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STATUS AND PROGRESS REPORT
JUNE 1960

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JUNE 1960

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

June 1960

This Status and Progress Report summarizes that portion of the Laboratory's work which is unclassified. Some of the topics are included every month, but the majority are reported on a bimonthly schedule.

PROGRAM 2000 - SPECIAL NUCLEAR MATERIALS

Chemical Plant Criticality Studies. - The critical thickness of uranium metal slabs (93.2% U^{235} enrichment, 18.7 g/cc density) reflected by a 6-in.-thick layer of methacrylate plastic (Plexiglas) has been determined as a function of slab area from the extrapolation of neutron multiplication measurements. The critical dimensions obtained from a number of experiments are 5.0 x 5.0 x 3.1 in., 10 x 10 x 1.3 in., 15 x 15 x 0.95 in., 20 x 20 x 0.80 in., and 25 x 25 x 0.70 in. These results extrapolate to 0.60 ± 0.05 in. as the thickness of a reflected slab infinite in area. A single experiment with a slab having a 12-in.-thick beryllium reflector gave critical dimensions of 5.0 x 5.0 x 1.4 in.; a 12-in.-thick graphite reflector resulted in a critical slab 8 x 10 x 1.3 in. (AEC Activity 2712)

Dissolver Solution Analyses. - Studies of the controlled-potential coulometric titration of plutonium in power reactor fuel processing end-product samples are complete. A report (ORNL-2921) has been issued which describes the method, its performance under ideal and routine operating conditions, and the effects of impurities normally encountered in this type of sample. Work is continuing on the application of this technique to the analysis of dissolver solutions and is being directed toward finding the optimum separation procedure to precede coulometric titration. The coulometric titration procedure described in earlier reports proved to be satisfactory for remote analysis in the High-Radiation-Level Analytical Facility when highly radioactive uranyl nitrate solutions (derived from dekad uranium rod) were analyzed. No separation was necessary for that application.

Standard uranium solutions containing the various ions and fission products expected to be encountered in power reactor fuel dissolver solutions were extracted from an aluminum nitrate salting agent into a triisooctylamine-xylene solution (ORNL CF-60-3-123). A subsequent stripping of the uranium into an aqueous phase with an $HClO_4-H_2SO_4$ solution, and a chemical pretreatment with ascorbic acid and NH_4OH , enabled a quantitative reduction and determination of the uranium with a controlled-potential coulometer. The results obtained indicated accuracy and precision within 1%. As an alternate method, uranium was separated from synthetic reactor fuel dissolver

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solutions by the use of an anion exchange resin (ORNL CF-60-4-62). The uranium was then determined by a controlled-potential coulometric titration. Relative standard deviations of 0.69% for samples containing 0.8 mg of uranium and 0.15% for 8.0 mg of uranium were obtained. (AEC Activity 2724)

PROGRAM 4000 - REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

EGCR Axial Power Distribution. - A three-dimensional analysis of the EGCR power distribution has been completed for the case in which the central control rod is fully inserted and the four control rods 24 in. from the center of the core are inserted to the midplane. The results of this calculation gave the axial power distribution in each channel of the core. The specific operating condition considered resulted in an over-all radial peak-to-average power ratio of 1.26, which is well below the limit of about 1.35 set by the available pumping power. The axial peak-to-average power ratio is quite high in some channels, however, and approaches 2.0 in the worst cases. (AEC Activity 4141.1)

EGCR Gas and Fuel-Element Temperatures. - The gas and fuel-element temperatures for the operating conditions described above were calculated. In the analysis it was assumed that all 232 channels in the core were loaded and that 80 of the 85 Mw of heat generated remained in the fuel elements, with the remainder being in the graphite sleeves and moderator. Temperatures ranging from maximum to minimum based on variations in coolant channel orifice settings were determined. Between the two extremes it was found that with 60% of the channels orificed to control the exit gas temperature and 40% orificed to control the fuel-element surface temperature, the maximum exit gas temperature would be 25°F above the mixed mean temperature for the gas from all channels and that the maximum nominal fuel-element surface temperature would be 1300°F. (AEC Activity 4141.1)

Fission-Gas Release from UO₂. - Studies of the rates of fission-gas release during out-of-pile heating of UO₂ specimens previously irradiated at low temperatures to low burnups have shown that a marked increase in the rate of release occurs when the sintering temperature is exceeded. In one test on a 95%-dense UO₂ sample, 31% of the Xe¹³³ was released in 30 min at 2000°C. In order to determine the mechanism of release at temperatures above the sintering temperature, a number of samples have been sintered at 1900°C for various lengths of time and reirradiated. Samples of fused UO₂ have also been irradiated, and measurements of rates of release of Xe¹³³ from these samples are being conducted.

Changes have occurred within the past month in the fission-gas retention characteristics of the thin plates of UO₂ being irradiated in the ORR instantaneous fission-gas-release experiment. Under normal conditions, a burst of activity resulted that was more than six times normal. The spectrum of the gas was abnormal, with the long-lived gases Xe¹³³, Xe¹³⁵, Kr⁸⁸,

Kr⁸⁷, and Kr^{85m} dominant. The release of energy stored in the fuel has been observed on two occasions. The amount of energy released was 6 cal/g on one occasion and 4 cal/g on the other. It is known that UO₂ with an oxygen-to-uranium ratio of 2.08 or higher will store energy and that a high oxygen-to-uranium ratio affects the gas-retention characteristics. Thus it appears that the UO₂ has become oxidized to some extent. Although the sweep gas is cleaned before use, even 1 ppm of O₂ could, over a period of several months, easily increase the oxygen-to-uranium ratio of the UO₂ from the initial 2.01 to more than 2.08. (AEC Activity 4141.1)

Reactions of Type 304 Stainless Steel with CO-CO₂ Mixtures. - The reaction products which resulted from exposure of type 304 stainless steel to CO-CO₂ mixtures at a CO-to-CO₂ ratio of 13:1 at 1800°F were found to be a bulk oxide of Fe₃O₄ at the gas interface and 8-μ-thick crystals of green Cr₂O₃ at the metal interface. The green crystals extended into the bulk oxide as well as into the grain boundaries. Oxidation of the metal accounted for 90% of the weight change and carburization for 10%.

The origin of Fe₃O₄ as the principal oxide in a high concentration of CO is not understood; it should not have formed under the test conditions. In fact, iron oxides of any type should be reduced to iron in a 13:1 mixture of CO and CO₂. It is postulated that Fe₃O₄ may be formed by (1) the decomposition of FeO, (2) the decomposition of Fe(CO)₅, or (3) oxidation of the spinel FeO·Cr₂O₃ by CO₂ to form Cr₂O₃ and Fe₃O₄. (AEC Activity 4141.1)

Diffusion of Nonvolatile Fission Products in Graphite. - A study has been initiated of the mechanisms involved in the release of fission product solids from unclad fuel elements consisting of UO₂ dispersed in graphite or other matrix material. The two principal release mechanisms are solid-state diffusion from the fuel lattice and recoil into the matrix. Measured rates of diffusion of individual fission products into AGOT graphite at temperatures between 750 and 1200°C gave diffusion coefficients that decreased with increasing atomic radii. On the other hand, studies in the temperature range 1300 to 1900°C indicated significant changes in the mass-transport mechanisms. Rapid fixation of elements that form stable carbides at these temperatures (e.g., Y₂O₃ and ZrO₂) takes place, and the migration of others that do not form stable nonvolatile compounds with the matrix (e.g., BaO and CeF₃) is accelerated. (AEC Activity 4141.1)

Outgassing of Graphite. - A 16 × 16 in. extrusion of graphite, that is, an extrusion of the size specified for the EGCR graphite columns, was received from the National Carbon Company for degassing studies. Three specimens (1-1/2 in. in diameter and 2 in. long) were machined from the extrusion, which was prepared from needle coke. During external resistance heating from room temperature to 1000°C, a total of ~ 27 cm³ of gas per 100 cm³ of graphite was evolved that consisted of about one-third H₂ and two-thirds CO₂ + CO. The volume of gas evolved during heating from room temperature to 600°C was greater than that released from 600 to 1000°C. The value of ~ 27 cm³ of gas for this needle coke is to be compared with a value of ~ 19 cm³ of gas evolved by specimens taken from a 16 × 16 in. extrusion prepared from Texas coke and a value of ~ 18 cm³ of gas evolved by AGOT-VCB specimens taken from a 4-1/2 × 4-1/2 in. extrusion of needle coke. (AEC Activity 4141.1)

Experimental Operation of Small-Scale EGCR Helium Purification System Installed on Forced-Convection Loop. - The EGCR helium purification system is being evaluated in tests of a small-scale system that includes a catalytic converter containing Girdler-type G-43 catalyst for converting H_2 and CO to H_2O and CO_2 , a cooler-condenser and silica-gel adsorber for removing H_2O , and a molecular-sieve adsorber for removing CO_2 . Tests are being run to establish the conditions for addition of the oxygen required for operation of the catalytic converter. During these tests the main loop is operated at approximately $100^\circ F$, and the purification system preheater is used to heat the bypass gas stream to $1050^\circ F$ ahead of the catalytic converter. Hydrogen gas is added to the main loop to a predetermined level, the desired flow rate through the purification system is established, and then O_2 is injected at the inlet to the catalytic converter at a specified rate.

In one test the H_2 concentration in the loop was raised to 0.13%, and a flow of 2.1 lb/hr was established through the purification system. Oxygen was then injected at the inlet of the catalytic converter at the rate of $10 \text{ cm}^3(\text{STP})/\text{min}$. After 32 min, O_2 was observed at the converter exit, and the O_2 injection was stopped. Circulation of the loop gas through the purification system was continued, and approximately 30 min later all the H_2 had been removed from the loop gas. Excess oxygen was observed at a level of 8 ppm. (AEC Activity 4141.1)

BeO-Moderated Reactors. - A series of calculations has been started to explore the nuclear characteristics of two-region, semihomogeneous BeO-moderated reactors in which the fissionable material ($U^{233}O_2$) is in a central cylindrical core and the fertile material (ThO_2) is entirely in a surrounding blanket. Initial results give a rather small core diameter of about 70 cm, which implies that the thermal power generation would be limited to approximately 100 Mw per core. A net conversion ratio of 1.10 was obtained after making allowance for parasitic capture, leakage, and processing losses. Parasitic capture includes fission products appropriate to 40% burnup of the fuel on a continuous fueling cycle. About 0.07 of the conversion ratio is attributable to the beryllium fast effect. Further calculations will be made of multiple-region reactors in an effort to retain the high conversion ratios resulting from complete segregation of fissionable and fertile materials while, at the same time, increasing the total power output to an economically attractive value. (AEC Activity 4141.1)

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. - The program of reactor maintenance and alterations continues. The inner surface of the core was inspected following wire-brushing; much of the surface coating had been brushed away. The lower (first) core hole was patched, but the patch had to be removed because of excessive leakage. Another impression of the hole will be taken, by use of a plastic block which has been precast into the approximate shape of the hole, so that another patch can be fabricated.

In addition to the reactor core work, the fuel primary recombiner was replaced. Investigations are being conducted to determine the reason for its malfunctioning. Tests of the reactor containment-shield penetrations continued, and repairs were made in instances where leakage was found to be excessive. (AEC Activity 4151.3)

HRT Chemical Pilot Plant. - The multiple hydroclone originally installed across the reactor heat exchanger was tested after removal from the cell, and it was found that the feed ports had become partially plugged, which would seriously reduce the efficiency of the unit. From radiation measurements, an accumulation of solids estimated at 100 g was present in the feed chamber (volume 300 cc). A significant fraction of these solids was flushed from the system during the tests, but this did not free the plugged hydroclones. Pressure drops had increased 30 to 50% since installation, and the induced underflow rate was found to be 8 to 10%, as compared with an expected 3 to 5%.

Fabrication of an improved multiclone was completed, and preoperation performance tests, including solids efficiency measurements, were made. It will be installed with the new reactor fuel-circulating pump prior to the next period of operation. (AEC Activity 4151.1)

Heterogeneous Equilibria in Aqueous Systems. - The solubility of ThO_2 at 300°C in $\text{HNO}_3\text{-H}_2\text{O}$ was determined for solutions having total nitrate concentrations between 0.06 and 10 M. The mole ratio $\text{Th}:\text{NO}_3^-$ at saturation was found to be 0.0001 at 0.10 M NO_3^- , 0.01 at 1.0 M NO_3^- , and 0.20 at 10 M NO_3^- . Two values for this ratio at 300°C , derived from previous ORNL work, are 0.15 at 5.3 M and 0.18 at 6.3 M NO_3^- ; these values are consistent with the present data. The corresponding solubilities of UO_3 in $\text{HNO}_3\text{-H}_2\text{O}$ at 300°C , expressed by the values of the ratio $\text{UO}_3:\text{NO}_3^-$, are 0.45 at 0.10 M, 0.53 at 1.0 M, and 0.55 at 5.0 M NO_3^- . Comparison of these two sets of data indicates that thorium is somewhat less soluble than uranium in concentrated nitrate solutions but very much less soluble than uranium in dilute nitrate solutions. (AEC Activity 4151.1)

Reactions in Aqueous Solutions. - Laboratory studies were made of the kinetics of the dissolving of solids in simulated HRT fuel, in well-stirred autoclaves at 250°C . Several solids which conceivably could have existed within the HRT core as a consequence of off-design operation were found to dissolve within minutes. The solids tested and the approximate half times for their dissolving under comparable conditions were:

	<u>Minutes</u>
UO_3	0.5
UO_2 ("reactor grade"; 28% U_3O_8)	0.5
UO_2 (crystalline, prepared by precipitation from molten salt, representing the most refractory material available)	2.2-2.4
U_3O_8	2.2-2.5
$\text{CuO}\cdot 3\text{UO}_3$	0.8-2.0
Reactor-solution solids (prepared by calcining simulated fuel solution at 500°C)	< 0.5

These results suggest that the solids tested, if formed in the reactor, should redissolve promptly under conditions of uniform temperature and good agitation, such as are being provided by the present modifications to the HRT. (AEC Activity 4151.1)

Analytical Chemistry. - A coulometric method was found to be applicable for the determination of uranium(IV) in slurries of $\text{ThO}_2\text{-UO}_x$ which may contain impurities including corrosion products of stainless steel. After the sample is dissolved in 7 M H_3PO_4 , the uranium(IV) is determined by coulometrically oxidizing it to uranium(VI) at a platinum anode maintained at a controlled potential of 1.4 v vs a silver-silver chloride electrode. Over the range from 1 to 10 mg of uranium(IV), the coefficient of variation is 1%. Of the corrosion products of stainless steel, only iron(II) interferes; this interference can, however, be eliminated by preoxidation of the iron at a potential of 0.4 v vs the silver-silver chloride electrode.

An extraction procedure has been devised whereby several grams of thorium can be rapidly separated for analytical purposes from many elements, including those of the rare-earth group. The thorium is extracted with 0.5 to 1.0 M thenoyltrifluoroacetone (TTA) in chloroform from a solution of pH 1.0. During the extraction the acidity is maintained constant by the continuous slow addition of an acetic acid-ammonium acetate buffer of pH 4.3. Under this condition, few elements other than thorium are extracted, and from 1 to 5 g of thorium can be extracted in 30 min or less; furthermore, no difficulties due to precipitation of basic salts of TTA or emulsion formation are encountered. The acetate in the raffinate can be readily destroyed with aqua regia, after which the solution can be used for further analyses. (AEC Activity 4151.1)

Slurry Circulation Experiments. - A pure thoria slurry (1600°C-fired, 2 μ mean particle diameter) was circulated successfully for 3200 hr in the 200B loop to obtain turbulent-flow heat transfer measurements at elevated temperatures. Tests were made in the concentration range 260 to 830 g of Th per liter and the temperature range 180 to 275°C. Inspection of the heat transfer section following the run revealed no slurry deposits on the heat transfer surface or adjacent piping. Additional slurry heat transfer measurements will be made with the same slurry at 1200 g of Th per liter. (AEC Activity 4151.1)

Slurry System Development. - The 300-SM system was successfully operated during a 1962-hr run with equipment such as would be used in a slurry-fueled reactor experiment. The run included 1312 hr of high-temperature slurry circulation at temperatures up to 300°C with ThO_2 slurry at concentrations up to 1050 g of Th per liter. Some operations which were demonstrated included charging, dilution, dumping, and resuspension after shutdown. Both the high- and the low-pressure system operated as designed during these procedures. Successful functioning of the 30-in. core vessel, the circulating pump, the heat exchanger, the pressurizer, the slurry valves, the slurry feed pump, and the slurry storage tank was demonstrated. (AEC Activity 4151.1)

Core and Blanket Vessel Development. - Measurements of fluid residence time at various locations in the HRT core model with reversed flow were made

by using a tracer-decay (or tracer-buildup) technique. These data supported the previous estimates for the maximum HRT fuel temperature of 305°C when the reactor is operated at a power level of 5 Mw and with a clean core wall.

Flow tests were made with a 2-ft-dia, 6-ft-long cylindrical core model. Fluid was introduced through annular swirl-producing vanes and removed through a polar outlet, with both inlet and outlet at the top of the cylinder. Vane designs with 10 and 45° exit angles promoted excessive bypassing of the inlet fluid directly to the outlet pipe. Additional tests with a 5-in.-dia cylindrical model are in progress to find inlet and outlet configurations which promote flow of inlet fluid along the cylinder with little recirculation of the interior fluid.

Preliminary tests of swirling straight-through flow in a 3-in.-dia cylindrical model showed promise for a flow pattern in which inlet fluid bathes the cylindrical wall throughout the length of the vessel. Further tests of this type of core flow are in progress. (AEC Activity 4151.1)

Component Development. - The replacement fuel-circulating pump for the HRT was prepared for installation. The replacement primary recombiner passed a performance test which indicated efficiency of H₂-O₂ recombination above 98%. (AEC Activity 4151.1)

Homogeneous Reactor Blanket Studies. - In the preparation of spherical pellets of ThO₂ by press-forming of oxalate-precipitated thorium and calcining, the optimum pressures for granulating powder varied from 5600 to 15,000 psi for powders fired at 800 to 1000°C. When the granulating pressures were outside this range, weak or cracked green pellets were produced on press-forming. In firing studies on pellets prepared from three powders, as the firing temperatures increased from 1300 to 1750°C the bulk densities increased from 7.2 to 8.9 g/cc and attrition by the spouting-bed test decreased from 3.3 to 1.6 wt %/hr. (AEC Activity 4151.1)

Molten-Salt Reactor Program

Chemistry. - Considerable latitude for interchange of ZrF₄, ThF₄, and UF₄ in breeder fuels without adverse effects on the melting point is expected on the basis of experiments showing that LiF-BeF₂ melts containing 60 to 70% LiF dissolve up to 15 mole % ZrF₄ at 500°C.

The MSRE fuel composition, LiF-BeF₂-ThF₄-UF₄ (65-30-4-1 mole %), lies on the crystallization path between 3LiF·Th(U)F₄ and 2LiF·BeF₂, giving a mixed precipitate of these two compounds on "equilibrium" cooling from 448 to 423°C. Below 423°C, 2LiF·BeF₂ and 7LiF·6Th(U)F₄ crystallize simultaneously.

The selective or fractional precipitation of oxides from blanket fuels is presumably governed by the sequence of increasing solubilities, Pa₂O₅ < UO₂ < ThO₂ < BeO. Additional evidence to establish this sequence was obtained when 50 to 75 ppm of protactinium was removed from LiF-BeF₂-ThF₄ (67-18-15 mole %) by adding UO₂, nominally quite insoluble, as a precipitant. The fact that 8 wt % UO₂ was found necessary for complete removal is probably due to coating of the surface of the UO₂ with reaction products.

Fusion of ZrF_4 with ZrO_2 at $1000^\circ C$ produced a single phase of $ZrOF_2$ with the same crystallographic properties as one of the products resulting from the treatment of ZrO_2 with molten NH_4HF_2 .

Two corrosion test loops, each of which was operated for approximately 900 hr in the MTR, were found to contain satisfactorily low concentrations of corrosion products (500 ppm or less Cr^{++}) in the fuel. Comparable behavior for a longer term in out-of-pile tests is shown by an INOR-8 pump loop (1300 to 1100°F gradient) which has maintained an expected steady-state Cr^{++} concentration of 550 ppm for 15,000 hr while circulating LiF - BeF_2 - ThF_4 - UF_4 (62-36.5-1-0.5 mole %). (AEC Activity 4152.1)

Metallurgy. - Tests were performed to determine the effect of exposure under creep conditions on the high-temperature tensile properties of INOR-8. Specimens exposed to air at $1250^\circ F$ exhibited no loss in the tensile yield strength at $1300^\circ F$. One specimen, exposed for 8564 hr at 10,000 psi, exhibited a tensile strength of 35,000 psi, but all other values were above 60,000 psi, which is approximately the value for the material in the starting condition. The tensile elongations were also satisfactory, having values ranging from 30 to 45%. The only specimen exposed to molten salt showed an increase in the yield strength of 13,600 psi over the initial value. No significant loss in either the tensile strength or the elongation was observed in this test.

Four additional grades of graphite were subjected to the standard screening permeation test. They were exposed for 100 hr to LiF - BeF_2 - ThF_4 - UF_4 (67-18.5-14-0.5 mole %) (BULT 14-0.5U) at $1300^\circ F$ ($704^\circ C$) and 150 psig. The results and the apparent densities of the different grades are summarized below:

Graphite Grade	Apparent Density		Bulk Volume Permeated by BULT 14-0.5U	
	Average Value (g/cc)	Number of Specimens	Average Value (%)	Number of Specimens
CEY	1.91	6	1.0	6
R-0025	1.87	24	5.7	24
RLM-24	1.83	6	5.7	13
MH4LM-82	1.81	24	8.2	24

Grades CEY, R-0025, RLM-24, and MH4LM-82 are in positions 9, 20, 21, and 24, respectively, when all 31 grades of graphite that have been screened in this way are listed in the order of increasing values of their bulk volumes permeated.

Since grades R-0025 and MH4LM-82 were fabricated as large pieces, the screening tests included specimens from different locations and orientations in order to investigate the uniformity of large graphite pieces. The results indicated that the large pieces of grades R-0025 and MH4LM-82 were uniform. (AEC Activity 4152.1)

Corrosion Tests. - Ten corrosion pump loops, eight of INOR-8 and two of Inconel, have been in operation with various fluoride salts during this month. The loops have been operated at high wall temperatures of from 1200 to 1400°F for periods of from 6700 to 18,000 hr. (AEC Activity 4152.1)

In-Pile Tests. - The second MSR graphite-fuel capsule irradiation experiment has completed four cycles of operation of the MTR, yielding approximately 1600 hr of operation. It is now being removed for examination.

Preliminary work has been done preparatory to disassembly of the first experiment in the segmenting cell in Building 3026. The hot-cell work has been reviewed by the Hot Cells and Sources Committee. (AEC Activity 4152.1)

Pump Development. - The molten-salt pump containing a salt-lubricated journal bearing continued in operation and has logged 1700 hr. Operation during this month was at steady-state conditions of 1225°F, 1200 rpm, and 75 gpm.

The MF-F pump endurance test was terminated for examination of the pump and loop. The test operated continuously for approximately 25,500 hr, circulating molten salt No. 30 at 1200°F.

A small frozen-lead-seal pump consisting of a centrifugal pump mounted vertically over a fractional-horsepower motor drive has operated continuously for two years. The pump circulates molten salt isothermally at 1200°F. (AEC Activity 4152.1)

Remote Maintenance Demonstration. - The motion picture "Remote Maintenance of Molten-Salt Reactors," in color and with sound, was completed.

Several minor tool alterations were made in order to facilitate easier performance of the various remote-maintenance operations.

Practice operations consisting of the remote removal and installation of the heat exchanger, pump motor, pump, and reactor vessel, by the use of revised tools and procedures, are in progress. (AEC Activity 4152.1)

Heat Transfer and Physical Properties. - A total of 5000 hr of circulation has been accumulated on the system for determining the heat-transfer coefficients for BULF-14 flowing through heated Inconel and INOR-8 tubes. The turbine flowmeter signal was spontaneously recovered at 4450 hr after a 700-hr loss. Subsequent comparison of the flow rates as indicated by the turbine meter and the pump characteristic curve showed a 3% deviation. A faulty pump bearing has necessitated a second shutdown.

No systematic variation of the heat-transfer coefficient has been observed. The previously reported discrepancy between the heat transfer in the Inconel and INOR-8 tubes has been resolved. The mean values of the heat-transfer modulus for both sets of data now agree closely; the Inconel-tube data, however, exhibit a greater experimental scatter. The results lie, on the average, 25% below the general heat-transfer correlations.

Measurements of the surface tensions of three NaF-BeF₂ mixtures were completed, and analysis is in progress. The data are in reasonable agreement with the values reported earlier for two LiF-BeF₂-UF₄-ThF₄ salts. Improvements in both instrumentation and experimental procedure have decreased the uncertainty of the determination from ±8% to ±3%. However, an uncertainty remains as to the liquid density. The densities of these three NaF-BeF₂ mixtures have been derived from measurements made in order to establish the effect of capillary immersion depth on the surface tensions of the salts.

Data at three temperatures from one of the salts show a maximum of 2% scatter about a mean line. A comparison with a prediction of the density shows comparable temperature dependence but a 7% discrepancy in magnitude, with the experimental result being the higher value. Some question remains, however, as to the exact composition of the mixture studied. (AEC Activity 4152.1)

Fuel Processing. - The solubility of LiF in aqueous 50 wt % ammonium fluoride solution at 100°C was found to be about 0.5 mg/ml, significantly less than its solubility in water. Thorium fluoride was soluble in aqueous 50 wt % ammonium fluoride, but in the presence of LiF the thorium solubility decreased markedly. Probably an LiF-ThF₄ compound is formed which precipitates thorium from the complex formed with ammonium fluoride.

Thorium fluoride was found to be insoluble in aqueous beryllium fluoride solutions. Solutions containing 0.50 and 0.77 g of BeF₂ per milliliter of solution were contacted with ThF₄ powder for 24 hr at 100°C with no apparent reaction; no thorium was found in the solution, the limit of detection in the analyses being about 8 mg/ml. The apparent absence of any interaction is in agreement with the BeF₂-ThF₄ phase studies (ORNL-2548) showing no compounds between these components. (AEC Activity 4152.1)

Molten-Salt Reactor Experiment. - Design of a 5-Mw (thermal) Molten-Salt Reactor Experiment was begun. The core layout is essentially completed and ready for detailing.

Preliminary designs of the primary heat exchanger and the salt-to-air heat pump have progressed to the point where they can be reviewed by heat exchanger manufacturers for fabrication recommendations and cost estimates.

Layouts of the complete reactor complex are in progress, with primary consideration being given to optimizing the component arrangement for performance of remote-maintenance operation.

Favorable reactions have been received from INOR-8 fabricators regarding the availability of the material and a reasonable time schedule. (AEC Activity 4152.1)

GENERAL REACTOR RESEARCH

Release of Fission Products on In-Pile Melting of Reactor Fuels. - In the program for the study of the release of fission products resulting from the in-pile melting of fuel elements (ORNL-2945), the initial experiments will involve melting pellets of UO₂ clad in stainless steel. Final design drawings of the experimental assembly for use in the ORR are estimated to be 75% complete; instrument drawings necessary for review of the experiment by the safety committee are being prepared. An Oracle program has been set up to provide improved estimates of temperature profiles within the assembly under various operating conditions. Special materials such as ceramics, flexible hose, and high-temperature thermocouple combinations have been ordered, and fabrication of parts for the assembly is in progress. A mockup of the reactor entry tube has been satisfactorily tested in the ORR. (AEC Activity 4171.1)

Basic Shielding Research. - Measurements of secondary gamma-ray production in a Pratt and Whitney Aircraft reactor shield are being conducted

at the Lid Tank Shielding Facility. The neutron spectrum incident on the inner surface of the Pratt and Whitney reactor shield has been simulated by placing slabs of various materials in front of the shield slabs. These materials duplicate the attenuation characteristics of all materials necessary for operation of this reactor. Gamma-ray dose rates, fast-neutron dose rates, and thermal-neutron fluxes are being measured in a tank of boric acid water behind various shield configurations. Neutron spectra are being measured inside one shield by placing fission foils at various depths in the shield. Analysis of the data will be done by Pratt and Whitney. (AEC Activity 4410)

Power Reactor Fuel Processing: Solvent Extraction. - Synthetic Consolidated Edison feed solutions were irradiated in a Co^{60} gamma-ray source and processed according to an Acid Thorex flowsheet. Gross gamma decontamination factors were 2500, 2000, 1500, and 6000 with solutions that had been irradiated to 0, 5, 10, and 40 whr/liter. Experiments are in progress to determine the reason for the high decontamination of the 40-whr/liter-irradiated feed.

Distribution coefficients for uranium were determined (some 200 values) for systems consisting of 5, 10, and 15% solutions of tributyl phosphate in Amsco and aqueous solutions containing 0.5, 2, 3, and 4 moles of HNO_3 and 0.1 to 100 g of nonirradiated uranium per liter, and tables and graphs were prepared. (AEC Activity 4441)

Power Reactor Fuel Processing: Mechanical Processing. - Leaching studies to determine the instantaneous dissolution rates of several types of unirradiated UO_2 pellets in boiling nitric acid were made. Wide variations, dependent on some property of the pellets other than density or chemical composition, were observed. Specification-grade PWR pellets dissolved at rates slightly lower than those found previously for reject-grade PWR pellets. Pellets from the Davison Chemical Company dissolved at even lower rates, as did also pellets reported on by Canadian experimenters. Typical instantaneous dissolution rates in boiling 10 M HNO_3 were 90 (reject PWR), 62 (specification PWR), 23 (Davison), and 18 (Canadian), all expressed in $\text{mg}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$. (AEC Activity 4441)

Power Reactor Fuel Processing: Sulfex Process. - Investigation of the instantaneous reaction rate of type 304L stainless steel tubing in sulfuric acid containing dissolved stainless steel was continued in an attempt to explain the wide fluctuation in dissolution rate previously observed in the operation of the Sulfex small-engineering-scale equipment. With 2 to 6 M H_2SO_4 containing 0 to 1.0 M dissolved stainless steel, the instantaneous reaction rate increased with increasing acidity. No correlation of the rate with the stainless steel concentration at constant acidity was apparent; within the scatter of the data it must be concluded that the reaction rate is independent of the stainless steel loading.

Heat treatment of Ni-o-nel to 1800°F before welding minimized local attack in Thorex dissolver solutions; borate lowered the corrosion rate by a factor of 5. (AEC Activity 4441)

Power Reactor Fuel Processing: Darex-Thorex Process. - A study of the reproducibility of the instantaneous dissolution rates of $\text{UO}_2\text{-ThO}_2$ pellets

in Thorex dissolvent was made; normal distributions were approximated at both 0 and ~ 50% of total dissolution. The rates were 0.325 and ~ 0.8 $\text{mg}\cdot\text{g}^{-1}\cdot\text{min}^{-1}$ at 0 and 50% dissolution respectively.

The addition of $\text{Cd}(\text{NO}_3)_2$ as a neutron poison to boiling 13 M HNO_3 -0.04 M NaF solutions decreased the dissolution rate of 95% ThO_2 - UO_2 pellets (Universal Match Corporation, 95% of theoretical density). In aluminum-free dissolvent the initial 10-min dissolution rate decreased from 2.0 to 0.67 $\text{mg}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$ as the $\text{Cd}(\text{NO}_3)_2$ concentration was increased from 0 to 0.075 M.

In 135 hr exposure to Thorex dissolver solution boildown conditions, titanium showed negligible corrosion. In HNO_3 containing 0.003 to 0.05 M HF, increasing the HNO_3 from 6 to 15 M decreased the titanium corrosion rate from 150-600 to less than 3 mils/month. Types 347 and 304L stainless steels were attacked intergranularly in Darex-Thorex waste solutions and therefore could not be used with these solutions. (AEC Activity 4441)

Power Reactor Fuel Processing: Zircex Process. - The study of the application of the Zircex process to previously clad U-Mo fuel alloy was continued. A 91.6% U-Mo alloy was exposed to mixed hydrogen chloride and air (10 to 70% HCl) for 1 hr at 400 to 600°C. Reaction rates of 8 to 10 $\text{mg}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$ were achieved. With chlorine at furnace temperatures above 450°C, the rate was 20 $\text{mg}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$. Removal of 98 and 80% of the molybdenum was achieved with chlorine and with mixed hydrogen chloride and air respectively. Adding air to hydrogen chloride doubled the reaction rate and caused the removal of over 99% of the chloride from the product by the quantitative formation of molybdenum oxychloride. Aluminum reacted with both hydrogen chloride and chlorine at a furnace temperature of 300°C at a rate of 11 $\text{mg}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$.

The corrosion rate of Nichrome V in mixtures of nitrogen and carbon tetrachloride increased with temperature, from 0.2 mil/month at 400°C to 5.5 mils/month at 600°C. (AEC Activity 4441)

Power Reactor Fuel Processing: Alternative Aqueous Processes. - A feed preparation and solvent extraction flowsheet was developed for the recovery and decontamination of highly enriched uranium from the LAPRE-II reactor fuel solution. The solution, which contains U(IV) in concentrated H_3PO_4 , is first diluted with water; ferric nitrate is added to complex phosphate and supply salting strength; and sodium nitrite is added to oxidize U(IV) to U(VI) so that uranium may be extracted with 6% tributyl phosphate. The flowsheet is designed for operation in non-critically-safe equipment.

The dissolution of U-10% Mo alloy in nitric acid to produce solutions 1 M in uranium always results in precipitation of the molybdenum. The solids from solutions in which the final H^+ concentration was greater than 5 M appeared to be MoO_3 ; at lower H^+ concentrations, insoluble uranyl molybdates were formed.

A series of tests was made on Pebble Bed Reactor fuel samples to evaluate process variables for graphitized vs ungraphitized fuel. The uranium recovery by leaching of ground (-16 +30 mesh) specimens with two portions of 15.8 M HNO_3 was slightly higher (99.8%) from ungraphitized fuel than from the graphitized samples (99.5%). When 1.5-in.-dia fuel spheres were treated with 90% HNO_3 (21.2 M), the graphitized samples disintegrated more rapidly than the ungraphitized ones. (AEC Activity 4441)

Power Reactor Fuel Processing: Criticality Control. - Boron glass rings in a conventional tank are being considered for criticality control. In tests with 75 and 150 gal of solution to measure mixing in packed tanks, at air flow rates of 0.14 and 0.55 scfm per square foot of tank cross section, a sparge ring was more effective than slotted or conventional draft tubes. Thorough mixing of a 75-gal batch required 3 to 6 min; a 150-gal batch required 4 to 20 min. No breakage and no change in the 77.4% void fraction were observed in these short-term tests. (AEC Activity 4441)

Waste Disposal Research and Engineering: Geochemical Studies. - High specific affinity for strontium by the mineral variscite $[\text{Al}(\text{OH})_2\text{H}_2\text{PO}_4]$ and by the precipitate of aluminum phosphate has been demonstrated with slurry techniques. Studies have been extended for strontium removal, using precipitation principles in a column-type system. The method consisted in using an alumina "bed" in columns and adding Na_2HPO_4 (10^{-3} M) to the waste. The pH of the 0.1 M NaNO_3 simulated waste was adjusted to 7.0; the waste contained 1.1×10^{-5} M strontium ions (1 mg/liter). With 1 g of aluminum oxide powder, more than 99.5% of the strontium was removed in the first 15 ml of solution. A 1-g vermiculite column under the same conditions removed 36.8%, and an iron oxide column removed 36.2%. After 70 ml the aluminum oxide and iron oxide columns continued to remove about 99 and 36% respectively; the vermiculite column had gradually decreased to about 15%. Studies are being extended to elucidate the mechanism of the aluminum phosphate reaction; other studies are in progress to exploit the possible application of the "alumina column" for waste disposal. (AEC Activity 4452)

Waste Disposal Research and Engineering: Disposal in Natural Salt Formations. - The field experiments with simulated Purex wastes have operated for five months. Maximum average temperatures in both the neutralized and acid cavities have been reached, and the anticipated slow drop is taking place. Average temperature rise in the neutralized experiment is 50°C and in the acid 40°C. Temperatures in the salt have changed little since April.

Dial-gage extensometers installed in the vicinity of the neutralized waste cavity measure changes (creep) in the floor-to-ceiling and column-to-column dimensions, and movement in the floor. Data taken before and during heating indicate that the room was stable prior to heating, and although the creep rate has increased with temperature, the room is now approaching a new condition of stability. Thus heating of the salt has not upset the equilibrium in the area.

Solutions of synthetic formaldehyde-denitrated Purex waste containing (1) no salt and (2) excess salt were irradiated in sealed tubes in the Co^{60} facility for 431 hr, during which time the total dose approached 10^9 rads. Both systems attained apparent steady-state conditions, as indicated by the leveling off of the pressures within the tubes. The pressures of the solutions containing no salt were of the order of 2 to 3 atm, while those of the salt-saturated solutions were 3 to 4 atm. Hydrogen and oxygen were the only gases that could be identified from the gaseous products of the reaction.

The solution in the acid pit at the field site was monitored for changes in acidity. A net decrease of 0.5 has been observed in the molarity of the free acid over a period of four months, within which period the solution temperature has attained 64°C from an initial ambient temperature of 21°C. The greatest rate of change in acidity occurred when the solution temperature was about 60°C.

The analysis of the gases above the solution showed that carbon dioxide, nitrous oxide, and nitric oxide were formed when the acid solution was placed in the pit. The nitric oxide content increased as the temperature was raised. When the solution temperature rose to 60°C, chlorine and nitrosyl chloride were first observed, and their concentrations increased at subsequent samplings. (AEC Activity 4452)

Waste Disposal Research and Engineering: Disposal of ORNL Radioactive Wastes. - In order to determine the amount, if any, of activity contributed by Burial Ground 4 to White Oak Creek, temporary monitoring stations were installed along the creek immediately above and below the site. Water was pumped continuously from the creek at each station, and once each day a 1-liter sample was taken from the liquid collected. These daily samples were composited usually in periods of one week.

For the period between February 16 and May 25, ten composite samples, representing a total of 67 days, were collected. The total transport of radionuclides by the upstream station included 18.2 curies of rare earths, 5.9 curies of Ru, 4.2 curies of Sr, 2.4 curies of Cs, 1.7 curies of Co, 1.1 curies of Zr-Nb, and 0.2 curie of gross alpha. Correspondingly, transport by the downstream monitoring station included 14.2 curies of rare earths, 5.5 curies of Ru, 5.0 curies of Sr, 2.2 curies of Cs, 1.7 curies of Co, 1.1 curies of Zr-Nb, and 0.2 curie of gross alpha. These results along with chemical analyses were treated statistically, and it was found that at the 5% level of significance there is no difference between the data at the two stations. Thus, during the sample period there were no detectable quantities of radionuclides or chemical waste contributed to White Oak Creek from Burial Ground 4. (AEC Activity 4452)

Waste Disposal Research and Engineering: Deep Well Disposal. - Continuation of core drilling at the "Four-Acre Site" has now outlined the extent of the grout sheet formed by the first fracturing experiment as follows: along the strike to the southeast the grout was found in a well drilled 275 ft out from the injection well but not in a well drilled 310 ft out. Along the strike to the northwest the grout was found in a well drilled 80 ft out from the injection well but not in a well 128 ft out. Up-dip to the northeast the grout has been found in a well at 345 ft but not in a well at 450 ft; drilling is now in progress at 400 ft. No work has yet been done down-dip, to the southwest. The curtailment of the extension of the grout to the northwest was clearly due to a local zone of tight folding in the shale at the horizon of the grout sheet, which did not break across the steeply dipping beds. The shale of the grout sheet in this area will be explored in more detail.

Some preparatory work has been completed for the second fracturing experiment. (AEC Activity 4452)

Waste Disposal Research and Engineering: Process Waste Water Treatment Plant Studies. - A conventional jar-test apparatus was modified to allow the simultaneous addition of chemical dosages in a study of Sr^{85} removal from water by aluminum phosphate floc. Preliminary results showed that pH varied with $\text{Al}(\text{NO}_3)_3$ and Na_3PO_4 dosages; it was, therefore, necessary to adjust the pH with either NaOH or HNO_3 .

To simulate ORNL Process Waste Treatment Plant conditions, all samples were taken after 2 hr of settling. Additionally, the supernate was clarified by sedimentation, filtration, or centrifugation. In general the removals of strontium by filtration and centrifugation were similar and greater than by sedimentation. Centrifugation gave the most reproducible results.

The best strontium removal, after settling, was found at pH 7. Steadily increasing removal efficiencies were observed up to a pH of 10, with the supernate filtered or centrifuged. Only a slight adsorption of Sr^{85} was found on the glass walls of the beakers, and this occurred only at the higher pH values. Turbidity and total weight of the floc reached the highest values at pH 7.

An increasing strontium removal at higher pH can be explained by assuming an increase in the specific sorption capacity of the floc due to changes in the chemical composition, probably $\text{Al}(\text{OH})_3$ formation. A floc formed by $\text{Al}(\text{NO}_3)_3$ and NaOH gave an optimal strontium removal at pH 10. The analysis of both the remaining liquid and the floc itself showed an increasing consumption of water molecules over the phosphate molecules with increasing pH.

The higher the concentration of chemicals added, the greater was the removal of strontium. An efficiency of 95 to 98% was found with 0.05 N solutions of $\text{Al}(\text{NO}_3)_3$ and Na_3PO_4 . The investigation of chemical dosages [$\text{Na}_3\text{PO}_4/\text{Al}(\text{NO}_3)_3$] in ratios of 1/1, 2/1, and 5/1 gave the best strontium removal at the ratio of 5/1. The settling of aluminum phosphate floc was not rapid, especially at higher pH values, and this is under study at the present time. (AEC Activity 4452)

Waste Disposal Research and Engineering: Clinch River Studies. - Continuous water sampling has been initiated at Clinch River miles 41.5, 14.5, and 3.8. The purpose of the routine sampling is to determine the radionuclides and the percentages carried on the suspended silt vs that carried in solution as a function of distance downstream. Preliminary results indicate that at the mouth of the Clinch River over 40% of the total activity carried downstream is Sr^{90} and that Ru^{106} , Ce^{144} , Cs^{137} , Co^{60} , and Zr^{95} - Nb^{95} are 23, 13, 13, 7, and 3% of the total respectively. Close to 70% of the cesium is attached to the suspended sediment, while only 5, 14, 22, 25, and 29% of Sr^{90} , Ru^{106} , Ce^{144} , Co^{60} , and Zr^{95} - Nb^{95} , respectively, are attached to the sediment. Based on parts per million of suspended sediment, these values agree quite well with the laboratory results. Laboratory tests of a composited Clinch River sediment show that at 30 ppm sediment load, 70% of the cesium and about 5% of the strontium will be associated with the sediment phase. The sediment composition was found to be 60% illite, 15% kaolinite, 10 to 15% vermiculite, and 10 to 15% quartz. The average suspended sediment load in the Clinch River at CRM 41.5 is 27 ppm and varies seasonally with the natural flow in the river, with high suspended sediment loads from November to April. (AEC Activity 4452)

Waste Disposal Research and Engineering: Long-Range Evaluation of Over-All Waste Complex: Release of Radioactivity to ORNL Environs. - A study was made in order to determine the consequences of purposeful or accidental releases of radioactive material to the immediate environs of ORNL. Factual data on the atmosphere, hydrosphere, lithosphere, and biosphere were examined and related to releases of radioactivity which have occurred or which might occur under certain circumstances.

Fallout patterns from stacks or other sources tend to follow the average annual wind pattern, but for estimating consequences of individual major incidents, long-term wind averages are approximate only for relative probability estimates.

There is little probability of ground-water contamination in areas removed from the ORNL site, and there are no developed ground-water sources within the area. The more hazardous fission products, with the possible exception of ruthenium, are adsorbed by the dominantly clay soils of the ORNL area, thus providing a safety factor in the case of accidental releases of radioactive cations. Adsorption, however, does not provide a permanent bond for some materials, for example, strontium, and slow leaching occurs. There are three shortcomings in the natural environment at ORNL: (1) the high rainfall results in leaching of adsorbed activity, (2) due to low soil permeability, surface runoff leads to erosion and sediment transport, and (3) the heterogeneity of ground-water movement patterns makes monitoring difficult.

Insects that emerge from contaminated waters or feed on contaminated plants contribute to the spread of radioactive materials within the area and possibly migrate outside the area. Considerable dilution with distance is involved, since the insects would not be expected to congregate off the area. Birds and animals may spread small amounts of activity over great distances. (AEC Activity 4452)

Waste Disposal Research and Engineering: Long-Range Evaluation of Over-All Waste Complex: Contamination Released to Clinch River by ORNL Waste System. - The various waste disposal operations at ORNL were examined in an attempt to evaluate the release of radionuclides to the Clinch River. Existing data were analyzed and some additional data were obtained for use in the study.

Monitoring of liquids released by White Oak Creek to the Clinch River is done on a routine basis to assure that the concentration of mixed fission products at downstream points of water use does not exceed the MPC values recommended by the NCRP and the ICRP. The monitoring system does not permit a detailed evaluation of the fate of individual radionuclides released by the various disposal operations. The process waste is continuously monitored for gross activity, and the Process Waste Treatment Plant and settling basin effluents are proportionally sampled. Most of the other contributing sources are grab-sampled only, and it is not possible to arrive at accurate figures for the total inflow of particular radionuclides to White Oak Creek.

It is estimated that the liquid-waste pits released 200 curies of Ru¹⁰⁶ to White Oak Creek during 1957 and 160 curies in 1958. Releases of Sr⁹⁰ from the pits are estimated to be less than 1 curie/year. Quantities of various radionuclides released by the Process Waste Treatment Plant to the

creek vary with ORNL operations. For example, in November 1959, approximately 23 curies of Sr^{90} was released, while only 2 curies was released in December 1959. The burial grounds for solid wastes contribute a small but indeterminate quantity of radionuclides to White Oak Creek. The strontium contribution due to leaching of White Oak Lake bed is estimated to be approximately 5 curies/year and is decreasing with time. (AEC Activity 4452)

Waste Treatment and Disposal. - In small-scale batch evaporation and calcination of synthetic waste in glass equipment, 70 to 80% of the ruthenium was found in the condensate and additional ruthenium plated out on the walls of the flask and condenser. Neither sodium and calcium nor magnesium, added to decrease sulfate volatility, had much effect on these results. With a nitric oxide sweep through the system, the ruthenium volatility decreased to about 1% and no plating-out was visible in the absence of additives; in the presence of additives, the ruthenium volatility increased to 2 to 30%, with magnesium having less effect than sodium and calcium.

In two semicontinuous calcination runs in a 4 x 18 in. stainless steel pot in a closed system with nitric oxide fed to the pot, the net noncondensable gas production was negligible. In a previous run without nitric oxide, the net gas production was about 5 liters of noncondensable gas, mostly oxygen, per liter of Purex feed.

Data from two runs in 6- and 8-in.-dia stainless steel vessels, 84 in. long, indicated that the mechanism of solid deposition is radial. Changing the vessel diameter from 6 to 8 in. did not affect the characteristics of the solid and confirmed that the maximum processing rate of 20 liters/hr, averaged over the feeding period, is independent of vessel radius, as predicted by the radial deposition model.

Duolite C-3, a phenolic-sulfonic resin, and Duolite CS-100, a phenolic-carboxylic resin, both removed 99.9% of the cesium and strontium from ORNL process water waste made 0.01 M in added NaOH. About 1500 resin-bed volumes of waste were processed in a three-day loading cycle. The strontium breakthrough capacity was about 50% greater than the cesium. More than 99.9% of the cesium and strontium were removed from the resin by upflow regeneration with ten bed volumes of HCl solution, 5 M for C-3 resin and 1 M for CS-100. The CS-100 resin has the disadvantage of changing bed volume considerably between acid and basic solutions. (AEC Activity 4452)

PROGRAM 5000 - PHYSICAL RESEARCH

ISOTOPE PRODUCTION

Radioisotope and Stable Isotope Production. - Charge materials were prepared for the current separations of the isotopes of tantalum, osmium, and chromium in Building 9731 and of lead and calcium in Building 9204-3. Charge materials were synthesized or prepared from commercial products for forthcoming collections of the isotopes of tellurium and molybdenum. The collections of the isotopes of barium and chromium were completed with ion currents equivalent to 356 g. Collections of the isotopes of lead, osmium, tantalum, and calcium are now in process.

Twenty-three new lots of calcium, indium, germanium, and zinc and five lots of isotopes returned from loans were prepared for inventory.

Seventy-four samples were recovered on a preliminary basis for retention and/or assay information. In this group of samples were three preliminary samples of Os^{198} and three samples of Os^{192} , which are being collected in the ORNL separators for the first time.

Conversions of inventory forms to more suitable forms included: Ni^{61} and Ni^{64}O to NiSO_4 , B^{10} element to B_2O_3 , B^{10} element to H_3BO_3 , Li^6 metal to LiF , and Hg^{198} , Hg^{199} , and Hg^{200} from aqueous nitrates to Hg metal. Eight samples of isotopic HgNO_3 in aqueous solution were encapsulated in Pyrex ampoules. Two samples of Hg^{196} , one sample of Hg^{204} , and six samples of $\text{Ca}^{46}\text{CO}_3$ were encapsulated in quartz containers for customer use.

Target preparations included 22 conversions and 35 targets. A method for the silver-plating of tantalum filaments was developed, and a number of such silver-plated filaments were prepared for the cyclotron operations group.

The large lithium shield for the Tower Shielding Facility was completed during this period. Four single crystals of copper with 110 flats were obtained for use in studies of increased retention of Ca^{46} isotopes.

Modifications associated with the program to reduce radiation in the radioisotope packaging area include rerouting of an overhead monorail hoist; addition of 1-in. steel scatter shielding suspended over the storage barricade wall; glass viewing windows installed on top of the barricade wall; fabrication and installation of new storage transfer manipulators and new packing manipulators; relocation of lighting for the storage barricade and the electrical panel boards; and installation of a new shielded conveyor system.

Radioisotope Research and Development. - Decalso (an aluminosilicate ion exchanger) was found to be capable of adsorbing Cs^{137} from 7.8 column volumes of 8 M Na^+ aqueous solution before 50% breakthrough. Waste of this type, containing 15 curies of Cs^{137} per gallon, is available in storage tanks at Hanford. Design is in progress to convert a 500-gal-capacity shielded transfer tank to serve as a container for a bed of Decalso on which about 30,000 curies of Cs^{137} can be adsorbed from the Hanford solution.

The difficulty in dissolving rare-earth double sulfates which had been heated to 425°C was overcome by mixing 14.3 g of fine copper powder with each 100 g of rare-earth salt prior to filtration, so that the copper became

part of the filter cake during the filtration cycle. The gases evolved from the copper during the dissolution of the cake in 4 M HNO_3 have the effect of forcing the particles of the cake apart so that HNO_3 can contact the interior of the cake.

The test thermal diffusion columns for C^{13} enrichment have been completed and are being tested with a methane system. (AEC Activity 5100)

Stable Isotope Development. - A second-pass separation of Pb^{207} from 4.5 g of lead yielded 200 mg of product containing 99.94% Pb^{207} . The product and the operating parameters achieved here verify the feasibility of applying second-pass techniques to secure high-purity samples of all lead isotopes.

Retention of separated Pb^{206} and Pb^{208} from enriched feeds has been markedly improved through the use of a collector pocket design which intentionally provides both a sputtering surface and a large area on which the sputtered material can condense. In the ion sputtering program, additional data has been accumulated by analysis of pockets from the production runs in Building 9204-3. (AEC Activity 5100)

PHYSICS

Scintillation Spectrometry and Instrument Development. - Equipment to observe the Mossbauer effect with the 70-keV gamma ray from Ni^{61*} has been developed, and the testing of the apparatus is essentially complete. The preparation of the source required development of a technique for electroplating small quantities of a rare nickel isotope, Ni^{64} , on aluminum. The Ni^{61*} will be obtained by the reactions $\text{Ni}^{64}(\text{p},\alpha)\text{Co}^{61}(\beta^-, 90 \text{ min})\text{Ni}^{61*}$. The gamma energy, Debye temperature, and mass of Ni^{61} require low temperature to carry out the measurements. The source and the absorber will therefore be cooled to liquid-nitrogen temperature. To measure the velocity dependence of the resonance absorption a device was built to move the absorber foil with constant acceleration. The velocity range is divided into nine equal intervals. The gamma rates in the velocity intervals are recorded in nine independent scalar units. Test measurements with a Ba^{133} source are in progress to determine the characteristics of the equipment. Measurements on the Ni^{61*} source will begin in the near future. (AEC Activity 5220)

High-Voltage Experimental Program. - A shell-model calculation of the stripping reaction and an analysis of a resonance found in the excitation function of the $\text{F}^{19}(\text{d},\text{p})\text{F}^{20}$ reaction have been made under simplifying assumptions. It was found that the ground-state spin of 2^+ , derived from beta-decay evidence, is consistent with reaction data and that the Schwartz set for spin resulting from the coupling of $J_{3n} = 5/2^+$ and $J_p = 1/2^+$ is reasonable. Spin values have been temporarily assigned to a few other levels in F^{20} .

The yields of gamma rays resulting from Coulomb excitation of states in Th^{232} and U^{238} with protons of 4 to 6 MeV energy have been measured. Gamma rays of 790, 740, and 613 keV energy from Th^{232} and of 1.02 MeV energy from U^{238} are observed. For Th^{232} , the variation in gamma-ray yields

with proton energy, the results from gamma-ray angular distributions, and the linear polarization measurements are consistent with the direct excitation of a 2^+ state at 790 keV which decays by means of E2 radiation to the $I = 0^+$ and 2^+ members of the ground-state rotational band. In view of the recent results from measurements of internal-conversion electrons made at Rice Institute, the 613-keV gamma ray is interpreted to be a transition from a 2^+ β -vibrational state at 773 keV to the 4^+ state at 163 keV. An interpretation of the observed gamma-ray yields is carried out by taking a γ -vibrational state at 790 keV and a β -vibrational state at 773 keV. The $B(E2)_d$ of the 2^+ state at 50 keV in Th^{232} is about 150 times the single-particle estimate. The $B(E2)_d$ of the vibrational states in Th^{232} and U^{238} are between 2 and 3 times the single-particle estimate. By combining Bernstein's relative internal-conversion electron yields with the gamma-ray data obtained here, a value of $(8 \pm 2) \times 10^{-2}$ for the strength parameter ρ is obtained for the E0 transition between the 2^+ state at 773 keV and the 2^+ state at 50 keV in Th^{232} . (AEC Activity 5220)

Heavy-Particle Physics. - The differential cross section for the reaction $\text{N}^{14} + \text{C}^{12} \rightarrow \text{B}^{10} + \text{O}^{16}$ was measured at several angles. It was found to be very small (~ 0.1 mb/steradian) and did not exhibit any marked structure. A new search is in progress to measure the cross section for the reaction $\text{N}^{14} + \text{N}^{14} \rightarrow \text{C}^{12} + \text{O}^{16}$. With a gas target and silicon barrier counters, this reaction can be detected if its cross section is of the order of 0.2 mb/steradian. The investigation of the reaction $\text{N}^{14} + \text{N}^{14} \rightarrow \text{N}^{13} + \text{N}^{15}$ at 24 MeV incident energy indicates that the excited states in N^{15} contribute more at lower bombarding energies. (AEC Activity 5220)

The 86-Inch Cyclotron: Nuclear Physics. - The decay of Lu^{171} was earlier shown to feed a $K = 1/2$ ground-state rotational sequence to $I = 9/2$ [Phys. Rev. 114, 1082 (1959)]. Another rotational band is possibly based on a $(5/2^- [512])$ level at 122 keV with rotational excitations of 85 and 109 keV in cascade and an E2 crossover of 195 keV. The rotational sequence based at 122 keV may be fed by transitions from an intrinsic state at 836 keV ($7/2^- [514]$) and its rotational level ($I = 9/2$) at 950 keV. A pair of levels at 95 and 167 keV are interpreted as the expected ($7/2^+ [633]$) state and its associated rotational level ($I = 9/2$) respectively. The orbital ($9/2^+ [624]$) is provisionally assigned to a 936-keV state. (AEC Activity 5220)

The 86-Inch Cyclotron: Applied Physics. - The isotopes Co^{57} , As^{74} , Sr^{85} , Y^{88} , Tc^{95} , Rh^{102} , Ce^{139} , Eu^{149} , and Bi^{207} were produced as service irradiations for laboratories in the United States, India, and the Netherlands for a total of 93 hr bombarding time. The average yield of As^{74} per hour was increased 41% above the 1959 average yield by varying the auxiliary field periodically during the bombardment. Concomitantly with this increase in yield, the germanium powder which is used as the target material does not display the effects of sintering; chemical dissolution is thus enhanced manyfold. An increase of 13% was similarly obtained in the yield per hour of bombardment in the production of Sr^{85} . A program was inaugurated in the study of (p,γ) excitation functions with the bombardment of iron. (AEC Activity 5220)

Electronuclear Machines. - The Cyclotron Analogue II, an eight-sector spiral-pole electron cyclotron, is being built to model the beam dynamics of a proposed 850-Mev proton AVF cyclotron. Magnetic-field calculations for the sector coils are now essentially complete. Numerically controlled machine methods were used in fabricating a prototype sector coil; the three turns of copper were machined and embedded in a machined aluminum plate, which serves as a heat sink. Information gained from the fabrication of the prototype sector coil was incorporated in the final design, and the purchase order for the sector coil assemblies was placed. The r-f system was completed and is undergoing test for adjustment of the regulator. The vacuum tank was received, and the system is being assembled. (AEC Activity 5240)

Relativistic Isochronous Cyclotron (ORIC). - Substantial progress was made on the construction of ORIC Building 6000 and on the design and fabrication of major cyclotron components. Although the building is still a few weeks behind schedule, the work is now moving along rapidly. The main shielding doors were received and are being installed. The massive walls of the experimental area are nearly finished. It is estimated that the entire building is about 40% complete.

Fabrication contracts have been let for many major cyclotron components, including 19 rectifier-type power supplies, a 650-kw r-f supply, magnet pole tips, 50-in.-dia dee stems and a 75-in.-dia dee-stem housing of copper-clad mild steel, and the crane system for transfer of the resonant structure.

The magnetic-field configuration has been studied in forty 2400-point runs. Programs for calculating particle phase slip are now being used successfully. The final optimization is now being completed.

The precision 80-in. gear for the full-scale magnetic-field survey was completed; the maximum cumulative error in spacing the 180 position slots at the periphery is less than 5" of arc.

Special Separations. - The Pu²⁴⁰ isotope from series P2S has been completed and shipped; also, several returned plutonium samples have been processed. Apparatus for a separation of uranium from neptunium has been assembled, and processing of approximately 30 g of neptunium is contemplated.

Ninety-two grams of the special-target U²³⁵ run has been recovered from an initial 100 g. A physical inventory of the various uranium and thorium batches on hand has been taken, and inventory adjustments were made on all levels except the thorium and 500 uranium levels. The 500-level adjustment is waiting recovery of carbon ash from the last U²³⁸ series.

Several batches of PbCl₂ charge material for Pb²⁰⁶ and Pb²⁰⁸ runs have been prepared from radiogenic ores. About 30 lb of lead carbonate enriched in Pb²⁰⁸ and about 10 lb of lead carbonate enriched in Pb²⁰⁶ were processed.

The following shipments were made: uranium, 9; neptunium, 4; plutonium, 3; and thorium, 2.

The engineering for the proposed plutonium area in Building 9204-3 has been transferred to the Catalytic Construction Company, with title I engineering scheduled for completion in August and title II scheduled for completion in October. (AEC Activity 5250)

CHEMISTRY

Surface Chemistry. - A sample of ThO_2 was prepared which should be satisfactory for planned ionic absorption studies provided that most of the particles turn out to be single crystals rather than agglomerates of crystals. If this is so, essentially all the available surface should be on the surface of the particles, facilitating interpretation of absorption data. The starting material was a ThO_2 sample with an apparent average particle diameter of 1.0μ and an average crystallite diameter of 0.09μ . Digestion for several days in $\sim 0.01 \text{ M HNO}_3$ at a temperature somewhat below boiling resulted in considerable particle degradation. By successive centrifugations a fraction was obtained which had settling rates about the same as spheres between 0.2 and 0.3μ in diameter. (AEC Activity 5310)

Chemical Engineering Research. - In a stacked clone contactor the equilibrium benzoic acid concentrations in the aqueous and organic phases of the system Amsco- 0.08 M HNO_3 -benzoic acid were found to be represented by the polynomial $Y = 1.866 \times 10^{-2}X^3 + 1.295 \times 10^{-3}X^4 + 1.906 \times 10^{-5}X^5 + 4.101 \times 10^{-7}X^6$, where Y = millimoles of benzoic acid per liter of Amsco phase and X = millimoles of benzoic acid per liter of aqueous phase. Data for the system Amsco- 0.08 M HNO_3 with five stages plus a top de-entrainer showed organic-phase retention times per mechanical stage ranging from 5 sec for flow rates of 84 cc/min aqueous and 450 cc/min organic to 21 sec for flow rates of 1580 cc/min aqueous and 200 cc/min organic. The absolute rate of entrainment of the organic phase with the aqueous (cc/min of organic entrained) increased approximately as the square of the aqueous flow rate when no provision for phase separation was made below the organic feed point. However, with one or more clone polisher stages (identical with an extraction stage) below the feed point the organic entrainment could be decreased as much as desired. A single polisher stage reduced the entrainment by about one order of magnitude.

A comprehensive review of the literature on the resistance to mass transfer between fluid phases indicated that all the resistance to transfer of inorganic solutes can be accounted for by hydrodynamic phenomena coupled with molecular diffusion of the solute in one or both phases. (AEC Activity 5310)

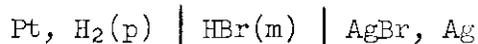
Thorium Oxide. - The final surface areas of a series of thorium oxide powders irradiated for 21-3/4 months in an LITR lattice position (flux = 2×10^{13} neutrons $\cdot\text{cm}^{-2}\cdot\text{sec}^{-1}$) were reduced from as much as $30 \text{ m}^2/\text{g}$ to $\leq 0.5 \text{ m}^2/\text{g}$ for materials fired at 650 to 1100°C . The surface area of a 1500°C -fired material ($0.8 \text{ m}^2/\text{g}$) was not changed by the irradiation. As with a previous series of powders irradiated for 16 months, irradiation in the reactor appeared to produce a sintering effect equivalent to that obtained in a firing of the oxide at 1500°C .

A summary of the results of more than 200 gas recombination experiments carried out with the sol-prepared palladium catalyst in thoria and thoria-urania slurries shows that specific catalyst activities are independent of the nature and concentration of the slurry solids. Specific activities are 30- to 40-fold higher when the hydrogen is slightly in excess of the stoichiometric ratio (H_2 to $1/2 \text{ O}_2$) than under slight excesses of

oxygen, and are low or negligible when a large excess of either oxygen or hydrogen is present. There is a threshold pressure of 50 to 200 psi for reaction for the gas which is not in excess, depending on the pressure of the other gas. The dependence of the over-all apparent reaction rate on the total gas pressure varies from zero to second order, indicative of a rather complex kinetic mechanism for the catalyzed recombination reaction. Occasionally the recombination of the hydrogen and oxygen is only partial, terminating in an equilibrium condition or an extremely slow recombination rate. There is an induction period for recombination of 5 to 25 min after oxygen pretreatment, depending on the total gas pressure and the hydrogen-to-oxygen ratio. (AEC Activity 5310)

Equipment Decontamination. - Cerium was found to be somewhat similar to ruthenium in behavior as a radioactive contaminant. When deposited onto steel from solution, it was removed by factors of 10^3 to 10^4 by boiling for 1 hr with dilute acids. When baked onto the steel in air at about 300°C , however, decontamination dropped by a factor of 100. A preliminary boiling with alkaline permanganate restored the decontaminability by acids. (AEC Activity 5310)

Inorganic and Physical Chemistry: Inorganic Solution Chemistry. - The emf of the cell



was measured from 25 to 200°C with HBr concentrations from 0.005 to 0.5 m and also to 150°C at 1.0 m. This allowed the determination of the HBr activity coefficients over the range 25 to 200° and 0.005 to 1.0 m.

Studies over the range 25 to 55°C with an Ag-AgCl reference electrode showed that the emf of the quinhydrone electrode is sufficiently reproducible to be used for activity coefficient measurements. (AEC Activity 5330)

Organic Chemistry. - In continuing the study of solvents of active interest in solvent extraction, thermally induced reactions of tributyl phosphate were examined. The reactions are catalyzed by the acid products. Up to 3% reaction, acid formation obeys first-order kinetics, the specific reaction rates being $0.39 \times 10^{-7} \text{ sec}^{-1}$ at 178°C and $132 \times 10^{-7} \text{ sec}^{-1}$ at 240°C . In addition to the large yields of dibutylphosphoric acid and 1-butene, competing reactions produce small yields of butanol, dibutyl ether, and tetrabutyl pyrophosphate. The butyl esters of butylphosphonic and dibutylphosphinic acids are initially more stable than tributyl phosphate. (AEC Activity 5330)

Radiation Chemistry. - The pressed alkali halide disk method for the infrared identification and quantitative determination of organic and inorganic solids is being used as a tool for study of chemical reactions induced by the transfer of irradiation energy from alkali halides to various other substances. Studies were initiated on the effects of altering the natures of the absorber (matrix) and receiver (foreign ion or molecule) on this energy transfer.

Dibasic acids, $\text{HOOC}-(\text{CH}_2)_n-\text{COOH}$, were used as chemical indicators to study the rates of energy transfer from potassium bromide as a function of n . With a molar ratio of acid to halide of 0.001, the rate of the zero-order disappearance of the dibasic acid (as indicated by the decrease in

the characteristic absorption of the carboxyl group) appeared to be independent of chain length beyond adipic acid. The relative decarboxylation yields for $n = 1, 2, 3, 4,$ and 8 were 1.0, 0.64, 1.64, 1.10, and 1.10 molecules per 100 ev.

To investigate the effect of variation of the absorber (matrix) on the rate of energy transfer, a number of alkali halides containing nitrate ion as an impurity were irradiated, and the decomposition of nitrate ion and the formation of nitrite ion were followed. In the series of halides in which the halogen was varied, the initial yield of nitrite increased with increasing size of the anion: KF, 0.091; KCl, 0.100; KBr, 0.238; and KI, 0.700 (in molecules per 100 ev).

Experimental evidence was obtained to support the view that the alpha-particle radiolysis of CO proceeds through ionic and excited species. The active ionic species, CO^+ , is deactivated by charge transfer to the product CO_2 . There is no evident depletion of the excited species, CO^* . (AEC Activity 5330)

Chemical Physics: Microwave and Radio-Frequency Spectroscopy. - Single crystals of sodium nitrite containing silver nitrite have been gamma-irradiated at 77°K , and the paramagnetic resonance spectrum has been examined. A single group of three sharp, anisotropic lines originating from an N^{14} hyperfine interaction was found. The paramagnetic species in the crystal must have C_{2v} symmetry, and calculations are being made on the basis that it is NO_2 . (AEC Activity 5330)

Chemical Separation of Isotopes. - Vapor pressures were measured for several solutions of BF_3 in phenol over the temperature range from -10 to $+40^\circ\text{C}$ and for BF_3 in dimethyl selenide from -30 to $+30^\circ\text{C}$. The vapor pressure data were used to calculate heats of reaction for these mixtures.

A half time of 420 days at 35°C was observed for the NH_3 exchange between $\text{Co}(\text{NH}_3)_6\text{Cl}_3$ and $\text{N}^{15}\text{H}_4\text{OH}$ in aqueous solution. (AEC Activity 5340)

Chemical Applications of Nuclear Explosions. - The rate of exchange of tritium between THO and HT over CaSO_4 increased during the first 50 min according to the parabolic law, indicating that bed diffusion controlled the rate. The exchange rate increased with temperature from 0.2% at 380°C to 9.2% at 700°C .

Passing powdered $\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$ through a plasma jet in argon ($\sim 10,000^\circ\text{F}$) gave a product corresponding in alkalinity to CaO , indicating essentially complete dissociation of the salt. (AEC Activity 5350)

Transuranic Studies. - In further work on anion exchange procedures for separating actinides and lanthanides, sorption of actinides and lanthanides on anion exchange resin from 2.5 M $\text{Al}(\text{NO}_3)_3$ was found to be as satisfactory as sorption from 7 or 8 M LiNO_3 . Increasing the acidity of 2.5 M $\text{Al}(\text{NO}_3)_3$ from 0 to 0.3 M HNO_3 had little effect on sorption, but at higher acid concentrations sorption decreased. Curium tracer and micro amounts of rare earths were satisfactorily separated on Dowex 21-K (50 to 100 mesh) resin, which is more stable to radiation than Dowex 1 resin, used previously. When 10 g of curium and rare earths per liter of resin was sorbed on a 63-cm column of Dowex 21-K (50 to 100 mesh) resin from 2.5 M $\text{Al}(\text{NO}_3)_3$ at 80°C , the curium was eluted by two column volumes of 1 M LiCl . The product contained

99.7% of the curium and 0.7% of the rare earths. The rare earths were completely eluted by ten column volumes of 10 M LiCl. Attempts to use coarse Dowex 21-K resin were not successful. When 20 to 60 mesh resin was used only 60 to 70% of the product was free of rare earths. (AEC Activity 5360)

Analytical Chemistry Research. - A method has been developed for the determination of trace concentrations of helium in neutron-irradiated beryllium oxide. The helium is derived from the reaction $\text{Li}^6(n,\alpha)\text{H}^3$ with lithium present as a contaminant in reactor-grade beryllium oxide. The determination is carried out by dissolving an irradiated specimen of the beryllium oxide, amounting to about 14 g, in a 350-g melt of potassium acid fluoride (approximate composition, $\text{KF} \cdot 3\text{HF}$) contained in an evacuated silver-lined reactor. After a reaction period of 24 hr at 120°C, the reactor is cooled to the temperature of liquid nitrogen, and the noncondensable gases are then pumped into a 1-cc transfer vessel. Hydrogen, which interferes if present in sufficient quantity, is removed by passing the gas over heated copper oxide, after which the helium is isolated and determined chromatographically on an 8-ft by 0.3-in. column filled with type 5A molecular sieve, with nitrogen as the carrier gas. Helium in concentrations of 3 ppm has been determined; the method is, however, suitable for the measurement of less than 0.1 ppm. The water formed in the reaction with copper oxide is trapped and used for the determination of tritium by a counting technique. The separation of water from the melt for tritium counting is also being investigated. (AEC Activity 040104091)

METALLURGY AND SOLID STATE PHYSICS

Fundamental Physicometallurgical Research. - Experiments with calorimetry at liquid-helium temperature have shown that dissolved silver increases the density of electron states and reduces the Debye temperature of hexagonal zirconium. The increases in the density of states have now been observed for each of the solutes silver, indium, and tin. The size of the effect increases in that order. This situation is consistent with the observed axial ratios of hexagonal zirconium, since an increase in axial ratio is observed with these solutes and with cadmium, and is believed to be related to an overlap of the d electrons in the Brillouin zones. The increase in the density of states would be especially strong when the Fermi surface rises in alloying, as for the solutes indium and tin. For the solute silver, the axial ratio increase would produce a downward shift of the electron concentration at which the overlap begins which is faster than the lowering of the Fermi surface produced by silver additions.

Twinning of electron-beam-melted vanadium has been observed during slow compression at liquid-nitrogen temperature. Single crystals were cut from an ingot having the following interstitials content: 0.033% C, 0.0047% O₂, 0.0005% N₂, and 0.0012% H₂. The oxygen and nitrogen concentrations were an order of magnitude lower than in the material studied by others. The twins were similar in appearance to those found in the other body-centered cubic metals - thin with serrated boundaries. The twin plane was found to be {112}, as expected. Studies here have now shown that all of the group VA

body-centered cubic metals deform by twinning with relative ease below room temperature if they are very pure and have a large grain size. (AEC Activity 5420)

THERMONUCLEAR PROJECT

Arc Research. - Spectroscopic studies of the carbon arc in strong magnetic fields have been extended to include the evaluation of line profiles. The random energy or "temperature" of the carbon ions increases rapidly with increasing distance from the anode. Since the half width of the line increases linearly with this distance, the mean ion energy increases quadratically. Quantitative Doppler profile measurements for both axial and transverse arc views of the line group at 4650 Å give temperatures of about 800,000°K (mean ion energy > 100 ev) for C⁺⁺ ions at a distance of 4 ft from the anode under appropriate conditions of background pressure and arc current. The line intensity profile is closely Gaussian (temperature profile characteristic) to within a few per cent and down to about 5% of the peak intensity. Other lines of C⁺⁺ and of C⁺⁺⁺ give qualitative agreement with the profile interpretation for the 4650-Å group. Lines of C⁺ are weak except near the electrodes, where marked line broadening also occurs. The Doppler "slant" effect, which is prominent in the anode region, persists with about the same magnitude in this region of high ion temperature, indicating that the magnitude of the azimuthal drift velocity of ions about the arc core does not vary greatly along the arc length. This suggests that the radial electric field (~ 100 v/cm), thought to be responsible for this azimuthal precessional drift, is sensibly constant along the length of the carbon arc. (AEC Activity 5540)

DCX-1 Facility. - The water-cooled baffles previously installed on the 32-in. diffusion pumps on the anode side of the machine seem to have reduced the oil back-streaming there to tolerable limits.

Experiments with the conventional two-electrode deuterium arc have continued. Gage pressures of approximately 8×10^{-6} mm Hg in the liner region and 6×10^{-5} mm Hg in the outer region have been obtained with anode gas flow rates of 4 to 5 atmospheric cc/sec and with stable arc operation. At lower gas flows the arc voltages rise and the more powerful arcs rapidly burn through the anode structure. Operation with various anode cooling geometries has not resulted in significant improvements.

A reflecting (i.e., three-electrode) deuterium arc has been installed and operated briefly. These preliminary runs indicated that additional cooling is required at the reflector electrode. (AEC Activity 5540)

Larger Magnetic Facility. - Preliminary design work leading to a good engineering cost estimate has been completed. Design work on most of the equipment is continuing. Magnetic tests on a scale-model injection snout are in progress. (AEC Activity 5540)

Vacuum. - Hydrogen pumping by titanium gettering in a system similar to the DCX, but with a liner of 4000 cm² surface area (1/6 the surface area of the liner in the DCX), has resulted in base pressures of 7×10^{-10} mm Hg

and pumping speeds up to 80,000 liters/sec. This is a sticking probability of about 1/2. Previously reported sticking probabilities have been 1/20. The present technique differs from the former in that the port between the inner and outer systems is trapped with X-13 (Zeolite), and the whole system was baked at a temperature in excess of 500°C for 24 hr. The titanium deposited was greatly (500 times) in excess of that required to remove the hydrogen on a molecule-for-molecule basis.

Preliminary measurements of the pumping speeds of evaporated titanium films for several gases in an unbaked system have yielded speeds as shown below:

Gas	Pumping Speed (liters/sec)		Per Cent of Theoretical Limit	Pressure (mm Hg)
	Total	Per Square Centimeter		
H ₂	50,000	1.7	3.8	1×10^{-6}
N ₂	200,000	6.7	56	2.7×10^{-7}
O ₂	24,300	0.8	7.4	7.1×10^{-7}
CO ₂	746,000	4.9	56	4.8×10^{-7}
CH ₄	None			

Indications are that pumping speeds for H₂ increase as increasing amounts of titanium are evaporated. Present evaporation rates are between 1.0×10^{-4} and 1.5×10^{-4} g·cm⁻²·hr⁻¹. Titanium alloy filaments show promise of increasing this rate by a factor of 5 or more.

Equipment for the systematic study of evaporated metal film pumping systems has been designed and is under construction.

Measurements of permeability to helium were made on Coors AD85 and on special glazed and unglazed porcelain of interest as electrical insulators. (AEC Activity 5540)

PROGRAM 6000 -- BIOLOGY AND MEDICINE

BIOPHYSICS

Internal Dosimetry. -- To check the initial high excretion rate of P³² in persons accidentally exposed to neutrons, a pilot study in which 100 mice were exposed to an average total flux of 1.8×10^{10} thermal neutrons/cm² has been completed. In this exposure P³² is formed in the body of the mouse, according to the reaction $P^{31}(n,\gamma)P^{32}$.

The urine and feces were separated, and the urine was analyzed for P³² and stable phosphorus. During the first 24 hr the excreta were collected at the end of each 4-hr period, but for the remainder of the experiment the

samples were collected once every 24 hr. The experiment was terminated when the activity reached background level. The results given below show that the initial excretion rate was rapid:

<u>Time</u>	<u>Radioactivity in Urine (disintegrations·min⁻¹·ml⁻¹)</u>
4 hr	2021
8 hr	1600
12 hr	1160
16 hr	1033
2 days	408
4 days	195
8 days	134
13 days	63
19 days	45

(AEC Activity 6440)

Waste Disposal Research and Engineering: Evaluation of Soil Disposal. -- Several commercially available grades of vermiculite, obtained from the Zonolite Company, Traveler's Rest, South Carolina, were evaluated for use in mineral-filled columns for the decontamination of the ORNL intermediate-level waste stream. The solution employed had the same stable-element composition as the present intermediate-level waste stream and contained the mass equivalent of $\sim 2 \mu\text{c}$ of Cs^{137} per milliliter. A flow rate of $1 \text{ ml}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$ was maintained. In the crude ore series there was a marked dependence of cesium selectivity (k_d) on particle size. The k_d varied from 9.9 ml/g for the largest particle size to 39.3 ml/g for the smallest particle size. Treatment of the crude ore with potassium to induce lattice collapse improved k_d by a factor of 2.5, and heat treatment, used to cause explosion or exfoliation of the vermiculite, resulted in a 20-fold increase in k_d . (AEC Activity 6440)

ENVIRONMENTAL SCIENCES

Ecological Studies. -- A coherent but simplified mathematical model of the circulation of carbon, mineral nutrient elements, and radioactive isotopes in ecological systems was simulated by small and large analog computers (Donner 3400; ORNL Reactor Controls Analog Facility, RCAF-X-10). The quantity of matter in each compartment of the model (e.g., above-ground vegetation, roots, dead organic litter, and soil) was simulated by voltage stored in an operational amplifier (integrator). The input to the system (e.g., photosynthetic production of carbon) and the rates of loss from one compartment and transfer to other compartments were adjusted by potentiometers. The effects of various steady or changing values of ecological

factors on the storage, time parameters, and oscillations of the system were measured on chart recorders.

Differences in the rate of transfer of an element out of a given compartment accounted for manyfold differences in the equilibrium level of the element accumulated in that compartment and in the time parameters describing the rapid or slow approach to this equilibrium level. The model and results provide many analogies to solutions of the Bateman equations for accumulation and decay of daughter products in a chain of radioactive decay and to linear lags in various control systems. The feedback of material from soil to plants is illustrated for the case of C^{14} and would be very important for the cycling of radioactive isotopes such as Sr^{90} and Cs^{137} which arise from environmental contamination by waste disposal and fallout. (AEC Activity 6480)

PROGRAM 8000 - WORK FOR OTHERS

Fission Product Recoveries. - Continued laboratory-scale studies of the proposed solvent extraction process for the recovery of strontium and rare earths from simulated Purex waste showed that the rare-earth product from two cycles of extraction with di(2-ethylhexyl)phosphoric acid and tributyl phosphate in Amsco 125-82 contains ~ 27 g of rare earths, ~ 0.06 g of Na^+ , and ~ 1.25 moles of HNO_3 per liter. This product represents a rare-earth concentration factor of about 20 and over-all separation factors from strontium and sodium of about 2.5×10^4 and 4.5×10^3 respectively. The strontium product from the fourth extraction cycle contains ~ 12 g of strontium, ~ 2 g of Na^+ , and ~ 1.3 moles of HNO_3 per liter for a concentration factor of ~ 70 , representing over-all separation factors from rare earths and sodium of $\sim 2.5 \times 10^3$ and $\sim 6 \times 10^2$ respectively. Both products are in nitrate solution and readily amenable to additional purification.

Two solvents, sodium tetraphenylboron in hexone and dinonylnaphthalenesulfonic acid in Amsco 125-82, extracted cesium from Purex waste solutions. Thus far, however, no acceptable means has been found to strip cesium from sodium tetraphenylboron without destroying its extractive affinity for cesium. Extraction of cesium from adjusted Purex waste with dinonylnaphthalenesulfonic acid in Amsco 125-82 was comparatively weak, but process application of this reagent may be possible if future tests show high selectivity for cesium over other waste components. (AEC Activity 8632)

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OAK RIDGE NATIONAL LABORATORY

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