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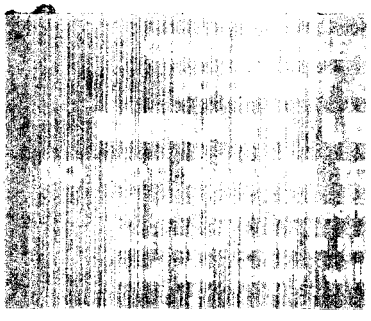
OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

OCTOBER 1960



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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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U.S. ATOMIC ENERGY COMMISSION

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MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

October 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.

SPECIAL NUCLEAR MATERIALS PROGRAM

Dissolver Solution Analyses. — Work to increase the sensitivity of the controlled-potential coulometric determination of uranium so that accurate results may be obtained in the microgram range is being continued. No definite results or conclusions have been obtained, but preliminary results indicate that uranium can be determined to within ±2% in samples containing 100 μg.

A method by which uranium may be determined coulometrically in solutions containing molybdenum was described in the status and progress report of August 1960 (ORNL-3009). Work on this problem has been essentially completed. On the basis of precision studies, relative standard deviations of 0.72% for samples containing 2.37 mg of U and 0.25 mg of Mo, and of 0.40% for 4.73 mg of U and 0.5 mg of Mo were found. Also, Fe, Cu, Cr, Ni, Ru, and NO_3 were studied as possible interferences in this method when present as contaminants in the sample. Of these materials, only copper was found to be a definite interference. The others do not interfere or can be eliminated by treatment of the sample prior to the uranium titration.

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

Pebble-Bed Reactor Experiment. — A preliminary design study has been completed for a pebble-bed type of gas-cooled reactor experiment which would operate at a thermal power level of 10 Mw and would provide sufficient data for the design of a full-scale pebble-bed power reactor.

The core of the experimental reactor is 8.1 ft long and 18 in. in diameter and is surrounded on all sides by a 3-ft-thick graphite reflector. The fuel consists of 1-1/2-in.-diam balls composed of 90.0 wt % graphite, 5.49 wt % UO_2, and 4.51 wt % ThO_2. The uranium is fully enriched. The core has 39% of its volume as space for the coolant gas (helium). It is intended that the fuel residence time be 246 days, which will give a uranium burnup of 10%. These conditions will subject a fuel ball to the same

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temperature, power density, and burnup as would be experienced in a full-scale power reactor. An unusual feature of the reactor design is the use of stainless steel control blades placed circumferentially in the reflector. Since the reactor is mainly reflector-moderated, this location provides effective control without interference with the fuel balls in the core.

Calculations performed for the 10-Mw test reactor design study included the critical fuel concentration, neutron fluxes as a function of energy and position throughout the reactor (needed for radiation-damage estimates), control blade worth as a function of depth of insertion, power density distributions for several control blade positions, neutron and gamma-ray heating in the reflector-moderator and in the pressure vessel, temperature coefficients of reactivity, and prompt-neutron lifetime.

GCR-ORR Loop No. 2. — Installation of lead shielding adjacent to the south facility plug at the ORR was completed during the shutdown period of September 25 to October 3, 1960. Completion of this shielding reduced the radiation levels in this area so that containment cell installation by Ferguson Construction Company could be started October 4 as planned. The contractor has now installed the steel support structure for the 14-in.-thick lead shielding beneath the cryostat room. Portions of the forms for the concrete walls are also installed.

Tests of UC_2-Graphite Fuel Elements (ORNL-MTR-48). — The test period of the ORNL-MTR-48-2 experiment has been further extended from seven to nine reactor cycles and is scheduled to terminate December 26, 1960. This will represent a U^{235} burnup of 27% based on an approximate rate of burnup of 3% per MTR cycle.

The helium purge flow could no longer be maintained after the first week of the fifth reactor cycle. The purge gas activity level was affected by the attempts to maintain the gas flow after the third reactor cycle. The activity level before termination of the purge gas flow appeared to be in the range of 100,000 ± 20,000 counts/min. At the beginning of the fourth cycle the activity was 80,000 ± 10,000 counts/min. It should be noted that this activity is the sum of the gaseous and the plated activities within the purge gas line.

Beryllium Irradiation Studies. — The second high-temperature beryllium swelling experiment has been completed, and the experimental assembly was removed from the ORR for examination. Specimens were held at 500, 600, and 700°C and irradiated to doses of approximately 1.5×10^{20} neutrons/cm^2 (> 0.66 Mev) during the one-month reactor cycle. Although the density measurements are incomplete, the data indicate that less than 1% swelling occurred. This is considerably less than that observed after the first irradiation. This and other evidence indicate that beryllium temperatures in the first swelling experiment were higher than the temperatures indicated by the thermocouples. Bend tests, hardness measurements, and metallographic examination will be performed.

Studies of Gas Mixing in Channels. — A preliminary series of experiments has been completed in which the helium tracer technique was used to study gas mixing in a channel containing three EGCR-type fuel-element clusters. Two relative rotational orientations were investigated: rods in line and rods rotated 30° between successive clusters. The results show

that helium introduced at a specific downstream location is not completely mixed with the carrier gas (air) even in two cluster lengths. It appears that the predominant mixing mode is radial rather than circumferential.

Helium Purification Studies. – A kinetic study of catalytic oxidation of hydrogen, carbon monoxide, and methane from helium in a fixed bed has been completed. Tests were conducted with 1/4-in. pellets of Girdler G-43 catalyst at 500°C and 300 psia. Bed depths of 3, 6, and 12 in. in the 2-in.-diam reactor with mass flow rates of 0.062 to 0.203 mole cm^{-2} min^{-1} were used with the contaminant concentration varied from 0.2 to 2.0 vol %.

Carbon monoxide is more effectively oxidized by oxygen in the catalyst than is hydrogen; however, methane is much more difficult to oxidize than either carbon monoxide or hydrogen. When more than one contaminant was in the helium, it was found that the hydrogen oxidation occurred in preference to either the carbon monoxide or the methane oxidation and that carbon monoxide oxidation occurred in preference to the methane oxidation. A conservative reactor design procedure would be to include a catalytic bed volume for oxidation of each of the contaminants based on the assumption that each contaminant oxidation occurred in series in the bed with the above-mentioned preference.

Initial results were obtained from a study of removal of radioactive iodine vapor from a high-temperature helium stream. It was found that activated charcoal removes iodine vapor from helium with high efficiency at temperatures as high as 430°C, but the iodine is slowly released from the charcoal upon continued helium flow. A bed of activated charcoal (Pittsburgh BPL) 3 in. in depth, operating at either 300 or 430°C, removed 99.9% of the iodine vapor from a helium stream moving at a velocity of 25 fpm. In these experiments the adsorbed iodine was not removed from the charcoal by helium flow for periods of up to 3 hr. However, later work indicated that continued helium flow slowly removed the iodine contained in the charcoal bed. Radioiodine vapor was adsorbed from a helium stream onto a 0.375-in.-deep charcoal bed operating at 320°C, and the elution rate was studied. After 144 hr of operation at temperature, it was found that 84% of the adsorbed iodine had been eluted.

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. – The HRT was operated with H_2O at high temperatures (core, 260°C; blanket, 230°C) and pressure (1400 psig) for approximately seven days to study the core-blanket mixing rate and evaluate equipment performance. Equilibrium blanket-to-core concentration ratios, determined by using chromic acid as a tracer, were 0.07 and 0.23 with blanket purge rates of 6 and 3 lb/min. The leakage past the two patched holes is much less than it was before the occurrence of the second hole and will not prevent the reactor program from continuing.

The method of pressurizer control devised to allow operation with steam in both pressurizers proved to be satisfactory.

Most reactor components operated satisfactorily during the equipment shakedown. An exception was a blanket-feed-pump diaphragm head, which failed (it had been in use since the reactor was built three years ago). The head was replaced, along with two chemical-plant components (see the section, "HRT Chemical Pilot Plant").

Low-pressure recombination experiments, made by using H_2 and O_2 from cylinders, indicated that the fuel secondary recombiner operated satisfactorily. The primary unit, however, did not satisfactorily recombine the simulated radiolytic-gas mixtures of the concentration expected during reactor operation. Further tests are planned to improve the efficiency of the primary recombiner, although it is not necessary for reactor operation.

The reactor experimental program will resume as soon as all light water has been drained and rinsed from the reactor components and piping.

HRT Chemical Pilot Plant. — Results from dissolver-solution samples showed that 203 g of corrosion-product solids was collected by the hydroclone system during the reactor core-backflushing operation. The solids contained 4% uranium, and the corrosion-product composition was also similar to that from previous operation. Circulating-solids concentrations during this period, deduced from the removal rate (20 g/hr), were much higher than in any previous operation, as expected. Later, after the new multiclone had been installed and while the system was being operated with hot H_2O, flow in the hydroclone loop was found to be almost completely blocked. The plug is presumably at the screen in the inlet line, which is designed to protect the hydroclones from plugging by large particles. In this period, also, current drawn by the hydroclone-loop circulating pump in the chemical plant became very erratic, indicating bearing failure. The pump was replaced with a spare, and the screen was removed and replaced with one containing 1/16-in. instead of 1/32-in. openings. The minimum hydroclone port openings are 0.100 in.

Initial nonradioactive full-scale tests of the uranyl peroxide precipitation fuel-cleanup process were only partially successful. Peroxide decomposition was readily controlled and what appeared to be a suitable precipitate was formed, but the sintered stainless steel filters did not contain the precipitate at pressure drops high enough to be attractive. In all laboratory tests with glass frits of the same nominal pore size, the UO_4 was contained quantitatively. Laboratory studies are in progress to determine if this problem may be solved either with finer filters or with filter aids.

Chemical Research for Aqueous Homogeneous Reactors. — Three-dimensional models representing the solubility relationships in the system UO_3-CuO-NiO-SO_3-D_2O at 300°C were constructed for solutions having total sulfate concentrations of 0.06, 0.10, 0.20, 0.30, and 0.50 m. The results of previously reported solubility studies were used to establish the location of the surfaces of saturation for the several solid and liquid phases which appear at the boundary limits of the region of unsaturated solutions. The particular saturating phases shown in these models are the solids $3CuO \cdot SO_3 \cdot 2D_2O$, $NiSO_4 \cdot D_2O$, $UO_3 \cdot UO_2SO_4 \cdot 5D_2O$, $CuO \cdot 2UO_2SO_4 \cdot 7D_2O$, $CuO \cdot 3UO_3$, and $NiO \cdot 3UO_3$ and a second liquid phase of variable composition.

The effect of concentration on the temperature of the appearance of a second liquid phase was determined for a solution having composition

ratios expected to be present in HRE-2 fuel solution. It was characterized by the following molal ratios:

UO_2^{++}/SO_4^{--}	0.358
Cu^{++}/SO_4^{--}	0.181
Ni^{++}/SO_4^{--}	0.118
D_2SO_4/SO_4^{--}	0.343

The minimum two-liquid-phase temperature observed was 321°C at a total sulfate concentration of 0.37 m. A similar solution containing more acid was tested for comparison. It had corresponding mole ratios of 0.312, 0.162, 0.106, and 0.420. Its minimum two-liquid-phase temperature was 330°C at a total sulfate concentration of 0.35 m. These results from solutions containing much higher concentrations of nickel than those previously studied were found to be in good quantitative agreement with the previous results when tested against the relationship between minimum two-liquid-phase temperature and relative acidity (D_2SO_4/SO_3). From this it appears that such a relationship may have practical value in a series of reactor operations involving relatively small changes in the composition of the fuel solution.

Analytical Chemistry. — An amperometric method was developed for the determination of thorium; the method can be applied by remote control to the analysis of slurries of ThO_2 and also to reactor fuels consisting of molten fluoride-salt mixtures of Li, Be, Th, U, and Zr. Thorium is titrated with the disodium salt of ethylenediaminetetraacetic acid (EDTA) in a deaerated, acetate-buffered medium at pH 2.5 to 4.5, to which Fe(II) is added. A platinum-foil indicator electrode is utilized at a potential of +0.45 v vs the S.C.E. The Fe(II) remains unoxidized until the end point is reached, after which an oxidizable Fe(II)-EDTA complex is formed, producing a linear increase in the current and, therefore, a break in the current-titrant curve. The end point is established as the point of intersection of lines drawn through the two straight segments of the titration curve which differ in slope. For milligram quantities of thorium, the coefficient of variation is less than 1%.

Within the applicable pH range, 2.5 to 4.5, a number of interferences can be avoided by choice of pH; Ce(III), Cr(III), and Al can be tolerated at pH 2.5 but not at 4.5, whereas the reverse is true for Zr. Lithium, UO_2^{++}, Mn(II), and Mg do not interfere at either pH. Zinc, Ni(II), Cu(II), Fe(III), Co(II), VO^{++}, and PO_4^{---} interfere in quantities equimolar with the thorium, whereas Cl^-, NO_3^-, and SO_4^{--} in molar concentrations tenfold greater than the thorium do not interfere. At a pH of 4.5, F^- can be tolerated at a molar concentration equal to that of the thorium.

An indirect polarographic method, initially described by Flaschka [Z. anal. Chem. 156, 321 (1957)], was modified for the determination of microgram quantities of thorium in reactor materials. The thorium reacts with lead ethylenediaminetetraacetic acid (Pb-EDTA), liberating an equivalent amount of Pb^{++} which is then determined polarographically at a half-wave potential of −0.45 v vs the S.C.E. By this method, from 2 to 16 μg of Th per ml can be determined with a coefficient of variation of about 6%. Although the sensitivity is of the same order as for colorimetric

methods for low-level thorium, the polarographic method, when applicable, can be adapted more easily to the analysis of radioactive materials by remote control. Interference studies revealed that, with the ORNL model Q-1673 derivative polarograph, 20 μg of Th can be determined in the presence of 270, 80, and 10 μg of U, Cr, and Mo respectively; Cu, Ni, and Fe, which appear to displace Pb from the Pb-EDTA complex, must be absent.

Slurry Circulation Experiments. — Run 22 in the 200A loop, intended for observing the high-temperature dropout behavior of a slurry of 3.65-μ-diam thoria fired at 1600°C, was completed. Settling of some slurry along the bottom of horizontal pipes was observed at about 3 fps in the temperature range 240 to 280°C. As the velocity was increased, a concentration gradient was noted, until at about 12 fps the slurry appeared virtually uniform.

Capillary-tube viscometer measurements were made with 200B-4 slurry, a 1600°C-fired 1.8-μ thoria. Vertical tubes with inside diameters of 0.011 to 0.124 in. were operated at room temperature with slurries containing 400 to 1000 g of ThO_2 per liter. It was found that a single shear diagram was formed by all data points which were in the laminar-flow region at a particular concentration, indicating that the particular system did not have appreciable wall slip. Shear rates up to 1500 (lbf·sec)/(lbm·ft) were used, which are higher than reported previously for aqueous thoria slurries. The indicated Bingham plastic constants, yield stress and coefficient of rigidity, were nearly a factor of 2 different when estimated from the complete shear diagram and from only that portion generated by the 0.124-in. viscometer. Some runs were made in a 0.124-in. horizontal tube which gave a somewhat different shear diagram, presumably because of settling tendencies.

Engineering Development. — The HRT mockup loop was operated at temperatures between 225 and 300°C with diluted HRT-type fuel solution to investigate uranium precipitation under conditions which might be obtained in the HRT blanket. The solution was stable at 1 g of U per liter, but some hydrolysis occurred at 300°C with 500 ppm of U. Instability was observed at 275°C when the uranium concentration was 10 to 18 ppm.

Modifications to the 5-in. cylindrical model of a proposed HRT replacement core were tested, demonstrating reduced residence time of fluid at the top and bottom of the core. The model is being further modified to include hemispherical ends.

Three 4-in. flanged joints, containing double Conoseals with buffer zone, were received. These joints are promising for future reactor applications since they cost less than the ring joints used in the HRT and have much less space for accumulation of solids.

Tools were designed for removing the lower patch from the HRT core and for reaming the core wall at the lower hole to provide a good seating surface. The practicality of improving the seating surface by reaming was demonstrated in the bench test of a full-scale model.

A mechanical system was developed for the convenient coupling of a Questar telescope to the omniscope utilized in examining the HRT core. The system was operated successfully at the reactor site to provide detailed examination of the core wall at high magnification.

Design. — Preliminary designs were completed of HRT replacement vessels containing long cylindrical Zircaloy core tanks. Either re-entrant or straight-through flow appears practical in the system, consistent with the ability to replace the core. The vessel can be installed either in the same location as the present core or in an adjacent cell on the basis of preliminary piping layouts.

The core vessel will be about 23 in. in diameter and 48 in. long with pure D_2O moderator, or it could be reduced to 19 by 38 in. if beryllium is used as a reflector. A thoria-pellet blanket is placed near the pressure vessel wall.

Drawings of diaphragm heads for the HRT feed pumps were revised and improved. The revised design can be fabricated at lower cost with greater assurance that all metal chips will be removed from the assembly.

A carrier was designed for transporting HRT fuel samples from the 7500 area to the X-10 area. The carrier is fastened to the frame of a truck and prevents a radioactive spill in the event of any foreseeable automobile accident.

MOLTEN-SALT REACTOR PROJECT

MSRE Design. — Detailing of MSRE components is progressing on schedule. Additional shielding of INOR-8 will be provided in the upper plenum of the reactor vessel to reduce the radiation level at the lower bearing of the circulating pump. The inlet volute of the vessel is being revised to reduce the locally high stresses and to eliminate fabrication difficulties. The reactor will be surrounded by a shield (16 in. of iron and water is being considered) to attenuate and capture fast neutrons.

The mechanical design of the MSRE primary pump is being reviewed. The design provides 10 in. of space for nuclear shielding between the liquid level in the pump tank and the region of the lower seal. A contract is being negotiated for casting the impellers and volutes. Specifications for the drive motor of the primary pump were released for purchase, and specifications have been started for the variable-frequency power supply for the primary pump and for the drive motor for the secondary pump. Detail drawings of the hot loop for the primary pump were completed.

A first-draft heat exchanger specification was reviewed, and revisions are being made. Heat exchanger manufacturers will be asked to provide mechanical design and fabrication of the primary heat exchanger.

Layout of the primary system is being revised so that it will be aligned with the building center lines (the original layout was rotated 30° off the building center line, parallel to the radiator center line). This change should simplify the crane movements required during maintenance.

Design of the heat dump has reached the detailing stage. The radiator doors will be held open by magnets. The doors close by gravity fall when the magnets are de-energized by a low-temperature signal from the radiator tubes or by indication of stoppage of the coolant pump. Such rapid closing action is desirable because preliminary simulator data predict that the coolant salt temperature will drop to the freezing point within 23 sec after pump stoppage.

Fabrication drawings of the drain tanks are nearly complete. Charging, draining, and occasional sampling in these tanks will be done through a single penetration in the gas space. The connection will be flanged off when the reactor is critical.

Specifications for INOR-8 plate, sheet, rod, pipe, and tubing are being published in final form. Major prospective suppliers say that they can produce a product complying with the chemistry and physical-properties sections of the specifications.

Component Development. — A frozen-lead seal around the shaft of a small centrifugal pump has operated continuously for 21,000 hr. The pump, mounted vertically over a fractional-horsepower motor, circulates molten salt at 1200°F.

Testing for leak-tightness and length of useful service of a metal-alloy freeze valve for use with the sampler-enricher is continuing. The 72% Ag—28% Cu alloy has been melted 17 times and held 60°F above the melting point (1500°F) for a total of 11-1/2 hr. For 15 closures out of the 17, the leak rate was $< 1 \times 10^{-8}$ cc of He per sec with a 30-psi differential pressure. The two unsuccessful closures were attributed to operator error rather than to a mechanical fault in the system. Diffusion of nickel from the INOR-8 valve parts has had little effect thus far on the alloy melting point and its wetting characteristics.

Measurements on the one-fifth-scale hydraulic model of the core continued. The center-line circumferential velocities along the volute were a constant 15 fps except at the inlet, where the velocity was 16 fps. This circumferential velocity component of 15 fps continued down the core wall, indicating little or no attenuation by the annular orifice. The average absolute velocity down the core wall was 15.6 fps. Radial swirl killers, which have a length equal to 36% of the lower head radius and are spaced 7-1/2° around the outer edge of the lower head, were installed, and a low radial pressure drop of 1.1 in. of fluid was measured. By use of a conductivity salt-injection technique to measure the fluid residence time at the bottom and the center line of the lower head, it was estimated that the temperatures there will not rise more than about 20°F above the mixed mean temperature in the lower head.

Construction of the freeze-flange thermal-cycle facility was completed, and the facility was charged with LiF-NaF-KF (46.5-11.5-42 mole %). Two freeze flanges, with pipe sizes of 3-1/2 and 4 in. and equipped with nickel-plated ring gaskets and standard clamp rings, were thermal-cycled three times between room temperature and 1300°F to establish operating procedures for the facility and to determine a base for future testing. Fabrication of a resilient clamp ring was completed.

A request for quotations for supplying prototype flanges in accordance with the 110,000-lb axial-strength design was prepared for issue.

The freeze valve loop, with resistance heat for the thaw cycle, has been operated through 16 fast or nearly full-power melts using the zirconium-bearing MSRE fuel with no apparent difficulties. Average melt times were 3 to 4 min, and the freeze times of 20 to 30 min with 60 cfm of cooling air were considered acceptable. About 6 cfm of air flow was required to maintain the plug once it was established. Pressure differences of up to 60 psi were held for periods of 16 to 18 hr with no indication of leakage. The estimated length of the plug, including the transition area, was

3 in. Eleven minutes was required to melt the plug with only the heat from the adjacent line heaters.

Remote Maintenance. — A proposal was made for the incorporation of semidirect maintenance capabilities in the design as a supplement to the remote-maintenance method now being prepared. Plans were begun for a scale model of the reactor and maintenance area to be used as a design aid in working out problems in maintenance.

Three types of manipulator-operable disconnects for reactor service piping are being prepared for evaluation. A thermal-cycle arrangement has been installed which uses 250-psi steam to provide a first screening test.

A layout of the secondary containment area has been approved, and design of this structure is beginning. Air-handling criteria have been established. All necessary decisions have now been reached to permit designing of the remote-maintenance facilities and equipment. Sealing and shielding of the primary containment vessel have been worked out conceptually and are now in design. The design of the first model of the enriching and sampling apparatus is 90% complete.

Pump Development. — The molten-salt pump containing a salt-lubricated journal bearing continued in operation with $LiF-BeF_2-UF_4$ (62-37-1 mole %) at 1225°F and has accumulated 4428 hr of running time, including 69 starts and stops.

The Inconel centrifugal pump (PKP type) continued in operation with $LiF-BeF_2-ThF_4-UF_4$ (65-30-4-1 mole %) at 1225°F, 1950 rpm, and 510 gpm and has accumulated 698 hr of running time. An estimate was made of the hazards associated with the back-diffusion test, in which Kr^{85} will be used.

Chemistry. — Quenched samples of $LiF-BeF_2-ZrF_4-ThF_4-UF_4$ (70-23-5-1-1 mole %) show that solidification on cooling begins at 440 ± 2°C with precipitation of a ternary compound containing LiF, BeF_2, and ZrF_4. Then at 431°C, Li_2BeF_4 begins to precipitate. Also, the proportion of LiF to BeF_2 can be increased to 73-20 without increasing the melting point more than 10 or 15°C, while a 10°C decrease in melting point is associated with the $LiF-BeF_2$ proportion of 67-26.

Particles of ZrO_2 grown in the molten fuel by exposure to impure helium were typically six-sided elongated hexagons (monoclinic), 50 × 35 × 10 μ or smaller; they settled well, did not adhere to the wall, and contained no detectable UO_2 (estimated limit of detection < 0.1%). A noticeable solid evolution range has been demonstrated for the nominal composition $ZrO_2 \cdot ZrF_4$.

The solubility of BF_3 in the fuel is adequate for convenient use as a removable poison according to recent measurements showing that Henry's law is followed (solubility = 8.26×10^{-5} mole of BF_3 per cc of fuel per atm at 600°C) and that the heat of solution is 15 kcal/mole.

Four 100-kg batches of MSRE composition fuel have been manufactured for engineering tests.

A final report on the $NaF-PuF_3$ system includes additional data, in the range between 33 and 55 mole % PuF_3, which confirm and extend a preliminary diagram showing a eutectic at 727°C and 23 mole % PuF_3, and an incongruent melting point for $NaF \cdot PuF_3$ at 842°C.

Metallurgy. — The decomposition of crystals of ammonium bifluoride in the presence of graphite has been found to be a successful technique for removing oxygen contamination from graphite. The oxygen, if not removed, would cause uranium dioxide precipitation from $LiF-BeF_2-UF_4$ salts. In precursory tests to determine the effect of the thermal decomposition products of ammonium bifluoride crystals on INOR-8, a reaction layer approximately 0.0005 in. thick formed on the INOR-8 in a 100-hr test at 1300°F. X-ray analyses of the layer indicated two or more complex structures that could not be identified by the current ASTM data cards.

Corrosion Tests. — Eight corrosion pump loops of INOR-8 operated with various fluoride salts during October. Loop 9354-3 was terminated for metallurgical examination after 20,000 hr of operation. The remaining loops have operated at 1300 to 1500°F for periods from 5000 to 19,000 hr. The graphite-molybdenum compatibility test section was assembled and was inserted into an INOR-8 thermal-convection loop. The test will begin operation with molten salt in November.

Reactor Analysis. — Two-group two-dimensional calculations were performed to obtain an estimate of the reactivity change resulting from graphite distortion and low-cross-section fission product buildup in one year's operation of the MSRE at 10 Mw. A net decrease of 1.3% $\delta k/k$ was obtained, with graphite distortion contributing an increase of 5.7% $\delta k/k$ and fission product buildup contributing a decrease of 7.0% $\delta k/k$.

The external shielding required during removal of the reactor vessel was evaluated for the case of one year's operation at 10 Mw. After 1 day of cooling, 2.9 ft of barytes concrete is required with the reactor vessel 10 ft away to reduce the radiation to tolerance if the source consists of the activated INOR-8 and 1% of the fission products. The principal activity in the INOR-8 is 72-day Co^{58} resulting from an (n,p) reaction in Ni^{58}; the calculated activity is based on fission-spectrum-averaged cross sections of 225 mb for activation of 72-day Co^{58} and 13 mb for activation of the 9-hr isomer Co^{58m}. Estimates of the cross section for the activation of the 72-day isomer range from 40 to 225 mb, so that the above estimate of the shield thickness is believed to be conservative. If no fission products are assumed to be retained in the reactor, the shield thickness is reduced to 2.8 ft.

Heat Transfer and Physical Properties. — Preliminary viscosity measurements were completed for the new MSRE fuel mixture $LiF-BeF_2-ZrF_4-ThF_4-UF_4$ (70-23-5-1-1 mole %) with a capillary efflux viscometer. The data, obtained with three different capillaries, exhibit considerable scatter. The scatter may have resulted from the unexpectedly large amount of ZrO_2 observed in the melt in a postoperational analysis. A conservative interpretation of the data yields a kinematic viscosity of 5.4 centistokes at 600°C and 2.15 centistokes at 800°C.

Assembly of the variable-gap thermal-conductivity device has been temporarily discontinued in order to concentrate available manpower on the determination of the viscosity, heat capacity, and density of the new MSRE fuel mixture.

Heat transfer studies with $LiF-BeF_2-UF_4-ThF_4$ (67-18.5-0.5-14 mole %) were resumed following replacement of the pump rotor. Total accumulated operational time is now in excess of 5700 hr. Test-section thermocouples

and power leads were also replaced during the shutdown period. The new pump arrangement was calibrated with respect to the turbine flowmeter. As the result of changes in the pump drive, the maximum flow rate now attainable is about 20% less than previously. Chemical analyses of the salt show significant differences between the original and the nominal compositions and between pre- and postoperational samples. Since the differences affect the values used for the thermal properties in the heat transfer correlations, a re-evaluation of the data is necessary.

It is planned to discontinue operation of this loop with the $LiF-BeF_2-UF_4-ThF_4$ (67-18.5-0.5-14 mole %) at the end of the current calendar year; the merit of additional studies with the MSRE fuel mixture will be considered at that time.

Fuel Processing. — A uranium recovery of 97 to 98% was consistently obtained in four runs with molten fluoride mixtures by means of a small steady-state fluorinator. Corrosion of the nickel container appeared to be serious. The fluorinator, a 1-in. nickel tube from which the salt overflowed through a 1/4-in. tube into a jackleg that held up enough salt to permit sampling, normally contained about 70 cc of salt. The salt flow rate was controlled by pressure transfer from a reservoir through a coiled 6-ft by 40-mil capillary tube. Flows around 5 g/min were obtained, corresponding to an average salt residence time in the fluorinator of about 40 min. The apparatus will have to be altered to obtain shorter residence times, which are desirable. Some difficulty was experienced in obtaining free flow of salt from the jackleg, thus backing the salt up in the fluorinator. This was overcome by operating at higher temperatures than desired.

The most meaningful runs were done with $LiF-NaF-ZrF_4$ (26-37-37 mole %) containing 0.2 wt % UF_4 in one case and 1 wt % UF_4 in the other. With an average salt residence time in the fluorinator of about 40 min, fluorination, with excess fluorine, at 600 to 650°C and at 700°C, respectively, resulted in > 98% uranium recovery. The uranium contents in the effluent salts were 25 to 50 and 100 to 200 ppm. The nickel concentration from corrosion increased throughout the run, never reaching a steady state. The observed nickel concentration corresponded to corrosion rates of the order of 1 mil/hr at 700°C and 0.1 to 0.2 mil/hr at 610°C.

MARITIME REACTORS PROGRAM

Physics. — A straightforward method for obtaining extrapolation distance boundary conditions for use in diffusion-theory calculations of control-rod worth was developed. The method consists in using the SNG transport code to solve for the flux distribution in an appropriate one-dimensional approximation to the control-rod cell. The SNG flux results may then be used to obtain equivalent transport theory control-rod extrapolation distance boundary conditions for use in a two- or three-dimensional diffusion theory calculation. Application of this method in a four-group, two-dimensional calculation of eight NS "Savannah" control-rod configurations gave excellent agreement with critical experiment results.

Loop Experiments. – Experiment No. 1 fuel specimens of swaged UO_2 have been reinserted in the pressurized-water loop facility for irradiation during one ORR 30-Mw cycle.

ANP PROGRAM

Shielding Research. – The modifications of the Tower Shielding Reactor II (TSR-II) which will make it suitable for use as a high-power (~5-Mw) shield test reactor at the Tower Shielding Facility have almost been completed. The components are being reassembled. Water flow tests are now being made on the annular fuel elements. The results of these tests will lead to optimization of the adjustable baffles. Following completion of the TSR-II assembly, its low-power operation at 100 kw with the Pratt and Whitney shield mockup will be initiated.

It has been demonstrated that the recently obtained, large sodium iodide crystal gives adequate resolution, and therefore an improved gamma-ray spectrometer is available. The improved device was used to initiate a program of measurements of the leakage gamma flux of the Bulk Shielding Reactor I (BSR-I). The leakage measurements made several years ago with an inferior spectrometer did not agree with calculated values. In addition to the improved spectrometer, more reliable calculated values are now on hand.

A determination was completed of the angular distribution of the thermal and epithermal neutron leakage from slabs containing methyl borate dissolved in methanol. These slabs were used to simulate the cross sections of lithium hydride. A 6-in.-thick slab and a 4-in.-thick slab, 1 ft in diameter, were available. A slab containing the boron solution was placed close to the BSR-I. On the side of the slab opposite the reactor there was an air box with collimating pipes leaving at various angles. The pipes contained indium and gold foils. The thermal and epithermal neutron angular distributions were measured with the indium foils at 1.4 ev and with the gold foils at 5 ev. The angular distribution was found to be independent of the slab thickness and the amount of boron, indicating that the slowing-down process was more effective than the poisoning. The experimental data agreed with diffusion theory calculations. Now that the experimental technique has been worked out, slabs of lithium hydride are being procured so that the experiment can be repeated on the material of interest.

Columbium Alloy Research. – The oxidation characteristics of Cb–2.5% Al, Cb–33% Ta–0.7% Zr, Cb–15% W–5% Mo–1% Zr, and Cb–10% Ti–10% Mo were examined at an oxygen pressure of 5×10^{-4} mm Hg at 1000°C. The weight gains in 300 min for these alloys were 2.20, 1.96, 1.2, and 3.12 mg/cm^2 respectively.

Experiments were conducted to determine the effect of nitrogen additions on the strength and bend characteristics of columbium; the additions were made to the pure metal. A considerable increase in yield strength, from approximately 30,000 psi to 39,000 psi, was caused by adding as little as 0.01% nitrogen. Columbium containing less than 0.2% nitrogen could be bent 90° without cracking or with only minor cracks. Columbium containing

0.2% nitrogen has a tensile strength of 74,000 psi, and formation of less than a 90° bend caused severe cracks that allowed further bending with little applied stress.

Welds were made on nine bend specimens of Electromet columbium alloy heat Cb-91. The base metal contained 7.48% Ti, 780 ppm O_2, 150 ppm N_2, and 490 ppm C. The weld metal analyzed 6.66% Ti, 740 ppm O_2, 150 ppm N_2, and 490 ppm C. Eight of the specimens were aged at 1600°F for 1, 2, 5, 10, 25, 50, 100, and 250 hr. The bend tests showed that the unaged material was ductile, that the specimens aged 1, 2, 5, and 10 hr were brittle, and that specimens aged for a longer period were ductile. These aging and overaging effects are very similar to those found in welds made on Cb–1% Zr alloy. Previously initiated efforts to determine the mechanism of aging in Cb-Zr alloy welds are continuing.

Tensile tests are in progress on aged sheet specimens from four heats of electron-beam-melted Cb-Zr alloys. The alloys are very low in oxygen and nitrogen but have different levels of carbon content. The effect of carbon content on the aging characteristics of otherwise exceptionally pure Cb-Zr alloy will become evident from these tests.

Beryllium Oxide Research. – Irradiation of the BeO specimens (test 5) continued in the ETR. It is expected that this test will be continued through the November ETR cycle, at which time the specimens will have received a dose of more than 10^{21} neutrons/cm^2. A sufficient number of center and outer thermocouples are performing satisfactorily in the BeO specimens to provide the required information on temperature gradients and thermal cycles. The BeO specimens from ETR tests 2 and 3 have been removed from their capsules and given a cursory examination at Battelle. There is no evidence of gross fractures or cracks in the BeO.

More than 500 g of BeO was steam-precipitated from four batches of 60% LiF–40% BeF_2. The material is now being treated with hot aluminum nitrate solution to wash off the fluoride solvent. Preliminary optical examination indicates that the material contains very little occluded impurities and that it is very-high-quality BeO.

Additional separation factors were obtained in the systematic investigation of the Versene–acetyl acetone purification of beryllium salts. When these factors were combined with previous data it became evident that the process held promise and that a worthwhile purification experiment could be devised. An experiment was therefore begun which aims at producing a 10-g batch of pure BeO.

Liquid-Metal Technology. – Sleeve-type Inconel inserts were tested in a refluxing potassium capsule test. The boiler temperature was 1600°F, the thermocouple well temperature in the vapor at the last insert was 1525°F, and the test period was 1000 hr. The weight changes of the inserts indicated removal of material from the vapor region and subsequent deposition in the liquid zones. The weight changes of the Inconel were an order of magnitude greater than those previously found for type 316 stainless steel in a 500-hr test.

Examinations continued of sheet tensile specimens from the type 316 stainless steel–boiling potassium loop test No. 2. The tensile test results indicated that no appreciable attack occurred on specimens subjected to either liquid or gaseous potassium at 1600°F. Metallographic examination revealed only slight grain-boundary attack to a depth of about 0.5

mil in the high-temperature region. Preliminary investigation of some small crystalline deposits on the sheet tensile specimens in the cold leg revealed a deposit 2 to 3 mils thick in which some $Cr_{23}C_6$ was detected by x-ray analysis. Analysis of the material deposited in a diffusion cold trap in loop test No. 2 showed that it was deficient in iron and that some preferential leaching of nickel and chromium had occurred in the hot zones during operation. Examination of representative samples of type 316 stainless steel from the loop structure itself revealed a subsurface void type of attack to a depth of 2 mils at the liquid level in the boiler. This was the only attack which could be detected by metallographic examination in the entire loop.

Preliminary thermal conductivity data have been obtained on an essentially pure lithium sample (99.8 wt % Li) and in a lithium-silver alloy (3 wt % Li). These data give values for the thermal conductivity of lithium ranging from 20 Btu hr^{-1} ft^{-1} $(°F)^{-1}$ at 740°F to 41.5 Btu hr^{-1} ft^{-1} $(°F)^{-1}$ at 1780°F. Determinations made on the silver alloy indicate that its thermal conductivity at 1520°F is 36.5 Btu hr^{-1} ft^{-1} $(°F)^{-1}$.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Release of Fission Products on In-Pile Melting of Reactor Fuels. — Earlier status and progress reports (ORNL-2945, -2980) stated that the design drawings for the in-pile melting of a UO_2 capsule were being finished and that some of the parts were being fabricated. The entry tube which will be mounted in the F-9 lattice position of the ORR and which will remain there as a facility for the meltdown experiments has been completed and installed. This facility will permit the installation and removal of individual experiments while the reactor is in operation. Since there will be occasions when the flow of water is stopped, a test was made to determine the temperature of the stagnant water, and it was found to be 192°F when the reactor was operating at 27 Mw. A device is being constructed to measure the vertical gamma-ray profile of the lattice part of the facility. These data are needed to assure that the necessary range of gamma heating power is within the travel distance of the positioning mechanism and to give a good estimate of the location, within the entry tube, where the irradiation and subsequent meltdown will take place.

A hydraulic unit for positioning the experiment will fit on top of the entry tube and will also be a permanent part of the reactor facility. This unit has been assembled and will be mounted, with the automatic control valves and control switches, in the laboratory and tested to determine its operational characteristics and its ability to maintain a given position under load.

The carbon traps and filter units, which will be used to collect volatile fission products, gases, and particulate matter for analysis in the first experiment, are being assembled and will be tested for flow rates and the holdup times of the rare gases.

The information and drawings necessary for the approval of the experiment by the Operations Division and the Reactor Safety Committee are nearing completion.

Basic Reactor Research. — A series of pulsed-neutron-source measurements with the Tower Shielding Reactor II (TSR-II) has been completed. Various amounts of dissolved boron neutron poisoning were homogeneously distributed throughout the core and most of the reflector of the TSR-II, resulting in varying degrees of negative reactivity. For the unpoisoned reactor, the observed reactivity (ρ) was zero with control rods partially inserted and -3.52 ± 0.12 dollars with the rods fully inserted. A boron concentration of 49.8 ppm made the reactor just critical with all rods fully withdrawn, and full insertion of rods gave $\rho = -4.77 \pm 0.14$ dollars. Poisoning to 99.6 ppm boron showed $\rho = -2.06 \pm 0.09$ dollars for rods fully withdrawn; partial insertion of rods (the unpoisoned just-critical position) gave $\rho = -4.01 \pm 0.14$ dollars. A boron concentration of 198.2 ppm gave a reactivity of -5.95 ± 0.20 dollars with all rods fully withdrawn. The measured reactivities will be compared with the results of calculations of the change in reactivity to obtain the value of the effective delayed neutron fraction for the TSR-II.

Power Reactor Fuel Processing: Darex Process. — Soluble-uranium losses from UO_2-ThO_2 pellets to boiling Darex and Sulfex solutions increased with increasing UO_2 content of the pellets. After 7 hr contact with boiling Darex dissolvent, 5 M HNO_3–2 M HCl, 0.45 and 0.65% of the uranium was solubilized from pellets containing 3 and 9% UO_2 respectively. Losses to Darex solutions were generally about 10 times higher than those to Sulfex (6 M H_2SO_4) solutions.

At 25°C the solubility of thorium nitrate in nitric acid decreased from about 620 to 180 g/liter as the acid concentration increased from 0 to 18.3 M.

Titanium was corroded at rates of 0.09 mil/month or less during 76 Thorex feed adjustment boildowns.

Power Reactor Fuel Processing: Mechanical Processing of Fuels. — The basic concept and the performance of the SRE dejacketing equipment were satisfactorily demonstrated in cold runs. Tests with unirradiated NaK-bonded fuel will be made next.

Core material was satisfactorily leached from twelve 1-in. chopped lengths of stainless-steel-clad ThO_2–5.3% UO_2 pellets (Universal Match Company) in boiling fluoride-catalyzed nitric acid containing both thorium and aluminum. Complete leaching of the 12 pieces was achieved in 27.5 to 46 hr with an average time of 36 hr and a standard deviation of 7 hr.

Power Reactor Fuel Processing: U-Mo Fuel Processes. — The rate of dissolution of uranium-molybdenum alloys in boiling nitric acid was maximum when the molybdenum content of the alloy was 3%. In 5 M HNO_3, uranium and uranium–8.4% molybdenum alloy dissolved at rates of 3 and 10 mg min^{-1} cm^{-2} respectively; uranium–3% molybdenum alloy dissolved at a rate of about 43 mg min^{-1} cm^{-2}.

The solubility of uranium–10% molybdenum alloy in nitric acid–phosphoric acid mixtures at 25°C was generally highest in solutions where the nitric acid concentration was about 6 M. At each nitric acid concentration, the solubility increased with increasing phosphoric acid concentration. In 6 M HNO_3 the solubility increased from 50 to 240 g of alloy per liter as the phosphoric acid concentration increased from 0 to 1 M.

The titanium corrosion rate was 0.06 mil/month or less after 1000 hr exposure in 3, 6, and 8 M HNO_3 containing 0.5 M $Fe(NO_3)_3$, 0.6 M $UO_2(NO_3)_2$, and 0.02 M MoO_3.

Power Reactor Fuel Processing: Zirconium-Uranium Fuel Processes. — A solution of 16 M HNO_3–0.58 M Cr–1.9 M F is being evaluated as a dissolvent for Zircaloy-2 and zirconium-uranium alloys in titanium equipment. Zirconium dissolution rates of 2 mg cm^{-2} min^{-1} and titanium corrosion rates of 3 mils/month were obtained at a zirconium concentration of 0.5 M. Lowering the zirconium concentration to 0.38 M increased the dissolution rate to 4 mg cm^{-2} min^{-1} but did not increase the titanium corrosion rate.

In additional Zirflex bench-scale tests of the dissolution of Zircaloy-2 in 4.5 to 6 M NH_4F–0.5 M NH_4NO_3, runs were made in an attempt to produce a dejacketing waste solution free of solids. Although low free fluoride concentrations in the range 0.4 to 0.8 M F^- are required to prevent precipitation of the ammonium fluozirconates, the fluoride cannot be totally depleted if the hydrated oxides of tin and zirconium are to be held in solution. Even with complete withdrawal of the NH_4OH-containing overhead condensate, solids formed when the fluoride was depleted. However, with F/Zr charge ratios of 6.5, relatively solids-free solutions containing ~0.7 M zirconium were produced with both 4.5 and 6 M NH_4F–0.5 M NH_4NO_3 with complete withdrawal or with rectification and reflux of the overhead condensate.

Power Reactor Fuel Processing: Processes for Advanced Fuels. — The most effective aqueous dissolvent yet found for sintered BeO (about 95% of theoretical density) is boiling 5.8 M NH_4HF_2; the BeO dissolved at an initial rate of 1.7 mg min^{-1} cm^{-2}.

Leaching of coarsely ground (–4 +8 mesh) solid-impregnated graphite–8% UO_2 fuel with boiling 15.8 M HNO_3 resulted in uranium recoveries of 99.8 and 94.5% from fuel with 70–80 and 350–420 μ UO_2 particles respectively. Recovery was > 99% for both fuels when ground to –16 mesh. Uranium recovery from solution-impregnated graphite was also lower than from graphite containing 80-μ UO_2 particles.

Ungraphitized fuel specimens of Pebble Bed Reactor fuel spheres prepared by National Carbon and Great Lakes Carbon were leached with 90% HNO_3 at 25°C. The resulting yellow solution contained < 0.3 mg of carbon per milliliter of solution. Treatment of the Great Lakes specimens with boiling 90 or 70% acid produced a deep wine-colored solution containing > 2 mg of carbon per milliliter.

Metallographic examination of a sample of UC indicated that one-third of the sample contained about 2% α-uranium metal, one-third about 1% metal, and one-third no metal. Since finely divided uranium metal reacts with water to produce hydrogen, a tentative explanation has been provided for the presence of free hydrogen in excess of that required for an H/C ratio of 4.00 in the products of UC hydrolysis. Experimentally, hydrolysis of samples of UC with water at 90°C yielded UO_2 and gaseous products with an average H/C ratio of 4.18, an average total carbon content of 3.81 millimoles per g of UC, and an average total hydrogen content of 15.94 millimoles per g of UC. This corresponds to an average uranium impurity content of about 4%.

Power Reactor Fuel Processing: Solvent Extraction Studies. — Varying the point of addition of acid in the Acid Thorex flowsheet from the feed point to each of the later extraction stages in succession tended to (1) decrease the acidity at the feed point and in the organic product, (2) decrease the ruthenium decontamination factor, and (3) increase the zirconium-niobium decontamination factor. The extraction of fission products increased with contact time at 55°C with Acid Thorex feed plate conditions. Optimized final uranium purification flowsheets under Pilot Plant equipment restrictions did not result in a decrease in decontamination with increase in tributyl phosphate concentration. A single-cycle Acid Thorex flowsheet resulted in decontamination factors of boron and cadmium from uranium of $> 10^4$ and $> 1.5 \times 10^3$ respectively.

Power Reactor Fuel Processing: Criticality Studies. — In evaluation tests to determine the physical and chemical stability of borosilicate glass Raschig rings as a neutron poison packing in tanks filled with a fissile solution, Pyrex glass appears to be satisfactory, but a 5.8% boron glass does not. Exposure of two borosilicate glass compositions, a special 5.8% boron glass and a 4.0% boron Pyrex, to 2 M and 6 M HNO_3 at 23° and 65°C for 650 hr produced no discernible attack on the Pyrex. The high-boron glass was crazed and cracked to varying degrees, indicating its unsuitability. The amount of boron leached from each glass has not yet been determined.

Treatment and Disposal of Radioactive Wastes: High-Activity Wastes. — Synthetic Purex waste was continuously evaporated and calcined to 900°C in a closed system consisting of a feed tank, a 4 × 18 in. stainless steel pot calciner, a glass condenser, a glass scrubber packed with stainless steel packing, and a gas recirculation pump. Nitric oxide gas was added as required to prevent pressure buildup in the constant-volume system. Sodium and magnesium ions were added to the feed to decrease sulfate volatility. A volume reduction factor of almost 10 was achieved. The residue had a bulk density of 1.9 (about 40% porosity). The condensate contained 95% of the total nitrate fed to the system, 47% of the ruthenium, and 0.5% of the sulfate. The consumption of nitric oxide in the experiment was about 0.4 mole per liter of waste or 0.06 mole per mole of nitrate in the feed. The quantity of ruthenium in the condensate did not reproduce the 1.5 to 3.0% obtained in batch experiments with the same feed plus nitric oxide. The discrepancy has not been explained but will be studied further.

In calcination experiments in 8 × 84 in. stainless steel pots with synthetic Darex (stainless steel nitrate), TBP-25 (aluminum nitrate), and Purex wastes, processing rates averaging 22 to 24 liters of feed per hour were obtained. These rates are surprisingly high for Darex and TBP-25 wastes, since the volume of liquid boiled over per unit volume of deposited solid was higher than for the Purex waste by factors of 1.3 to 1.6.

In a batch experiment, equal volumes of simulated Purex waste and calciner condensate acid were combined and evaporated by a factor of 2 in the presence of nitric oxide. The condensate was decontaminated from ruthenium by a factor of about 10^4, indicating that this proposed recycle scheme would be satisfactory. In another batch experiment, one volume of Purex waste and one volume of calciner condensate were combined, diluted with six volumes of water, and evaporated by a factor of 8 in the absence

of nitric oxide. The evaporator condensate contained about 1% of the feed ruthenium, indicating that more dilution with water is required to make this proposed recycle scheme satisfactory.

Treatment and Disposal of Radioactive Wastes: Low-Activity Wastes. — Clinoptilotite, a zeolitic mineral, was tested in the low-level ORNL waste treatment process under the same conditions as those for the phenolic ion exchange resins. With cesium- and strontium-spiked ORNL waste made 0.01 M in NaOH (pH ~12), gross activity (1% level) broke through at ~900 bed volumes of waste treated, as compared with 1500 to 2000 volumes for phenolic-sulfonic and phenolic-carboxylic resins. Duolite C-65, a phenolic-phosphoric resin, had a breakthrough capacity of about 1000 bed volumes for removing Cs^{137} from ORNL waste, about the same as for the phenolic-only resin previously tested. The ion exchange beds for a full-scale (750,000-gal/day) ORNL low-level waste treatment have been tentatively sized at 4.2 ft inside diameter by 8.4 ft deep. Two beds in parallel would be required in a plant which used two exhaustion-regeneration cycles per week (1500-bed-volume throughput capacity), and three beds in parallel would be required in a plant which used one cycle per week (2000-volume capacity). The chemical cost would be 21.4¢ per 1000 gal for the two-cycles-per-week operation, or 19.1¢ for a one-cycle-per-week operation.

Waste Disposal Research and Engineering: Geochemical Studies. — Vermiculite was found to be relatively ineffective for removal of strontium from simulated waste containing 0.1 M $NaNO_3$ and 1.1×10^{-5} M strontium at pH 7.0. Ways to improve the effectiveness of vermiculite were suggested from data obtained with Al_2O_3, which were reported earlier. By increasing the pH and by the addition of soluble Na_2HPO_4, the strontium removal was greatly increased, as shown by the following results for passage of 100 ml of waste through a 1-g column of vermiculite: At pH 7.0 and with 10^{-3} M Na_2HPO_4, strontium removal averaged 22%; when the pH was raised to 11.0 and the Na_2HPO_4 concentration was maintained at 10^{-3} M, the removal averaged 35%; when the Na_2HPO_4 concentration was increased to 5.0×10^{-3} M at pH 11.0, the removal averaged about 92%. The appealing feature of using vermiculite for waste disposal is that it has already been demonstrated to be very selective for cesium; studies to further improve the effectiveness of vermiculite for strontium are continuing.

Waste Disposal Research and Engineering: Disposal in Natural Salt Formations. — The liquid temperature in the acid cavity of the field experiments at Hutchinson, Kansas, has dropped from 63°C when the power was discontinued on August 2 to 25°C on October 19. Temperatures in the surrounding salt structure are now 24 to 25°C and are changing very slowly. Ambient temperatures in the structure are 21 to 22°C. The neutralized cavity is continuing to operate at steady state with the liquid temperature at approximately 71°C.

Four additional experiments are in operation in the mine. One is designed to study the transfer of heat from slender cylinders into salt. A second is being operated to evaluate the behavior of a shallow pool of waste solution on the floor of a sealed cavity. A third experiment is planned to study the alteration in shape of both full and partially full cavities completely in salt. A fourth small experiment is a measurement of the effect of several heat sources on the temperature of a single adjacent point. For the most part these experiments utilize equipment installed for the large field experiments.

Waste Disposal Research and Engineering: Deep Well Disposal. — On September 3, at a site about 2000 ft south of the HRT, the Halliburton Company injected about 100,000 gal of cement grout into the Conasauga shale at a depth of 934 ft by hydraulic fracturing. On September 10, a similar batch of 130,000 gal was injected through the same well at a depth of 694 ft. Both operations were performed essentially without incident, and the possibility of pumping large volumes of fluid mixture into this formation at rates of several hundred gallons a minute was clearly demonstrated. Test drilling for determining the orientation and extent of the fractures has been started, and some conclusions may be drawn from the data already available. The injection pressures were substantially greater than the weight of the overburden, a necessary condition for the formation of horizontal fractures. Pumping the grout in produced a slight but clearly measured uplift of the land surface out as far as 1000 ft from the injection well, which can only mean that the grout sheets conformed to the structure of the shale.

Waste Disposal Research and Engineering: Process Waste Water and Treatment Plant Studies. — On the basis of laboratory studies in accordance with the principles of response surface methodology, increased feed rates of lime, soda ash, and clay were suggested for improved removal of strontium and cesium from process waste. Laboratory results showed that by adding 2.5 times the stoichiometric amount of lime, 520 ppm of excess soda ash, and 360 ppm of Grundite, 96% of the Sr^{90} and 92% of the Cs^{137} were removed. The increased rates of chemical feed were applied to the plant for a period of one week, during which time 3.3 million gallons of waste was treated. Strontium removal was increased to 94% and cesium removal to 91%.

Waste Disposal Research and Engineering: Clinch River Studies. — Annual surveys made on the Clinch River have indicated that radioactive materials are sorbed on the bed sediments. Data from these surveys are qualitative in nature. Quantitative information is being obtained from core samples of these bed sediments, 1 in. in diameter, taken from eight to ten points in cross sections of the river. The cross sections were spaced at about 1-1/2-mile intervals from the mouth of White Oak Creek to the mouth of the Emory River. The variation in the gamma activity with depth is being determined for each core. From these data the distribution of radioactivity in the bed sediments will be described. The materials in selected cores are being separated and analyzed for their mineral, vegetable, and animal composition.

Waste Disposal Research and Engineering: Long-Range Evaluation of Over-All Waste Complex. — A study of all factors affecting the hazards of tank storage of liquid fuel reprocessing wastes is in process. All known sources have been searched for methods of predicting the consequences of accidental release of tank contents. It is found that dispersion into the atmosphere can be readily characterized. Methods have been found which show promise of describing movement through geologic structures, ground water, and surface water.

A partial evaluation of the hazards of a hypothetical tank site chosen for economic reasons has been made, and preliminary investigations of the hazards of the Interim Fuel Reprocessing Facility proposed for ORNL are being conducted.

PHYSICAL RESEARCH PROGRAM

PHYSICS AND MATHEMATICS RESEARCH

Scintillation Spectrometry and Instrument Development. — The Mössbauer effect was observed in Ni^{61}. An Ni^{64} source and a natural nickel absorber foil were used. The Ni^{61*} was obtained by the reactions

$$Ni^{64}(p,\alpha)Co^{61}(\beta^-,99\ min)Ni^{61*} \quad ,$$

which gave a nickel Mössbauer nucleus in a nickel host lattice. The magnitude of the effect at 80°K is about 4% for a 400-mg/cm^2 absorber foil (natural nickel). The temperature dependence yields a Debye temperature between 413 and 437°K (Debye model). The velocity dependence shows no Lorentz shape. The measured half-width is 1.65 mm/sec. A recent electronic determination of the lifetime of this 71-kev state indicates a natural half-width of 0.37 mm/sec. The increased half-width that was observed may be understood in terms of a magnetic hyperfine splitting. The moment of the ground state is known. The internal field H and the magnetic moment μ_e of the first excited state are unknown, but the measurement gives a connection between the two quantities. The first excited state of Ni^{61} is probably a single-particle state, so that μ_e may be estimated with reasonable confidence. The internal field H calculated from the estimated value of μ_e is 57 kilogauss.

High-Voltage Experimental Program: Levels in N^{14} at 11.74 and 11.82 Mev. — The known angular distribution of the 3.68-Mev gamma rays from the inelastic scattering of protons by C^{13} is not sufficient to determine uniquely the spin and parity of the 11.74-Mev N^{14} level for the case of arbitrary channel spin mixing. To resolve this ambiguity the angular distribution of the inelastically scattered protons was measured. Of the previous possibilities of $J^\pi = 1^+, 2^\pm$, and 3^-, the assignment 1^+ has been selected. A new level was found at 11.82 Mev in N^{14}, having a width of about 100 kev and decaying through the 3.09-Mev level in C^{13}. The angular distribution of the inelastically scattered protons from this level shows strong interference effects.

High-Voltage Experimental Program: The N^{14} Threshold. — An attempt was made to observe the $N^{14}(He^3,n)F^{16}$ threshold. A lead nitrate target was used. No threshold was found near the value that has been reported. Buildup from the beam gradually produced enough C^{12} to clearly show the $C^{12}(He^3,n)O^{14}$ threshold near its reported value.

Heavy-Particle Physics. — Measurements of energy and angular distributions of alpha particles from the nitrogen bombardment of fluorine were completed. The distributions are consistent with an exponential cutoff in the angular momentum factor of the nuclear energy level density. The angular distributions are symmetric about 90° in the center-of-mass system, as required by the statistical model of nuclear reactions. The experimental results give no clear-cut evidence concerning the shape of the

energy spectrum of the alpha particles. The elastic scattering of N^{14} by Mg^{24} was measured from 24 to 120° in the center-of-mass system. No structure was found; the analysis of the data is not yet complete. Transfer reactions from N^{14} on B^{10} and B^{11} are being investigated with two silicon barrier counters in coincidence to detect the particles and identify the event by angle and energy information.

The 86-Inch Cyclotron: Nuclear Physics. – A systematic behavior of low-lying nuclear levels of odd-odd nuclei is observed in the internal-conversion permanent-magnet spectrograph. The 44.7- and 67.1-kev transitions observed in Lu^{174} are in cascade and obey exactly the $I(I + 1)$ interval rule for spin sequence 1, 2, 3. A similar case was observed in Ho^{162} for transitions of 38.3- and 57.8-kev energy. The associated inertial parameters $3\hbar^2/\mathcal{J}$ for the postulated rotational states are 67 and 58 kev for Lu^{174} and Ho^{162} respectively. The corresponding $3\hbar^2/\mathcal{J}$ for the ground-state rotational sequence of the even-even daughter nuclei are 76 and 81 kev.

The 86-Inch Cyclotron: Applied Physics. – The isotopes Na^{22}, V^{48}, Fe^{55}, Co^{57}, As^{74}, Sr^{85}, Pd^{103}, I^{124}, I^{125}, Pm^{145}, Eu^{147}, and Eu^{149} were produced as service irradiations for customers in the United States and India. Considerable effort was expended in developing and fabricating suitable targets to best utilize the unique characteristics of the ORNL 86-Inch Cyclotron, especially the availability of ~2 ma of internal beam. Demands for several curie sources of Co^{57} (essentially carrier-free) and for Na^{22} have necessitated experiments designed to increase these yields.

The Isochronous Cyclotron. – The contractor has made rapid progress on the construction of the ORIC Building 6000; the work is now 90% completed. The motor-generator set to supply power to the magnet was put in place, and the two large doors (~60 tons each) in the shielding were installed.

Major items for which design specifications were completed and orders were placed include the control console and control board, the rf shorting plane mechanism, and components for the magnet regulators. Items received were the vacuum manifold, the dee-house transport, and five of the 19 AVF magnet power supplies. Major cyclotron components for which designs are still to be completed are the cyclotron dee, the beam extraction system, and the valley coils and pole-face windings.

An error by the fabricator of the full-scale pole tips has required a re-examination of the design to permit use of the steel tips. In continuation of model magnet studies, about 30 field measurement runs were completed. More accurate data on circular trimming coils obtained in these runs are now being evaluated.

Mass Spectrometry and Related Techniques. – Positive ions produced in ethylene by Ni^{63} beta particles (maximum energy 0.063 Mev, source activity approx 1 mc) were studied with a mass spectrometer. The results were compared with those for ionization by Po^{208} alpha particles (energy 5.1 Mev, source activity approx 0.06 mc) and by 75-ev electrons under the same conditions (pressure in the ionization chamber approx 0.01 mm Hg). The relative proportions of the secondary ions $C_2H_5^+$, $C_3H_5^+$, and $C_5H_9^+$

arising from all of the sources are remarkably similar. This work represents the first known application of beta particles as an ionizing source in the mass spectrometer.

Stable Isotope Development. — Second-pass calutron separations of 5- to 10-g quantities of isotopically pre-enriched material have been proved feasible. This work has been extended from the initial separation of Pb^{204} to the isotopes Pb^{206} and Pb^{207} and makes samples of lead available having 99.975% Pb^{206} and 99.95% Pb^{207}. Material recovery from the system exceeded 85%. Natural abundances of the above isotopes are 23.6% for Pb^{206} and 22.6% for Pb^{207}.

Efforts are being continued to provide improvement in the Ca^{46} being separated for medical research. Ion beam resolution is sufficient to provide a more highly enriched sample than is actually achieved. Substitution of various collector materials did not materially reduce that portion of contaminants present in the ratio of normal abundances. Control pockets have been added recently to certain collectors in an attempt to correlate parameters and identify the major source of this "normal abundance" type of contamination.

The determination of the ratio ("sputtering ratio") of target to bombarding species in the sputtered material within collection pockets is receiving increased attention. Initial measurements made with uranium bombarding copper have been extended to include determinations of lead on graphite, iron on graphite, and iron on copper. At the same time, retention of ionic species on the bombarded target area can be determined. In the collection of multigram quantities, the ratio of the target to the bombarding species in the sputtered material remains relatively constant for any given pair of materials, whereas the percentage of bombarding species remaining on the bombarded area is inversely dependent on the quantity entering the pocket.

Ion source developments have included refinements to the newly constructed low-temperature unit and the development of a movable ionization filament which in limited tests has essentially doubled the length of calutron runs in the separation of the calcium isotopes. The concept involves an elongated filament which is moved manually at fixed intervals to ensure equal positive ion erosion of two emission areas, each identical in size with the area utilized by a conventional filament.

Following the successful separation of the isotopes of osmium, efforts have been directed toward achieving improvement in future platinum, palladium, and iridium separations. Initially these elements were processed in an ion source heated to > 2500°C (for iridium) by electron bombardment. Present approaches include the direct chlorination of these elements in an all-quartz, charge-oven, arc-chamber assembly heated by resistance-type graphite heaters. Unit construction is complete, but test runs have not yet been started.

Special Separations. — Decontamination of the outmoded plutonium processing equipment has continued. Since the present plutonium area will continue to serve for shipping and storage of return samples of Pu, Np, Th, U^{233}, and U^{234}, preliminary plans have been formulated to provide a new exhaust system for this area.

CHEMISTRY RESEARCH

Thorium Oxide Studies. – A 3200-hr irradiation in the LITR at 250°C and at a thermal flux of 2×10^{13} neutrons cm^{-2} sec^{-1} was completed on a settled bed of thoria pellets (50) in D_2O. The pellets were prepared by treating 1400°C-fired Davison Chemical Company pellets with dibasic aluminum nitrate and refiring at 1750°C. When the irradiation autoclave was opened, 20 of the pellets were found to have broken into fragments and fines. The remainder had essentially the same bulk density as the original material, but showed some damage from chipping out of surface fragments and breaking off of semispherical end portions. All surfaces appeared to be quite porous. A substantial quantity of a black vitreous substance was found in the upper half of the irradiation autoclave. Pellets removed from a laboratory control experiment showed no apparent damage, and there was no evidence of the black vitreous material.

Gas recombination experiments with a thorium–0.5% uranium oxide slurry containing a palladium catalyst (pumped loop sample L2-26S-6-4) in which the initial oxygen partial pressure was maintained at 200 psi (280°C) and the deuterium partial pressure raised from 50 to 700 psi showed a one and one-half order dependence of the initial recombination rate on deuterium partial pressure. Only partial recombination of the gas mixture was observed in all experiments, the per cent recombined increasing as the ratio of hydrogen to oxygen in the system (slurry + gas) approached the stoichiometric $H_2 + \frac{1}{2}O_2$. In experiments where the deuterium partial pressure was maintained at 200 psi and the oxygen partial pressure varied from 50 to 400 psi, a zero order of initial recombination rate dependence on oxygen partial pressure was observed. After each recombination experiment the reaction autoclave was cooled to room temperature, the residual gases removed, and the autoclave resealed. The autoclave was then heated to 200°C, the oxygen added first, and then the deuterium.

Equipment Decontamination. – In the pH range 0 to 9, oxalate-peroxide decontaminating mixtures at 95°C showed a maximum peroxide life of several hours at a pH of about 4.5. At lower pH, oxalate was oxidized to CO_2 and water. Above pH 6, the peroxide decomposed to oxygen and water.

The rate of corrosion of stainless steel by 1% H_2SO_4 (0.3 N) at 95°C was reduced to 0.001 mil/hr by the addition of 3% H_2O_2 (1 M). The mixture proved to be an excellent decontaminating agent for stainless steel. The best solutions for decontamination from niobium included combinations of oxalate, peroxide, and fluoride. The corrosive effect of the fluoride was greatly reduced by peroxide. Ruthenium decontamination tests after alkaline permanganate pretreatment showed oxalic acid and hydrogen peroxide preparations to be more beneficial than sulfuric or nitric acid, fluoride, or several proprietary compounds.

Deoxidine 170, a proprietary inhibited phosphoric acid metal cleaner, was used to remove red scale from an HRT rupture disk with an activity of 200 r/hr at 1 ft. Ruthenium and niobium were then removed by alkaline permanganate and oxalic acid–hydrogen peroxide solutions. The final decontamination factor was 330, and the remaining activity as analyzed by gamma spectrometry was apparently all due to Co^{60}.

Gas loop piping contaminated with I^{131}, Ru^{103}, Ru^{106}, and Ce^{141} from air loop tests with unclad UO_2 in the LITR was furnished by General Electric

for decontamination tests. Oxalic and nitric acids with added H_2O_2 were the most effective of several reagents for this application.

Radiation Chemistry of Organic Moderators. – Work has been started on a study of the radiolytic and pyrolytic decomposition of organic materials potentially useful as moderators or coolants. The project has been limited so far to the design of equipment and the planning of operations and techniques necessary for the study. In addition, the literature survey has been continued and expanded to include additional topics concerned with high-temperature reactions of hydrocarbons. It has been possible to obtain chromatographically pure biphenyl to use in the experimental runs. Laboratory work will start with the study of the pyrolytic decomposition of biphenyl; major interest will be centered about the rates and mechanisms of reactions involved. Decompositions of 0.1 to 0.5% conversion are of interest in order to limit the reactions to those concerned in the initial decompositions of the hydrocarbon. The products will be isolated and identified primarily by gas and vapor chromatography, with the use of infrared and ultraviolet spectrophotometry when desired.

Analytical Chemistry Research. – The extraction of zirconium from acid solutions with tri-n-octylphosphine oxide (TOPO), tris(2-ethylhexyl)-phosphine oxide (TEHPO), and di(2-ethylhexyl)phosphoric acid has been further investigated. Test results obtained with the radiotracer Zr^{95} revealed that micro and macro amounts of zirconium have identical extraction characteristics. The low degree of extraction with TOPO and TEHPO previously reported for zirconium in sulfate solutions was found to be due to the presence of nitrate. In the complete absence of nitrate, zirconium is readily extracted from 0.2 to 7 M H_2SO_4. In the extraction of zirconium from sulfate media with mixtures of the neutral phosphine oxides and di(2-ethylhexyl)phosphoric acid, no enhancement is achieved over the extraction attainable with the individual compounds.

Improvements have been made in equipment and techniques for use in studying the absorption spectra of molten fluoride salts. A small resistance furnace, consisting of a graphite tube 1-1/8 in. in diameter and 2 in. in length, was designed and fabricated to replace the furnace presently in use for heating the spectrophotometric cell assembly. The improved furnace can be heated to temperatures above 1400°C. It is useful not only for attaining high temperatures but also for providing more uniform heating within the temperature range from 400 to 1000°C than was formerly possible. A method has also been developed whereby a known amount of a powdered salt can be loaded into a platinum tube at room temperature for subsequent melting and spectrophotometric study of a pendent drop. Among the spectra which have been recorded are those of CoF_2 in molten LiF over the temperature range from 850 to 950°C and also the near-infrared spectrum of UF_4 in a molten LiF-NaF-KF eutectic over the wavelength range from 1.2 to 2.5 μ.

Inorganic Solution Chemistry. – Zirconium phosphate and similar acid salts have the property of adsorbing cesium extremely strongly, particularly from acidic solutions. Under these conditions many higher valent ions such as the alkaline earths are not significantly adsorbed. Advantage may be taken of this property to remove the short-lived (2.6-min) Ba^{137} from its long-lived parent Cs^{137} by a convenient "milking" procedure.

In a search for adsorbents for Ba^{137} from these acidic solutions, barium sulfate was found to be satisfactory. The radioactive Ba^{137} presumably exchanges with nonradioactive barium ions on the surface of the $BaSO_4$ particles. While the reaction is fast, precipitated $BaSO_4$ has too fine a particle size to be useful in columns. However, certain precipitates which incorporate barium sulfate – for example, a mixed precipitate of tantalum oxide and barium sulfate – can be obtained with adequate particle size, and columns prepared from such mixtures combine reasonable flow characteristics and exchange rates. A combination of these two adsorbents thus permits the continuous removal and readsorption of the short-lived Ba^{137}. In this manner a mobile radiation source can be constructed which decays with the half life of the short-lived Ba^{137}.

Organic Chemistry. – For the reaction of formic acid with chlorine,

$$HCOOH + Cl_2 \xrightarrow[20^\circ]{\text{visible light}} 2HCl + CO_2$$

the O^{18} isotope effect was found to have the value $k_{16}/k_{18} = 1.0022$ after correction for the effect of isotopic mass on collision frequency. This very small net O^{18} isotope effect indicates that abstraction of hydrogen from oxygen by chlorine atoms makes no appreciable contribution to the reaction mechanism. This confirms the conclusion drawn from earlier studies of the deuterium isotope effect, namely, that the rate of the reaction is determined by the rate of abstraction of hydrogen from carbon by chlorine atoms.

Radiation Chemistry. – The sensitivity of the elemental semiconductor germanium to radiation suggests its use in studies of radiation effects upon catalyst activity. As a preliminary to such a study, samples with different degrees of n and p character, produced by chemical doping, were used as catalysts in the decomposition of formic acid and in the H_2-D_2 exchange reaction. For both reactions the p-type samples were more active than the n-type. An apparently new observation is that the formic acid decomposes almost exclusively to $CO_2 + H_2$ on p-type germanium, but about equally to $CO_2 + H_2$ and to $CO + H_2O$ on n-type.

Chemical Physics: Microwave and Radio-Frequency Spectroscopy. – The paramagnetic resonance spectrum previously seen in gamma-irradiated single crystals of $NaNO_2$ containing a small amount of $AgNO_2$ was also seen in single crystals of $NaNO_2$ not containing $AgNO_2$, but with much less intensity. The anisotropic hyperfine tensor deduced from this spectrum was interpreted as arising from N^{14} in NO_2. The measured hyperfine interaction parameters are very satisfactorily accounted for by unpaired electrons occupying the 2s orbital of N, the 2p orbital of N directed along the intersection of the two mirror planes of NO_2, and the 2p orbitals of the oxygen atoms which are directed in the plane of NO_2 and are perpendicular to the line joining the oxygen atom with nitrogen. An interesting result of this interpretation is that negative spin density is associated with the nitrogen orbitals.

Chemical Physics: Molecular Beam Studies. – The molecular beam apparatus for studying the mechanics of the hydrogen exchange reaction was

substantially completed. Preliminary experiments were carried out in which a modulated beam of atomic deuterium (at 2500 to 3000°K) collided with a molecular hydrogen beam at 77°K. A small magnetic mass spectrometer which can be rotated about the scattering center was used to analyze the scattered beam. Scattered, modulated HD was observed, and work is in progress to ascertain whether this observed product is truly formed by the homogeneous crossed-beam reaction.

High-Temperature and Structural Chemistry. – In two preliminary test series, the electrical conductivity of cerium metal dissolved in cerium trichloride at various concentrations up to the saturation limit of 9.5 mole % was found to be 6 to 7 times greater than the conductivity reported by others in the recent literature. The discrepancy is considered to be due to the absence, in the present tests, of refractory oxides, the presence of which distorted the earlier data through the reaction $Al_2O_3 + 2Ce + CeCl_3 \rightarrow 3CeOCl + 2Al$.

Chemical Separation of Isotopes. – The infrared and Raman spectra of liquid $B^{10}F_3 \cdot (CH_3)_2O$ and $B^{11}F_3 \cdot (CH_3)_2O$ were examined. The measured isotopic shifts and their tentative assignments are shown below:

Frequency (cm^{-1})		Shift (cm^{-1})	Assignment
B^{11}	B^{10}		
322	322	0	> BF_3 deformation
344	344	0	
499	500	1	BF_3 deformation
661	672	11	B-O stretch
804	809	5	B-F stretch, symmetrical
918	925	7	C-O stretch
1174	1221	47	> B-F stretch, asymmetrical
1219	1258	39	

From these data, equilibrium constants of 1.045 at 25°C and 1.033 at 100°C were calculated for the isotopic exchange reaction

$$B^{10}F_3(g) + B^{11}F_3 \cdot (CH_3)_2O(\ell) \rightleftharpoons B^{11}F_3(g) + B^{10}F_3 \cdot (CH_3)_2O(\ell)$$

Transuranium Elements. – The proposed method for processing highly irradiated plutonium is to extract plutonium into tributyl phosphate from acid nitrate solution and then to neutralize the aqueous raffinate and re-extract with tributyl phosphate to recover americium and curium. Since the rare earths behave in the same way as the trivalent actinides, they will also be extracted. Distribution coefficients of cerium and europium between 30% TBP and aluminum nitrate solutions containing acid were studied

to determine the effects of aluminum and acid concentrations. Increasing the $Al(NO_3)_3$ concentration (no free acid) from 1 M to 2 M increased the europium distribution coefficient from 3.2 to 480 and the cerium distribution coefficient from 1.8 to 180. Americium and curium are more extractable than cerium but less extractable than europium, and the distribution coefficients decreased rapidly with increasing acid concentration. For example, when equal volumes of 30% TBP and 2 M $Al(NO_3)_3$ were equilibrated, increasing the acid concentration in the aqueous feed from 0.05 M to 0.6 M decreased the rare-earth distribution coefficients by a factor of 10. This is apparently a result of extraction of acid, since about 90% of the acid was extracted from the feed that was initially 0.6 M in HNO_3. Even though the presence of acid decreases extraction, small amounts of acid in the feed of a countercurrent contactor do not appear to be serious. This acid depresses the distribution coefficients only near the feed plate and in the scrubbing section. Only one or two extra extraction stages are required to accomplish the same rare-earth recovery when 2 M $Al(NO_3)_3$ feed is made 0.5 M in HNO_3.

METALLURGY AND MATERIALS RESEARCH

Preparation of Pure Single Crystals. — Spectrographic analyses indicate that satisfactory purification is being achieved in single crystals grown from slowly frozen LiF melts; the temperature gradient and the annealing technique are being altered in an effort to improve the size of the single crystals produced.

Alloy Theory Research. — X-ray measurements of the lattice constants of the hexagonal phase of zirconium with Ag, Cd, In, Sn, and Sb show the following values to within approx 1 part in 2000:

$$a = 3.2326 - 0.276x \quad ,$$

$$c = 5.1473 - 0.44x + 0.181xn - 0.016x^2n \quad ,$$

$$c/a = 1.5923 + 0.056xn \quad ,$$

where x and n are the atomic fraction and the valence of the solute in the alloy. The axial ratio is expanded in proportion to the number of s-p type electrons contributed by the solute. The fact that each solute electron has an equal effect suggests that there are very few s-p type electrons in solid zirconium and that the four outer electrons are d type. The same situation is suggested by the effects of Ag, In, and Sn on the electronic specific heats of hexagonal zirconium. The electronic specific heat coefficients also change with rates directly proportional to the solute valence. No change in either the axial ratio or the electronic specific heat would be expected if a solute atom made no net transfer of nearly free electrons to the alloy. Experiments are in progress to see whether palladium is a solute of this type.

X-Ray Diffraction and Metallographic Research. — Measurements of the temperature diffuse scattering for a powder sample of UO_2 have been made. The measurements were made at room temperature and have been converted to

absolute units. These data, combined with integrated intensity determinations from the Bragg maxima, should allow a determination of the Debye temperature for this material.

Metal Surface Studies. – The investigation of the oxidation properties of the refractory metals has been continued with a study of tantalum single crystals. The (100) planes of tantalum were found to oxidize most slowly, whereas the most rapid rates of oxidation were exhibited by the (111) and (110) planes.

In agreement with previously reported data for polycrystalline specimens, the oxide on the single crystals occurred both as a surface oxide and in the form of platelets which extended into the metal substrate. Other investigators have reported that the platelets formed parallel to (100) planes of the metal substrate. However, the present study has shown that, at least for specimens oxidized at 500°C and at an oxygen pressure of 1 atm, the platelets form parallel to the (320) planes rather than to a major crystallographic plane of tantalum.

Fundamental Investigation of Radiation Damage in Solids: Electron Spin Resonance of Irradiated TiO_2. – The electron spin resonance of a defect in the rutile form of TiO_2 produced by neutron irradiation has been observed at a temperature of ~300°K. The spin state, S, and the principal values of the g tensor appear to be similar to those observed for a paramagnetic center in reduced TiO_2. There are differences, however, in the orientation of the principal values of the g tensor and in the components of the spectra. Another difference is that temperatures below 20°K are necessary for the observation of the centers in the reduced material. The results of the measurements suggest that the center in the neutron-irradiated material may be a trapped electron in an oxygen vacancy. In this case the paramagnetism could arise from Ti^{+++}. A comparison of the above results with those on Ti^{+++} in the alums indicates that the spin-lattice relaxation time is much longer. The line width at 300°K is only 1 oersted. In the alums $g \sim 1.2$, whereas in TiO_2 $g \sim 1.96$, suggesting that the orbital moment is more strongly quenched.

Fundamental Investigation of Radiation Damage in Solids: Recoil Effects in Isotopically Enriched Germanium. – Germanium dioxide having an isotopic enrichment of 95.8% Ge^{74} was obtained from the Isotopes Division, Oak Ridge National Laboratory, by personnel of the Bell Telephone Laboratories, and was fabricated into a high-purity single-crystal n-type ingot of germanium. Hall coefficient plates have been cut from a 1.6-g section of this ingot, supplied by the Bell Telephone Laboratories, and thermal-neutron irradiation experiments have been conducted. The material is more n-type after irradiation than before, which indicates that recoil subsequent to thermal-neutron absorption and capture-gamma-ray emission does not produce multiple defects. Vacuum heat treatments, at 450°C, further increase the net electron concentration, and the final result agrees almost exactly with the net increase expected from published cross-section data.

CONTROLLED THERMONUCLEAR RESEARCH

Arc Research. – An anomaly in ion drifts toward the cathode of the magnetically confined carbon arc has been cleared up. Earlier measurements indicated that in the anode region C^{++} ions had 4 ev drift energy toward the cathode but that this drift was essentially zero near the cathode. It has been found that ions at the cathode end have a drift energy of about 27 ev toward the cathode in the peripheral region of the arc but still negligible drift in the core of the arc. This has been verified by making Doppler studies of spectral line profiles in which only the peripheral region was viewed and comparing them with studies in which the core and the periphery were viewed. The quantitative evaluation of the line profile associated with the core plus the peripheral region also reveals a slight asymmetric increase in intensity to long wavelengths with respect to a true Gaussian profile. This is to be expected if the ions in a weakly excited region (the periphery) are moving away from the spectral apparatus. It also suggests that the pumping characteristics of such magnetically confined arcs may be optimized by appropriate design of baffle configurations at the cathode end.

DCX-1 Facility. – The analyses of the energy-loss experiments have been continued.

With the neutral-particle spectrometer adjusted to detect particles in a narrow energy interval, it has been observed that the detector output is sharply sporadic. That this is a real effect has been verified by using several electron multiplier detectors and a scintillator-photomultiplier combination.

Investigations of the plasma radio-frequency spectrum (with gas breakup) have been extended to include work with a single unshielded loop dipole, an electrostatically shielded dipole, and a double loop probe. The spectra observed with these probes were much alike. Relatively little dependence of the spectra on the orientation of the loop axis was noted, but dependence on the radial position of the loops was observed. Oscillographic displays of the total probe rf output as a function of time after the H_2^+ beam was cut on characteristically show an initial quiescent period followed by a smooth gradual amplitude increase that terminates in turbulent modulation. At least in one set of observations the quiescent interval varied as $(I_{H_2^+})^{-0.8}$ and the maximum amplitude before turbulence varied approximately as the H_2^+ current.

The light output from the plasma region (again with gas breakup) has been studied by use of a photomultiplier and synchronous detection techniques. The spectrum from 0.15 to 1000 Mc was investigated. A broad proton cyclotron band and several other frequency groups were noted.

The gas-arc work has been extended to trials of several rotating water-cooled anode structures, with the result that some progress has been made in the problem of dissipating the anode heat.

Larger Magnetic Facility. – The theory of plasma buildup in the DCX-2 has been extended to include the details of plasma accumulation with an arc and also to include energy losses, time dependence, and other effects.

By use of the most probable values of all parameters, the critical input current without arc is expected to be 500 ma; with an arc it is expected to be 150 ma. The calculations to date do not include the effects of an anomalous energy dispersion such as that observed in the DCX-1.

The general layout of the machine is now well established. Molecular ions will be injected at a point between the booster coils and the mirror coil. The region of uniform magnetic field will be approximately 2 m long, or half the distance between the mirrors. Detailed calculations show that it is possible to maintain a mirror ratio of 3.5, a 2% field dip at the injection point, and uniformity over the center region to a few parts in 10^4. The electrical and mechanical parameters of the coils are fixed, and the structural design is proceeding.

The 18-in.-long molecular-ion accelerator to be used on the DCX-2 has been operated at 630 kv with beam currents up to 70 ma.

The principle of the magnetically shielded injection snout has been tested for magnetic performance. Operation of the snout provides adequate field reduction inside and field perturbation of less than 0.5%, 2 in. from the end of the snout.

Vacuum. — The metal-vapor pumping technique has been applied in an experimental tube which simulates the entrance or exit tube for the high-energy beam of ions in DCX-like devices. In practice it will be desirable to isolate the ion source region (operating pressure approximately 10^{-6} mm Hg) from the central region, where the pressure may be as low as 10^{-8} mm Hg. It was found that the titanium vapor deposited on the inside of a series of cells could be used to differentially pump a beam tube passing through them. An over-all length of 20 in. was sufficient to permit a pressure difference of 100 (2×10^{-6} to 2×10^{-8} mm Hg) between the ends of a 2-in.-diam beam tube for greater than 2 hr.

Plots of the vapor-stream temperatures have been made for three configurations of the jet bead in a 20-in. oil diffusion pump of the Distillation Products Industries design. It has been definitely shown that the presence of oil (vapor or liquid) leaks in the jet assembly other than through the jet itself can produce large distortions in the jet pattern, accompanied by effects on pumping speed, steadiness of pressure, and oil back-streaming.

The Consolidated Electrodynamics Corporation model 21-620 mass spectrometer has been attached to the DCX, and a large number of runs were made at pressures between 5×10^{-5} and 5×10^{-7} mm Hg. For most of the runs the DCX had been baked to less than 200°C and the mass spectrometer was connected to the inner liner of the DCX by a 4-ft unbaked copper tube. The impurities in the system were observed to be primarily mass 18 (water vapor) and mass 28 (nitrogen or carbon monoxide) at all pressures, with an indication that methane is also present. At pressures above 10^{-5} mm Hg the impurities were less than 1% of the pressure, but increased to more than 10% at pressures as high as 5×10^{-6}, depending upon the particular run. Base pressures, of course, were predominantly due to these gases.

The activities in the field of vacuum chemistry included the assembly and operation of the specially designed equipment for study of metal-film pumping systems. Trial evaporations of titanium have been made with

evaporation rates greater than 0.05 mole/hr. The base pressure after titanium evaporation in this vacuum system was observed to be about 5×10^{-7} mm for a period of 7 hr, with only cold-water baffling of the diffusion pumps.

A synthetic zeolite (13X) was tested as a baffling material and found to maintain a pressure of 4×10^{-8} mm for several hours. These results are ascribed to greatly enhanced pumping speed of this material for water vapor after vacuum baking.

Apparatus has been obtained for the purpose of leak testing large electrical insulators of unglazed porcelain.

BIOLOGY AND MEDICINE PROGRAM

BIOMEDICAL PROBLEMS IN ATOMIC ENERGY OPERATIONS

Waste Disposal Research and Engineering: Evaluation of Soil Disposal. — A vermiculite-filled column 10 ft in length and 2 in. in inside diameter is being evaluated for the removal of Cs^{137} and Sr^{85} from simulated ORNL intermediate-level waste. The vermiculite was treated with potassium in order to induce lattice collapse, which improves cesium sorption. The influent was made 10^{-3} M in KH_2PO_4; the potassium maintains a collapsed lattice structure, and the phosphate improves strontium removal.

To date, 175 liters of solution has passed through the column at a flow rate of 40 ml/min. This is equivalent to ~80,000 gal of waste pumped through a 7-ft-diam column. No Cs^{137} or Sr^{85} has been detected in the effluent. Survey meter readings at the exterior surface of the column show that little or no activity has passed the 5-ft point and indicate that the column is less than 25% saturated. Extrapolation of data from small-scale laboratory columns indicated that approximately 1000 liters of solution could be passed through the column before 50% breakthrough, and the results seem to agree quite closely.

Ecology and Marine Studies. — Twenty-nine wild small mammals from the White Oak Creek drainage system were analyzed to provide preliminary data on the bioaccumulation of radionuclides. These data are serving to characterize the position of the mammals in relation to other organisms in the cycling of radioelements. A two-year study of the population dynamics of the small mammals of White Oak Lake bed is supplying the basic information for extrapolation of the analytical data to the total number of animals on the lake bed.

The most abundant radionuclides found in the mammals, listed in order of decreasing abundance, are Sr^{90}, Ru^{106}, Cs^{137}, and Co^{60}. Detectable amounts of Ce^{144}, Zn^{65}, Zr^{95}-Nb^{95}, Mn^{54}, and Cs^{134} were also present in some of the animals. The quantities of the four most abundant radioisotopes in the mammal population on a unit area basis (microcuries per square meter) are: Sr^{90}, 6.3×10^{-5}; Ru^{106}, 1.7×10^{-5}; Cs^{137}, 1.3×10^{-5}; Co^{60}, 0.90×10^{-5}. These preliminary estimates, when compared with Sr^{90} and Cs^{137} previously determined for herbivorous insects and passerine birds

of the lake bed ecosystem, show that the amount of the Sr^{90} in the mammals is three times as great as in the insects and 36 times as great as in the birds, whereas the amount of Cs^{137} is approximately three times as great in insects and mammals as it is in birds.

ISOTOPE DEVELOPMENT PROGRAM

Recovery of Fission Products and Transuranics from Waste. — The Fission Product Recovery flowsheet for extraction of strontium and rare earths from Purex waste by di(2-ethylhexyl)phosphoric acid (D2EHPA) was tested with nonradioactive solutions in miniature mixer-settler equipment in order to observe the stability and physical operability of the chemical flowsheet prior to hot-cell tests in which high-level Purex waste will be used as feed. Physical operability was excellent, with no emulsions or precipitates, throughout the runs covering the first, second, and third cycles.

In the first cycle, strontium and rare-earth concentrations were too low for reliable analyses, so that recoveries and losses were not determined accurately. Iron separation was better than predicted; after 17 hr of operation, 4.8% of the iron in the feed was being extracted but only 0.02% was being stripped into the first-cycle product, to yield an iron separation factor of 4.4×10^3. Previous experiments indicated a first-cycle iron separation factor of 1.4×10^3.

In the rare-earth—strontium partitioning and the rare-earth stripping steps, which comprise the second cycle, 98.2% of the rare earths was recovered as product, with 1.8% of the rare earths being lost to the used organic stream. Separation factors were: rare earths/sodium, 9×10^3; rare earths/iron, 130. Strontium in the rare-earth product was below the level of analytical detection.

In the third cycle, essentially 100% recovery of the strontium may be assumed, since strontium losses in the raffinate and used solvent were below the detection limit. From iron analyses, 0.8% of the iron in the third-cycle feed was present in the strontium product; the strontium/iron separation factor was 108.

Radioisotope Research and Development. — The Sr^{90} recovery run in progress at the Fission Product Pilot Plant has yielded approximately 226 g of $SrTiO_3$ powder to date. Two pellets, weighing 65.35 and 65.15 g, respectively, were fabricated from the purified powder. During the sintering process both pellets developed cracks, but they are still usable in the assembled thermal source. The heat output of the two pellets was measured calorimetrically; the values were 0.1012 and 0.1011 w/g respectively. On the basis of a preliminary determination by the Analytical Chemistry Division that the activity of the $SrTiO_3$ powder is 16 curies/g, the thermal output of Sr^{90} is 6.3 w/kilocurie. The concentration of Sr^{90} in the product $SrTiO_3$ will be confirmed by an additional series of analyses.

Radioisotope and Stable Isotope Production. — Charge materials were prepared for the separation of the isotopes of Cd, Ca, Ga, Os, Ta, and Fe. Anhydrous fused nickel chloride and dysprosium chloride have also been prepared for separations in the expanded program, although the collections have not yet been started. Small quantities of $NiCl_2$, $MgCl_2$, and

iridium metal have been weighed into charge containers for developmental work. The chemical recovery of osmium, which was used as charge material but was not successfully resolved into its isotopic components, has continued and has been expanded to included calutron wash solutions and diffusion pump and Kinney pump oils, as well as metallic and graphite parts. The major portion of the osmium appears to be associated with the graphite parts and is currently being removed by oxidation, then subsequently regained in the metallic form by zinc reduction of the hydrobromic acid trap solutions. Collections of the isotopes of Ca, Fe, Os, Ta, Pb, Ga, and Cd were made, with ion currents equivalent to 2550 g. Separations were performed in the four calutrons in Building 9731 and in 16 of the calutrons in Building 9204-3.

Eight new lots of the separated isotopes of Cr, Fe, Pb, and Os and three returned lots of Nd and Ag were recovered, refined, and prepared for inventory. Included are gram quantities of Os^{189} and Os^{192}, which were enriched in the ORNL mass separators for the first time.

Eighty-four samples of Ca, Pb, Os, Ta, and Ga were recovered on a preliminary basis for study of retention, mass analysis, sputtering, and other developmental data.

Conversions of inventory forms to more suitable forms included $Ge^{73}O_2$ to Ge^{73} metal, $NaCl^{35}$ to $AgCl^{35}$, $NaCl^{37}$ to $AgCl^{37}$, Ni^{61} metal to $Ni^{61}O$, Li^6 metal (96.1%) to Li^6F, Li^6 metal (99.3%) to $Li_2^6CO_3$, and Li^6 metal (96.1%) to $Li_2^6CO_3$. One sample of Hg^{202} stocked in the form of aqueous nitrate was sealed in a Pyrex ampoule for distribution. Small samples of $Sm_2^{144}O_3$ and $Sm_2^{147}O_3$ (35 and 46 g respectively) were encapsulated in quartz ampoules for irradiation.

The recovery of the enriched Pb^{208} recycle was completed, and the Pb^{210} product was recovered.

Several 500- and 700-level uranium batches have been purified and combined to simplify inventory listing and reduce the number of batches on hand.

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