl and LiCl solutions indicated that in many cases adsorption from LiCl solutions is considerably stronger than from HCl solutions. Distribution coefficients of Mn(II), Co(II), Zn(II), Au(III), Sc(III) and Ga(III) were found to be about a factor of 100 larger in 12 M LiCl - 0.1 M HCl than in 12 M HCl. This certain anion exchange separations which were only marginal in HCl media (e.g., separations involving Mn(II) and Sc(III) appear more feasible in LiCl solutions.

Ultracentrifugations of Th(IV) in 1 M $NaClO_4$ solutions were carried out as a function of pH and hydroxyl number n (average number of hydroxide ions per thorium ion). The following preliminary degrees of polymerization N were found for .015 M Th(IV) solutions: At n = 1.0, N = 2.5; at n = 1.5, N = 3; at n = 1.95, N = 4.5; and at n = 2.4, N = 9. In contrast to the earlier results with Bi(III), Zr(IV) and Hf(IV), there appears to be no region in which a single monodisperse low molecular weight polymer predominates. Studies are now under way with Th(IV) in chloride solutions, and the preliminary results indicate that in this medium considerably higher degrees of polymerization are found than in the corresponding media (i.e. for a given value of n). (AEC Activity 5300)

Chemical Physics - The activation energy for the reaction between K and HBr was measured and found to be 3.4 ± 0.1 kcal. This value was obtained from four experiments at varying temperatures of K and of HBr, and the data for the different runs taken in pairs agree within the uncertainty indicated. The distribution function for energies of collision for this case (molecular beams of the reactants colliding at right angles) does not allow a simple graphical method such as the Arrhenius equation allows in ordinary kinetics.

The distribution in angle of the product KBr was similar in all experiments. The peak of the experimental distribution curve lies nearer to the potassium direction than would be expected on the simplest assumptions about the reactive collisions, but detailed conclusions await further calculations.

Paramagnetic resonance studies show that each of the atomic hydrogen hyperfine lines in samples of H_2SO_4, H_3PO_4 and $HClO_4$ irradiated at 77°K with gamma rays has a pair of weak satellite lines. Measurements at nominal fields of 8000 and 3000 gauss show the satellite spacing to be field dependent and to correspond to the flipping of environmental proton spins concurrent with the flipping of the electron spin. Theoretical work on transition probabilities is giving information on the distance of the environmental protons to the weakly coupled atomic hydrogen. (AEC Activity 5300)

Radiation Chemistry - Nitrate ion enhances the rate of reduction by cobalt gamma-radiation of ceric ion in 0.8N sulfuric acid solutions. The amount of enhancement is directly proportional to the concentration of nitrate ion. This enhancement is interpreted as resulting from the decomposition of nitrate ion to nitrite ion and oxygen, followed by the reduction of ceric ion by nitrite ion. The decomposition of nitrate ion occurs as a direct result of energy absorption by the nitrate ion and is similar to the photochemical reduction of nitrate ion.

In the ceric ion sensitized photo-oxidation of water, the OH radical is a postulated intermediate. Evidence supporting this postulate was obtained through a study of the influence of added solutes such as Br^-, Cl^-, Ag^+, H_2, and HCOOH which are known to react with the OH radical. A comparative study is being made of the photoreduction and gamma-ray induced reduction of ceric ion.

In the gamma ray decomposition of anhydrous nitrate crystals, oxygen and nitrite ion are produced in equivalent amounts. At 25°C, the initial yields of NO_2^-, in molecules per 100 ev, were $LiNO_3$ = 0.02, $NaNO_3$ = 0.25, KNO_3 = 1.57, $CsNO_3$ = 1.76, $AgNO_3$ = 0.2, $Ba(NO_3)_2$ = 2.0, $Pb(NO_3)_2$ = 0.6, $La(NO_3)_3$ = 1.6 and $La(NO_3)_3 \cdot 6H_2O$ = 2.5. These results indicate that the radiation stability of the nitrates depends primarily upon the field strength of the cation to which the nitrate ion is bound.

A gas analysis system adaptable to the analysis of various reaction mixtures irradiated by radon was designed and fabricated. This system can handle several cc. of multi-component mixtures at S.T.P. Gas is transferred either by Toepler pump or by a column of mercury preventing preferential diffusion. Several analyses of known synthetic mixtures agree within 1%.

The CO + Xe reaction mixture was analyzed. The ratio of the decrease in pressure of the CO to the increase in pressure of the CO_2 is about three, in agreement with the results of Lind and Bardwell who reported the alpha-ray decomposition of pure CO. As previously reported the Xe does not affect the velocity of decomposition of CO even though Xe has a lower ionization potential than CO. (AEC Activity 5300)

Chemistry of Fused Salts - The heat capacities of uranium trifluoride, UF_3, and molybdenum disulfide, MoS_2, were measured from 15° to 300°K. The heat capacities at 25°C of UF_3 and MoS_2 are 22.83 cal./mole deg. and 15.64 cal./mole deg. respectively. Calculation of the related thermodynamic quantities is not completed. The results show conclusively that the heat capacity of MoS_2 follows at T^2 dependence between 15° and 70°K. This is of theoretical importance in connection with work described in previous reports concerning this behavior in other substances.

Electrical conductivity and density of molten sodium bromide were measured in the temperature range 750 to 950°C. The density may be expressed by the relation $\rho = 2.9518 - 0.8169 \times 10^{-3}\ T$, where ρ is in g/cc. and T in °C,

and the standard deviation, σ, is 0.0003. Specific conductance is given by the expression $\kappa = -0.4392 + 5.6318 \times 10^{-3}\ T - 1.5721 \times 10^{-6}\ T^2$, where κ is in mhos/cm. and σ is 0.0024.

Comparison of the measured equivalent conductances of molten $NaNO_3$ and TlCl with those calculated from self-diffusion coefficients of Na^+ in molten $NaNO_3$ and Tl^+ in molten TlCl indicate that about 95% of the current is carried by the cations in these salts, thereby affording support for suggestions advanced previously. (AEC Activity 5300)

Uranium Chemistry of Raw Materials - A small pilot plant for the testing of liquid extraction of uranium from acid leach liquors was built and typical results show a uranium recovery of 99+ percent using Armeen 2-12 in Amsco G as the extractant. The iron content of the uranium product was low (iron was the only extractable contaminant in the synthetic feed), phase separation was good, entrainment in either phase was negligible, and there was no indication of emulsion. A flowsheet has been briefly investigated in the laboratory which appears feasible for recovery of both uranium and vanadium. The uranium and vanadium(V) are extracted simultaneously into the organic phase and then separated by selective stripping. Acidic chloride solutions strip the uranium effectively after which the vanadium can be stripped with sodium carbonate or sodium hydroxide solution.

As reported previously, the basic stripping of uranium from kerosene solutions of dialkylphosphoric acids is accomplished without third phase formation when the kerosene is modified with a small amount of certain alcohols.

Of the alcohols tested in the range of 8 to 13 carbon atoms, primary alcohols and secondary alcohols with the hydroxyl near the end of the chain are more effective than symmetrical secondary alcohols. The amount of alcohol required varies with the amount of reagent, (wt % ROH) = 0.6 + 3.5 ($\underline{M}$ R_2HPO_4) for di(2-ethylhexyl)phosphoric acid and 2-ethylhexanol at 30°C.

Erratum: ORNL-1781, p. 29, in the reported $[U^{+4}]$ solubility in equilibrium with $U(HPO_4)_2 \cdot 2H_2O$, for $.5 \times 10^{-4}$ read 5.5×10^{-4}. (AEC Activity 5300)

Thorex Process Development - Construction of the Thorex pilot plant is 100% complete. The columns are now being operated with solvent, and the first formal nonradioactive runs are planned for the first week in November. (AEC Activity 5300)

Analytical Research - A modification of Lingane's method of coulometrically titrating with electrolytically generated silver ion, was applied to the determination of microgram amounts (4 to 400 μg) of chloride in HRT fuel solutions. The effect of chromium was studied and a slight change in the cut-off potential was found necessary to eliminate chromium interference. This method permits remote automatic operation and will be useful for analyzing highly-radioactive solutions for chloride.

Neutron activation analysis was applied to the particle size distribution of ThO_2 in water slurries. An externally mounted scintillation counter (NaI, crystal thallium activated) was used to observe the sedimentation rate of the neutron-irradiated ThO_2 particles, after an air sparging, by measuring the Pa^{233} (27.4d) daughter radioactivity of Th^{233} (23.3m). Since the rate of sedimentation follows Stoke's Law, the counting rate observed at definite time intervals represents the percent concentration of a given grain size.

A procedure for the determination of rhenium in molybdenum ores and in flue dust was developed. Rhenium is separated from the bulk of the material by use of Dowex 2 resin which adsorbs the rhenium from a 1 N NaOH solution. The rhenium was eluted from the column with 2 N $HClO_4$ and estimated polarographically. The results of the analyses showed rhenium to be present in various ores in concentrations from 3 to 7000 ppm.

METALLURGY

Ceramics Research - A technique was developed for fabricating tubing from a mixture of BeO and UO_2. The tubing, which was requested by GE-Lockland for use in guided missile research, will contain 16 wt % UO_2 and will be 0.250 in. OD, 0.170 in. ID, and 4 in. long.

Thorium oxide rods, fabricated by isostatic pressing and sintering Ames ThO_2 powder with and without the addition of 1/2 wt % CaO, and canned in aluminum, were irradiated in the LITR. The specimens containing CaO (density $\sim$96% theoretical) and the one without CaO (density $\sim$87% theoretical) were subjected to exposures of 2.4×10^{20} nvt and 1.7×10^{20} nvt, respectively. No gross damage in either case was observed on the ends of the specimens where the can had been removed.

A SiC-Si body suitable for service in a proposed ceramic reactor was developed by impregnating a shape prepared by baking a mixture of 68% Dixon purified graphite (10 micron average particle size) and 38% glucose. Further work is in progress for introducing fuel into the body.

Specific heat determinations in the range 0-1000°C have been made on HfO_2 and SiC-Si by using the precision ice calorimeter; the value for HfO_2 in the range is 0.197 cal/g and for SiC-Si, 0.243 cal/g.

In the waste disposal investigations it was found that porous fired-clay slugs are capable of removing through adsorption and collective adsorption a considerable number of radioactive particles and ions from waste solutions. Geologic logging of 4 cored holes, 200 ft deep, put down in the X-10 area to explore the subsurface in a proposed site for field testing waste disposal methods indicated that below 60 ft the formations are relatively free from porosity. Investigation of natural barriers for sealing pits for containing liquid wastes indicated that ball clay from Paris, Tennessee, was a suitable sealing material. (AEC Activity 5400)

Physical Metallurgy of Reactor Materials - Construction and installation of an automatic feed mechanism for arc-melting zirconium-base alloys was completed. Test melts are to be made as soon as feed rods are available.

The tempering of martensite in a hypoeutectoid zirconium-silver alloy was studied. The process of tempering at 800°C may be described as follows: (1) the precipitation of Zr_2Ag with an immediate decrease in hardness, (2) the growth and spheroidization of the precipitate, (3) nucleation and growth of strain-free alpha grains. The final structure consists of fairly large spheroidized particles of Zr_2Ag in a matrix of strain-free alpha grains.

As a proposed treatment preliminary to chemical processing, zirconium-base fuel elements have been converted to a mass of powdery oxide by heating in the temperature range 850° to 950°C. Methods for leaching the uranium from the oxides are being investigated by the Chemical Technology Division. The aim is to produce a powder from which the uranium can be removed by leaching, thus leaving an unreactive powder of zirconium oxide for disposal.

The reduction of uranium tetrafluoride mixed with other fluorides by melting in contact with molten aluminum to produce aluminum-uranium alloys is being investigated. When properly carried out, well over 90% of the uranium is reduced from the fluoride phase and enters the metal phase. The influence of temperature and composition of the fluoride phase on recovery in the metal phase is being studied, and developmental work is under way on the method for carrying out the reduction. (AEC Activity 5400)

Fundamental Physico-Metallurgical Research - The investigation of the zirconium-silver phase diagram was continued with zirconium containing 0.01 instead of 0.03% iron. It was found that the maximum solid solubility in the α phase falls between 0.3 and 0.7 at. % silver for the lower iron content alloys, whereas the higher iron content alloys dissolve less than 0.14 at. %. A more detailed investigation of the eutectoid reaction $\beta \rightleftarrows \alpha + \gamma(Zr_2Ag)$ showed that the reaction occurs at 823 $\pm$ 1.5°C with a β-phase composition of 3.75 at. % silver. Even the alloys with 0.01 at. % iron, however, have a three-phase ($\alpha + \beta + \gamma$) region 12°C below the eutectoid temperatures; thus, it is clear that the Zr-Ag system is especially sensitive to impurities in this region.

An investigation of the temperature of the peritectic reaction β + liquid $\rightleftarrows$ $\gamma(Zr_2Ag)$ with an alloy of 30.5 at. % silver showed a peritectic temperature of 1175 $\pm$ 10°C. This temperature was confirmed by experiments with an alloy of 19.5 at. % silver in which the $\gamma(Zr_2Ag)$ crystals were observed to melt at 1175 $\pm$ 5°C.

The fiber axis distributions were determined for thorium rods extruded at 850°C at speeds of 1 fpm and 600 fpm. The most preferred crystallographic directions of the fiber axis in the rod extruded at the slower rate were $\langle 111 \rangle$ and $\langle 001 \rangle$; the ratio of the volume fraction $\langle 111 \rangle / \langle 001 \rangle$ was 13.2. The microstructure of this specimen showed no signs of recrystalliza-

tion. The most preferred direction of the fiber axis in the rod extruded at the faster rate--completely recrystallized--was near <114>. This behavior is similar to that of aluminum which forms a strong <111> and a weak <001> texture when extruded so as to minimize the degree of recrystallization. As the degree of recrystallization increases, the ratio of the volume fraction <111> /<001> decreases, giving a strong <001> and a weak <111> texture with large amounts of recrystallization. (AEC Activity 5400)

Fundamental Investigations of Radiation Damage - There is convincing evidence that low-temperature fast-neutron bombardment introduces "hole traps" into n-type germanium. A germanium sample was irradiated to an nvt_f of 2.4×10^{16} at a temperature of 114°K and then warmed in the γ field to 260°K at a uniform rate and recooled. The conductivity vs temperature curve shows two well-defined maxima at 134° and 165°K; this oscillatory behavior is interpreted as the successive escape of trapped holes from two discrete energy levels and their recombination with electrons. When the specimen was recooled at the same rate the conductivity curve showed no oscillations, which indicated that the traps were annealed. When the sample was returned to the low temperature the conductivity was about half the value prior to heating; a decrease would be expected if the hole traps had annealed, because an important source of photoconductivity would have been removed. (AEC Activity 5400)

OTHER PHYSICAL RESEARCH PROJECTS

Research Participation and Traveling Lecture Programs - The Research Participants who spent the summer at the Laboratory have completed their assignments and returned to their home universities. Of the 57 participants representing 35 universities, 46 were engaged in physical science research. One participant reported in September for a five-month appointment in the Mathematics Panel.

The Traveling Lecture Program for the academic year 1954-1955 has begun, with 31 lectures already scheduled, 16 under Program 5000. (AEC Activity 5XXX)

BIOPHYSICS

Ecology - Treehole mold of Acer saccharum and Quercus sp. from Delaware and Ulmus fulva and Liquidambar styraciflua mold from the AEC-controlled area were subjected to different amounts of hard gamma radiation from the Co^{60} source at Y-12. The dosages given were equivalent to 1×10^5 r, 2×10^5 r, 5×10^5 r, 7.5×10^5 r, and 1×10^6 r. The experimental samples, which weighed 50 or 100 g each, plus controls of like weight were then kept in Berlese funnels for 10 to 14 days for extraction of the surviving arthropods. The off-site (Delaware) mold arthropods had the following percentage decreases in population from the mean values for the controls: 5% at 1×10^5 r, 25% at 2×10^5 r, 62% at 5×10^5 r, 82% at 7.5×10^5 r, 99+% at 1×10^6 r. The on-site arthropods decreased 25% at 1×10^5 r, 60 to 70% at 2×10^5 r, 90 to 95% at 5×10^5 r, 95 to 99% at 7.5×10^5 r, 99+% at 1×10^6 r. At the present time, there is no explanation for the apparent greater sensitivity to the radiation of the on-site arthropod populations.

A second series of Liquidambar styraciflua treehole mold from the controlled area was similarly irradiated. The arthropod population decreased 40% at 1×10^5 r, and 60% at 2×10^5 r. The greater dosages were not given to this series. (AEC Activity 6510)

Evaluating Hazards Associated With Radioactive Wastes - From fission product yield data and the currently accepted maximum permissible concentration values for water and air given in NBS Handbook 52, the critical nuclides present in processing waste solutions were evaluated. Strontium was found to be the most critical.

Process Waste Treatment Experimental Plant Studies: A pilot-scale evaluation of the efficacy and economics of ion exchange processes for the treatment of ORNL process wastes was completed as planned (ORNL-1781, p. 33). The table below summarizes the results of the ion exchange studies. A complete report of the findings of the investigation was submitted, and discussions indicated that the Operations Division favors the use of a chemical precipitation process rather than the ion exchange process for the treatment of the Laboratory's process waste waters.

ORNL Process Waste Studies - Pilot Plant Studies
Ion Exchange

Run No.	Treatment	Volume Throughput (gal/ft^3 of resin) Hardness Break-through	90% Activity Breakthrough	Overall	Percentage of Activity Removed at End of Run
1	IR-120 (H^+)	8,000	7,200	9,350	67.5
2	IR-120 (Na^+)	7,400	6,210	9,180	77.2
3	Lime-soda ash softening + IR-120 (H^+)	5,800	3,200	15,109	84.5
4a	Diatomacreous earth filtration +IR-120 (H^+)	6,210	6,300	30,780	55.4
b	Diatomaceous earth filtraction +IR-120 (Na^+)	7,700	7,020	30,780	59.4

(AEC Activity 6510)

Math for Health Physics - Monte Carlo estimates of the average energy loss to a 30-cm slab of tissue from a broad beam of neutrons were made. The calculations were carried out as a function of slab penetration for neutron beams of 0.1 Mev, 5 kev, and 0.1 kev incident on one edge of the slab.

For each beam, 10,000 neutron histories were calculated, assuming energy loss by scattering with H, O, C, and N and by the absorption reactions $H(n,\gamma)$ and $N(n,p)$. The tissue absorption of the γ-rays resulting from $H(n,\gamma)$ was also a Monte Carlo estimate. About 25 hr of ORACLE time was required per beam. (AEC Activity 6510)

Isotopic Distribution in Man - The spectrographic laboratory for analysis of trace elements in human tissue completed a quantitative study of 10 elements in some 300 samples from 24 autopsies of instantaneous, accidental death. This work was reported to the American Chemical Society in Birmingham, Alabama, on October 21. (AEC Activity 6530)

Education, Training and Consultation - The 21 AEC Fellows now starting the program in Radiological Physics registered in the Physics Department, Vanderbilt University, September 27, for graduate work under the Fellowship program. (AEC Activity 6720)

Research Participation and Traveling Lecture Programs - The Research Participants who spent the summer at the Laboratory have completed their assignments and returned to their home universities. Of the 57 participants representing 35 universities, 11 were engaged in biological and biophysical research. There are presently three participants in the Biology Division.

The Traveling Lecture Program for the academic year 1954-1955 has begun, with 31 lectures already scheduled, 15 under Program 6000. (AEC Activity 6720)

RADIOISOTOPE SALES AND COSTS

Type of Transaction	September	FY to Date
Domestic Sales	$ 74,802	$247,868
Foreign Sales	3,745	14,305
Project-Cash Sales	2,050	16,220
Project-Transfer Credits	1,130	1,435
Technical Cooperation Program Credits	-0-	1,510
Plant Credits	1,217	7,502
Civilian Defense Credits	-0-	-0-
Cancer Program Credits	24,752	63,098
*Miscellaneous	-3	36
Total Income	$107,693	$351,974
**Income-No Production costs incurred Subtract from total income	-329	-5,793
	$107,364	$346,181
Radioisotope Costs	$ 74,714	$228,404
Radioisotope Shipments	946	2,981

*Miscellaneous Income - D_2O, Freight, Missing Parts, TCP.

**Income from H^3, He^3, B^{10}, and miscellaneous items in excess of handling costs.

GROSS OPERATING COSTS

	Cost for September	FY 1955 Cost to Date
Programmatic Operating Cost - Net	$ 2,494,530	$ 7,875,110
Plant and Equipment Cost	292,866	698,120
Construction Program "H"	5,021	15,660
Work for Other Parties - Transfers	30,289	80,937
Inventory Changes	8,344	77*
Reimbursable Work for Other Parties	141,118	440,752
Deferred Charges	1,591*	3,372*
Total Laboratory Cost - Net	$ 2,970,577	$ 9,107,130
Estimated Cost for Net Month - Net	$ 2,500,000	$11,607,130

*Credit

[redacted]

PERSONNEL SUMMARY

	Number of Employees October, 1954	New Hires October	Terminations October
Administration	62	0	0
Operations*	121	1	0
Engineering, Shops and Mechanical	821	3	1
Laboratory and Research	1867	33	15
Protection	147	1	0
Service	370	2	2
	3388	40	18

*Includes Electrical Distribution and Steam Plant as well as the Operations Division.

A total of 758 Laboratory personnel are located in the Y-12 Area.

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June 1954	ORNL-1748
July 1954	ORNL-1760
August 1954	ORNL-1781
September 1954	ORNL-1793

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