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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

APRIL 1961

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

APRIL 1961

DATE ISSUED MAY - 4 1961

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

APRIL 1961

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

Chemical Plant Criticality Studies. – The tests designed to establish safety practices for 5-3/8-in.-OD, ~13-liter-capacity, cylindrical polyethylene containers of enriched uranyl nitrate solution were continued. The solution had a concentration of 410 g of 92% U^{235}-enriched uranium per liter. Ninety-eight containers were arranged in two tiers, each a 7 × 7 square array, separated 5.6 in. vertically. The assembly was critical with a horizontal surface-to-surface separation of the containers equal to 8.34 in. This separation can be compared with 7.8 in. between the 81 units in a critical 9 × 9 single tier.

A linear array of containers, axes vertical and centers collinear, was constructed adjacent to a 6-in.-thick hydrogenous reflector. Nineteen containers with sides in contact were critical in such an array.

It was further observed that, whereas a 6 × 6 array of containers spaced 5.64 in. surface-to-surface without reflector or moderator between the units was critical, submerging the entire array in water made it significantly subcritical. Application of a 66.8-liter/min water spray from a fog nozzle to an otherwise unmoderated and unreflected array, a simulation of a fire-fighting practice, increased the reactivity by an amount approximately equal to the value of one container on the periphery of the array.

Dissolver Solution Analyses: Uranium. – A method is being developed by which uranium(VI) can be determined by coulometric titration in the presence of relatively large amounts of iron. The pretitration, which reduces Fe(III) to Fe(II) and thus eliminates the interference of the cation, takes place at a platinum cathode at a potential of +0.05 v vs the S.C.E. The U(VI) is subsequently reduced at a mercury cathode at a potential of –0.30 v vs the S.C.E. By use of the two-electrode system, it is hoped that an iron-to-uranium weight ratio in the order of 50 to 1 can be titrated.

A synthetic APPR type of dissolver solution was analyzed for uranium in a hot cell of the High-Radiation-Level Analytical Facility. Uranium was separated from other metals in the stainless-steel-type dissolver solution by use of an anion resin column. Uranium was adsorbed on the resin

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from a sulfate medium, converted to the chloride form on the column, and eluted with dilute HCl. The uranium was then titrated coulometrically in a sulfate medium. A relative standard deviation of 0.76% for the synthetic sample containing 5.04 mg of U per ml was obtained for 25 replicate determinations.

Dissolver Solution Analyses: Plutonium. – Studies have been made of the use of dilute HF and HF-HNO_3 solutions as elution agents for milligram and submilligram quantities of Pu(IV) from anion exchange resins (complexed as the nitrate). The optimum HF concentration was found to be 0.01 to 0.03 M; HNO_3 was apparently not required.

The use of boric acid, cerium, and aluminum as complexing agents for the fluoride ion was studied in order to eliminate the interference of free fluoride in the controlled-potential coulometric titration of plutonium. Cerium(IV) and aluminum were found to be useful for small amounts of fluoride, while boric acid did not eliminate the interference of even small amounts of fluoride.

Application of the controlled-potential titration of plutonium to analysis of dissolver solutions of Al-Pu alloy fuels is being studied. These solutions consist of Al, Pu, Hg, F, and Fe in various strengths of HNO_3. A supporting electrolyte of 0.2 M $AlCl_3$–0.2 M HCl is used for this type of sample (Al complexes F, Cl complexes Hg); the E°_{Pu} is 0.71 v vs the S.C.E. To minimize the interference of iron, reduction of plutonium is made at +0.59 v vs the S.C.E.; oxidation of plutonium is made at +0.89 v vs the S.C.E.

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

Irradiation Program. – Bulk-density measurements by mercury immersion were made on UO_2 fuel samples from ten ORR-irradiated prototype capsules. The small postirradiation decreases noted could be attributed to microcracking of the UO_2, which was observed in most cases. Since the precision of measurement was estimated to be about ±0.07 g/cm^3, the observed density changes, which ranged from +0.01 to –0.28 g/cm^3, were generally insignificant.

Metallographic examination of the UO_2 from group II ORR-irradiated capsules revealed a generally variable grain size, with many voids left by pull-out of a second-phase material during polishing. A similar second-phase material in control samples was identified as UN_2 by x-ray diffraction techniques. Two pellets from capsule 06-2 exhibited very fine grain size at the pellet-to-pellet interface.

The release of xenon, krypton, and iodine from the UO_2 sample now being irradiated in the LITR in the instantaneous fission-gas-release experiment is increasing with time at a constant flux and a moderate temperature. This may be due to thermal cycling or to structural changes in the UO_2. The simultaneous release of Xe^{133}, Xe^{135}, Xe^{138}, Kr^{85}, and Kr^{85m} is being studied in an effort to establish the holdup times for these gases in the fuel.

Three experimental assemblies containing tube-burst specimens of type 304 stainless steel (Superior Tube Company heat No. 23999x) were disassembled; they had been irradiated in the ORR at a flux of $\sim 10^{13}$ neutrons cm^{-2} sec^{-1} (above the sulfur threshold). In comparison with data from out-of-pile control tests, the data obtained with these specimens indicate that at test temperatures of 1300, 1500, and 1600°F, the ductility at rupture is reduced by a factor of 2 to 10, depending on temperature and stress. It is important that at 1500 and 1600°F, the strain at rupture decreases with decreasing stress (increasing time) and approaches 1%. At 1300°F, ruptures frequently occurred with less than 1% average strain. Only at 1500°F is the rupture strength of this material reduced by any significant amount.

Beryllium tube-burst specimens are being irradiated in the B-8 core position in the ORR at 700°C. The stress-rupture data indicate about the same effect of the exposure as that observed at 600°C. There is considerable scatter, however, in both the in-pile and out-of-pile data. For tubes machined from hot-pressed block, the reduction in the 100-hr rupture strength at a dose of 1.8×10^{19} fast neutrons/cm^2 (> 1 Mev) is approximately 23% in terms of stress. Interpolation of the 600°C data yields a value of 24% reduction in the 100-hr strength.

Metallographic examinations of beryllium specimens irradiated at elevated temperatures have indicated that when swelling occurs, large bubbles, $> 10^{-3}$ cm in diameter, appear. These bubbles are rather widely spaced, indicating that the helium moved considerable distances to pressurize them. In addition to the large bubbles there are numerous small cavities about 10^{-5} cm in diameter or smaller. These are of the size where surface tension forces should restrain the swelling. In the case of the larger ones, however, these forces are small and the gas should cause creep of the beryllium and swelling.

A fourth UC_2-graphite matrix, graphite-encapsulated fuel element is being irradiated in the MTR. The test will be terminated with the second reactor cycle because the high purge-gas activity indicates that the element is not retaining fission gases.

Reactions of Type 304 Stainless Steel with CO-CO_2 Mixtures. — It was recently determined that type 304 stainless steel exhibits an accelerated rate of oxidation after 500 hr at 1800°F in an atmosphere in which the CO_2-to-CO ratio is 0.669. Based upon previous tests, it appears that for this temperature the breakaway reaction will occur with CO_2-to-CO ratios between 0.46 and 0.669.

A subsequent test was conducted in an atmosphere of 0.245 vol % CO_2 and, as expected, the breakaway reaction commenced after 100 hr at 1800°F. Test specimens arranged in a temperature gradient showed that the oxidation rate increased significantly above 1715°F. The oxide scale formed above this temperature was black, crystalline, and magnetic. Oxide whiskers were observed at random locations on these specimens. Formation of the oxide $3Cr_2O_3 \cdot Fe_2O_3$ is thought to be the cause of the breakaway reaction.

Graphite Degassing Studies. — Specimens machined from the central region of a 6-in. slab from block No. 4 of the EGCR graphite were degassed from room temperature to 1000°C, using external resistance heating. The specimens were 1, 1-1/2, or 2-1/4 in. in diameter and 2 in. in length. A

total gas volume of ~14 cm^3 per 100 cm^3 of graphite was found for these specimens, with no significant difference per unit volume for the different sizes evident. All released ~0.1 cm^3 of SO_2 per 100 cm^3 of graphite between 300 and 600°C. No significant effect of geometry on the volume of gas released as a function of time was apparent at 300, 600, or 1000°C. Typical expressions for the release of gas are: (1) $V = 0.090 \log t + 0.11$ at 300°C and (2) $V = 0.53 \log t + 0.70$ at 600°C, where V is expressed as cm^3 of gas per 100 cm^3 of graphite and t is given in minutes. Similar treatment of the data obtained at 1000°C results in a curve which is characterized by different slopes during the course of the degassing. In one run, for example, the data fitted the expression $V = 3.0 \log t - 1.4$ for the time interval 150 to 350 min, whereas during the period from 1250 to 5600 min, the equation $V = 5.5 \log t - 8.8$ was applicable. No consistent effect of geometry was evident.

Graphite-Metal Compatibility Studies. — Forced-convection loop No. 5 containing type AGOT graphite, metal test specimens, and helium has now completed 650 hr of operation with a test section temperature of 1400°F, a system pressure of 30 psig, and water injection at a rate of approximately 0.1 cm^3(liquid)/hr. The H_2, CO, CO_2, and H_2O impurity levels resulting from the water injection have been maintained at or below those specified for the EGCR for the entire period by use of a small-scale purification system.

Coated-Particle Fuel Element Development. — The development of processes for applying coatings of high density and low permeability to individual fuel particles has enhanced the feasibility of producing practical ceramic fuel elements containing oxide-coated particles of UO_2 or pyrolytic-carbon-coated uranium carbides. Preirradiation evaluation studies of coated-particle fuel elements are in progress that will provide a basis for evaluating these materials under reactor operating conditions. Fission-gas-retention studies and irradiation experiments have been initiated. Compatibility and diffusion studies are being planned, and chemical processing methods are to be investigated.

GCR-ORR Loop No. 2. — The cell in-leakage test preceding installation of equipment in the cell has been completed. In-leakage was found to be 26 std ft^3/day with a pressure difference not less than 1.86 psi. The criterion for total cell in-leakage was established as 42.5 std ft^3/day at a pressure difference not less than 1.5 psi.

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. — On April 6 the HRT resumed operation at its full power of 5 Mw (1400 psig, 270°C in the core, 230°C in the blanket). The power was maintained at this level for more than 200 hr during the month. Throughout this time no indications of fuel instability could be detected. Although there have been no serious difficulties, steady operation was upset several times because of poor feed-pump performance.

The fraction of power generated in the reactor blanket region increased from 50% to 65% of the total power while the reactor was subcritical on April 17. Fuel samples indicated that the blanket concentration had increased accordingly and that the mixing between the core and blanket regions was greater by several pounds per minute. The reasons for the shift are not known at this time.

The experimental program will continue until May 1, when the reactor is scheduled to be shut down permanently.

Thorium Blanket Studies. – Efforts to prepare smooth, rounded, attrition-resistant 1/8- to 1/4-in.-diam thoria particles of high strength and integrity were continued.

Thorium oxides prepared by partial denitration of thorium nitrate with superheated steam at 320 to 450°C had crystallite sizes from 50 to 90 A and gross N/Th atom ratios from 0.35 to 0.08. In 2 to 4 M hydrosols prepared from these oxides, particle sizes and N/Th ratios varied, but homogeneity was improved by aging for 14 to 40 hr at 90°C. For particle strength and integrity of evaporated gel fragments fired at 1150 to 1250°C, the optimum N/Th ratio of the sol appeared to be from 0.13 to 0.15.

In supercentrifuge studies, sols with higher N/Th ratios deposited soft flocs which had an N/Th ratio of ~0.14 and formed glassy, strained fragments when fired; those with lower ratios gave a nonhomogeneous deposit which fired to chalky, weak fragments. For the sols with an N/Th ratio of 0.13 to 0.15, the pH of the supernatant was > 3 but < 4, the nitrate concentration was ~0.005 M, and the thorium concentration was $< 10^{-4}$ M.

Chemical Research for Aqueous Homogeneous Reactors. – In a study of the system UO_3-SO_3-H_2O from 150 to 300°C, with particular emphasis on the nature of the UO_3 solid phases, the stable hydrates which were found corresponded to Dawson's et al. (J. Chem. Soc. 1956, 3531) $UO_3 \cdot 0.8\ H_2O$ orthorhombic I phase at 150°C, $UO_3 \cdot 1.0\ H_2O$ orthorhombic II phase at 225°C, and $UO_3 \cdot 1/2\ H_2O$ at 300°C. Their identities were established by comparison of x-ray diffraction patterns with those of Dawson et al. The solids were found to be stable at the respective temperatures in the presence of solution phases from 0.001 to 0.1 m in SO_3. Upon changing the temperature from 150 to 225 or 300°C, a very slow conversion of the solid phase over an 18-hr period was noted; the slowness of this conversion is believed to have been responsible for previous reports that $UO_3 \cdot H_2O$ rather than $UO_3 \cdot 1/2\ H_2O$ was the stable solid at 300°C in this and related systems.

Analytical Chemistry. – An amperometric method, previously described for the titration of thorium (ORNL CF-61-2-34), was adapted for the determination of copper and certain of the rare-earth elements in synthetic fuel and blanket solutions under test for homogeneous-reactor use. The titration with ethylenediaminetetraacetic acid (EDTA) is carried out in a medium buffered with a mixture of chloroacetic acid and sodium chloroacetate at a pH of 2.5 for copper and at 4.5 for rare-earth elements, utilizing Fe(II) as the indicator. Those elements of the rare-earth group which form EDTA chelates with stability constants of $10^{-17.5}$ or less (Tb, Dy, Ho, Er, Tm, Yb, and Lu) can be titrated satisfactorily by this method. For those samples containing from 3 to 8 mg of these elements and also for those containing 1 to 2 mg of copper, recovery by the amperometric method is, on the average, essentially complete.

Since this amperometric method can be readily adapted for remote operations, it provides a convenient means for determining copper in irradiated, homogeneous-reactor fuel or blanket solutions.

Slurry Circulation Experiments. — The 200B loop, containing a test section for measuring friction factors in turbulent flow, was operated at temperatures of 30 to 240°C with a 1600°C-fired thoria slurry. At a concentration of 1415 g of ThO_2 per liter, the observed friction factors were those predicted from the conventional "smooth tube" line. At concentrations of 800 to 1200 g of ThO_2 per liter, the friction factors agreed closely with the water calibration curve, suggesting that pipe roughness was influencing the momentum transfer at the wall. In every case the effective Reynolds number (to correlate with friction factor) was based on the estimated viscosity of a dispersed slurry at the temperature and concentration of the run.

Engineering Development. — The full-scale flow model of the HRT was operated further as a reactor simulator when coupled to an analog computer analyzing variations in injected salt conductivity. Analysis of the simulated neutron flux-time plots, comparing the frequency and amplitude of the fluctuations at several power levels, showed a very close coincidence to those of the HRT. It was concluded that the observed fluctuations in HRT neutron level were very probably caused by variations in flow of the jet in the lower hemisphere. It was established, also, that the fluctuations remained reasonable up to simulated core power levels of 5 Mw.

The model was also operated with a flow nozzle which can be installed by remote means into the HRT core. The preliminary tests of this nozzle are quite promising: The short-circuit fraction of the entrance jet is reduced from 30 to 16%. The maximum temperature rise in the core volume is reduced from 170 to 120% of average. Oscillations of flow within the core have natural periods of 2 sec or less, greatly reducing the predicted power fluctuations. Solids are not retained within the core. The use of the nozzle may permit an increase of HRT power by about 50%.

The spare HRT circulating pump was run in on the hot-test stand. During disassembly for postexamination, some scratches were caused in the lower radial pump bearing. It is believed, however, that the scratches will not impair the functioning of the bearing.

A 6.8-in.-long extension to the Omniscope used to inspect the HRT core was received from the vendor. The extension, which will go through the remaining screens in the core, was checked out and found to be optically satisfactory.

Design. — A remotely operated clamp was designed to reinforce a cracked tee in the HRT low-pressure system. The clamp removes any possibility of fracture of the tee under pressure loads which could occur during an emergency dump. Improved freezer coils, 10 in. long, were designed for isolating the damaged tee in the event that the coils installed initially proved to be undersized.

MOLTEN-SALT REACTOR PROJECT

MSRE Design. — The MSRE fuel piping layout was revised to allow the pump to be moved from the top of the reactor vessel. Stress analysis of the new layout gave acceptable values. A pump mounting scheme, which provides for necessary pump movement, was designed.

The reactor-vessel drawings are being revised as a result of the new pump location. The heavy INOR-8 shield plug has been removed from the head of the reactor. A 10-in.-diam outlet with a flange extension and a fuel outlet tee has been provided. The flanged pipe permits insertion of a plug which carries control-rod thimbles and an auxiliary access port for graphite sampling. These changes are being detailed. Control rods of articulated boron carbide slugs are being designed.

An electrical heater design is being prepared which incorporates pipe support in the integrated heater unit. The sampler-enricher is being relocated slightly as demanded by the new pump location.

A design for pipe heaters has been made that will permit straight, vertical removal of practically all heaters.

Structural design work continued. A change toward simplication of the radiator door hangings made necessary some changes in the structure associated with this area.

Design studies of the shielding layout for the drain-tank cell have indicated no obstacle to maintenance.

Designs for the maintenance-control-room shield windows, the maintenance-control-room layout, and the manipulator specifications were released for comment.

Component Development. — Measurements made on the one-fifth-scale fluid-dynamics core model indicate that the heat transfer coefficient at the center line of the reactor-vessel lower head will be approximately 800 Btu hr^{-1} ft^{-2} $(°F)^{-1}$, which is more than adequate.

The engineering test loop began operation, with the MSRE coolant salt being used as a flush salt.

The oxide film on the copper sample capsule was removed satisfactorily by holding it at 800°C in a hydrogen atmosphere for 4 hr. If not removed, the oxide would contaminate the salt sample.

An oxygen-removal section of the helium purifier was used to reduce the oxygen concentration in a helium stream from 100 ppm to less than 1 ppm, with flow rates of up to 4 liters/min. The oxygen-removal agent was titanium sponge operating at 1200°F.

The reactor-vessel heater test operated satisfactorily for seven weeks with no obvious heater deterioration. A 60-amp remote electrical disconnect used in this test also performed satisfactorily.

Remote Maintenance. — In work on remote furnace-brazing, induction heating and an inert-gas purge were used successfully in bringing a section of small pipe to brazing temperature without excessive oxidation. Satisfactory oxygen control during the temperature cycle was demonstrated by the good wetting of the braze metal.

The pump maintenance mockup was expanded to include the auxiliary piping, and tools are being fabricated to operate the flanges.

A 1-1/2-in. integral-ring-joint spring-clamp flange for use in the sampling system was cycled 69 times between room temperature and 375°F in the line, and was assembled 13 times without a measurable leak. A similar joint for 1/2-in. pipe went through 800 cycles above 1200°F and eight assemblies without trouble. As-built drawings were completed.

MSRE Procurement and Construction. – The preliminary proposal and the preliminary hazards evaluation report for the MSRE were submitted to the AEC.

Drawings and specifications were completed for the site preparation work to be done at Building 7503 by a cost-plus-fixed-fee contractor. Demolition and minor building alterations will begin as soon as a directive is received from the AEC.

Potential suppliers of INOR-8 plate, sheet, rod, pipe, and tubing for MSRE fabrication have been consulted on problems related to the procurement of these materials. Suppliers agree that INOR-8 which meets the specifications can be produced. Negotiations have begun for purchase of the rod, sheet, and plate, and invitations to bid on the pipe and tubing will be released soon.

Pump Development. – The test pump with a molten-salt-lubricated journal bearing has accumulated 9000 hr (one year on April 7) of continuous operation with $LiF-BeF_2-UF_4$ (62-37-1 mole %) at 1225°F. The pump was started and stopped 75 times during the year.

The Inconel centrifugal pump (PKP type) has accumulated 5100 hr of continuous operation with $LiF-BeF_2-ThF_4-UF_4$ (65-30-4-1 mole %) at 1225°F, 1950 rpm, and 510 gpm. Preliminary testing indicates back diffusion of Kr^{85} up the shaft annulus against nearly 4 liters/min of helium purge down the shaft annulus.

Performance data were obtained in water tests of the MSRE fuel-pump model equipped with an 11-in.-diam impeller. Baffles and splash shields were devised to provide satisfactory control of bypass flows into the pump tank.

The vendor has poured two castings of the volute for the MSRE fuel pump. The second casting was of improved metallurgical soundness but was not within dimensional tolerance; a third casting will be poured in accordance with modified casting patterns. Fabrication of components for the test stand of the prototype pump was completed except for two small items.

A program to compute thermal stresses in the pump tank was devised for the Oracle. Input functions of pump-tank temperature distributions to be used with the Oracle program were deduced from the GHT-code data for four operating conditions.

Chemistry. – Molten fluoride fuels are known to become contaminated with oxide from exposure to graphite containing the commonly encountered residual or surface oxides in the internal voids of the graphite. Since the graphite is not wetted or penetrated by the fuel, a gas-transport mechanism might seem plausible for conveying oxide, particularly since CO_2 and CO can be baked out of graphite. However, it now appears that neither CO_2 nor CO reacts with the fuel appreciably. In a recent experiment 430 liters (STP) of CO was bubbled through 50 moles of MSRE fuel at 600°C in a nickel container without alteration of the concentration of

ZrF_4, UF_4, or ThF_4; further, the nickel ion concentration of the melt remained at the initial value of 40 ppm.

The draining of MSRE coolant from annuli made of INOR-8 on the inside and graphite on the outside was studied. For 3-in.-diam assemblies, which simulated a cladding concept, a 1/16-in. annulus proved adequate for complete drainage in an oxide-free system. Exposure to air, with consequent precipitation of oxide and scum formation, interfered with drainage.

Permeation of 13 vol % of AGOT graphite by MSRE fuel, achieved with a 60-psi pressure and subsequent freeze-thaw cycles (simulating temperatures encountered during in-pile experiments), caused no significant change in the graphite structure as indicated by the electrical resistivity at room temperature.

During the past six months 1200 kg of purified MSRE experimental fuels and coolants have been produced. Another 300 kg will bring the supply in line with current requests. Impurities typically range (in ppm) as follows: Cr, 100 to 300; Fe, 225 to 250; Ni, 25 to 40; S, < 3; O, < 600. The oxide analyses apply to samples which have been exposed to dry air at room temperature and hence represent an upper limit.

In-Pile Tests. – The third MSRE graphite-fuel capsule test (ORNL-MTR-47-3) will be inserted in the MTR during the May 1 shutdown. Startup will be on or about May 5, 1961, and the experiment is scheduled for removal on July 24, 1961.

Reactor Analysis. – Preliminary estimates were obtained on the effect of INOR-8 cladding on the relative fuel-cycle costs of large-scale molten-salt reactors. The reactors considered were cylinders having a diameter and a height of 20 ft; the volume fraction of fuel in the core was 0.20, the salt contained 10 mole % ThF_4, and the clad reactor had 40-mil-thick tube walls.

The cladding resulted in increases of 55 and 80% over the fuel-cycle costs of the unclad reactor for 3- and 2-in.-diam fuel channels respectively. A range of 10 to 20 kw/liter in the core power density did not give a significant variation in the minimum fuel-cycle cost for either the clad or the unclad case.

Heat Transfer and Physical Properties. – A re-evaluation of the data for forced-convection heat transfer with the mixture $LiF-BeF_2-UF_4-ThF_4$ (67-18.5-0.5-14 mole %), using modified thermal properties, indicates a reduced data scatter. Measurements on the heat capacity and viscosity of the salt removed from the loop will begin shortly. The effect of viscosity on the calibration of a turbine-type flowmeter is being examined.

Measurements on the enthalpy of the coolant mixture $LiF-BeF_2$ (68-32 mole %) have been completed. For the solid (50 to 360°C), the enthalpy (in calories per gram) is given by the expression:

$$H_t - H_{30} = -6.22 + 0.225t + (4.24 \times 10^{-4})t^2 \ ;$$

and for the liquid (480 to 820°C) by:

$$H_t - H_{30} = 123.9 + 0.185t + (2.97 \times 10^{-4})t^2 \ .$$

The solid-liquid transition was not sharply defined, extending over the range 360 to 480°C; there are suggestions of at least two phase transformations in this region. The heat of fusion, evaluated between 360 and 480°C, was 151.4 cal/g.

No additional information is available on the viscosity, density, thermal expansion, or thermal conductivity of the molten salts.

Fuel Processing. – The solubility of BeF_2 in liquid HF was increased significantly by the presence of NH_4F. Both LiF and BeF_2 had a high solubility in solutions containing around 5 wt % NH_4F. The solution could not be readily separated from undissolved salt, since the suspended solids settled very slowly and were filtered from the solution with difficulty. The solubilities in HF containing 5 to 10 wt % NH_4F are high enough to be of practical interest for processing:

Concentration of NH_4F		Solubility			
		LiF		BeF_2	
wt %	moles/kg	g/kg	moles/kg	g/kg	moles/kg
0.92	0.25	71.7	2.7	34	0.72
1.77	0.48	107	4.1	53	1.1
4.34	1.17	103	3.9	60	1.28
17.2	4.65	86	3.3	56	1.2

The solubilities indicate that NH_4F increases the BeF_2 solubility by forming a soluble BeF_3^- or BeF_4^{2-} ion in solutions containing up to 5 wt % NH_4F. In the absence of NH_4F the BeF_2 solubility would be 25 to 30 g/kg, and the increase in BeF_2 solubility above this is approximately equal to the NH_4F concentration for the first two cases and somewhat smaller for the third. In each experiment an excess of LiF-BeF_2 (63-37 mole %) was present, and the solution was stirred at 25°C with a magnetic stirrer for at least one day.

ANP PROGRAM

Shielding Research. – Measurements were made of the radiation from the TSR-II which penetrated the Pratt and Whitney reactor shield mockup and the crew-compartment mockup. The fast-neutron and gamma-ray doses within the crew-compartment mockup had previously been calculated by ANP shielding groups at Pratt and Whitney, Convair, and ORNL. The TSR-II power calibration will require additional checking before the experimental data can be compared with the results of these calculations.

Preliminary measurements were made in water at the Bulk Shielding Facility of neutron spectra from a fission plate. The Li^6I scintillation counter was used. In the program for calculating the energy and angular distribution of neutrons leaking from and within shields, additional calculations on leakage within lithium hydride were carried out by

NDA. Lithium hydride bodies were shipped to General Atomic for experimental studies using neutron pulses from targets placed in the General Atomic electron linear accelerator.

Columbium Alloy Research. – Examination of the second group of Cb–1% Zr alloy in-pile tube-burst specimens showed that their surfaces were bright and similar to those of unirradiated material. The ruptures were of the blowout type previously observed in the out-of-pile tests. Preparations are being made for complete metallographic examinations of the specimens. Apparatus is being constructed for an experiment at a lower applied stress that will permit a longer period of irradiation prior to failure.

Liquid-Metal Technology. – The Inconel loop for studying boiling-potassium corrosion is being operated at a boiler temperature of 1600°F. The heat removed in the cooler corresponds to a potassium vapor flow rate of 200 g/min and a vapor velocity of approximately 50 fps in the 1/2-in. sched-40 pipe of the vapor section. These figures are based on calculations in which it is assumed that only saturated vapor reaches the condenser and that the potassium vapor behaves ideally. It is planned to continue operations at the current conditions for 3000 hr.

Preliminary heat transfer data were obtained for liquid potassium in laminar flow (Peclet modulus between 20 and 40) at an average fluid temperature of 950°F, using the 1-in.-diam temporary test section of the boiling-potassium heat transfer apparatus. The results were in reasonable agreement with data reported in the literature for both mercury and the lead-bismuth eutectic. Following the laminar-flow experiment (during which instrument calibration was accomplished), the noncondensable cover gas (argon) was removed by evacuation, and potassium boiling at ~50% vapor quality was instituted. Pertinent results are as follows:

Run No.	Boiler Exit Temperature (°F)	Boiler Heat Flux (Btu hr^{-1} ft^{-2})	Heat Balance (%)	Vapor Quality (%)	Boiler Exit Velocity (fps)
1	1376	35,100	77	53	107
2	1377	40,400	88	53	122
3	1399	43,000	93	57	130
4	1383	41,900	92	49	127

The inlet liquid flow rate ranged from 77 to 98 lb/hr (0.11 to 0.14 fps). Liquid temperatures at the inlet were held at 1130 to 1160°F as a safety precaution in these initial studies; the saturation temperature was controlled by varying the exposure of the condenser coils. Large-scale fluctuations in the boiler tube-wall temperatures, the boiler inlet and exit pressures, and the boiler inlet flow rate were observed. These excursions occurred at an average frequency of 3 cycles/min; typically, the boiler wall temperature rose slowly (over about 20 sec) to a value about 130°F above the saturation temperature and then fell very rapidly to the initial temperature. Concurrently, the boiler inlet pressure climbed from 12 to

27 psia, and the liquid flow dropped from 0.25 to 0.20 gpm. This behavior suggests that the boiling is in the slug-flow regime. The pressure, temperature, and flow recorders are being modified so that a more detailed examination of these oscillations can be made. A 30% reduction in the flow (to 63 lb/hr) at a flux of 43,000 Btu hr^{-1} ft^{-2} resulted in a boiler-tube temperature rise to 1900°F in approximately 70 sec; safety circuits turned off the boiler power at this point.

Beryllium Oxide Research. – Additional small-scale preparations of beryllium hydroxide of unusually high purity have been made by the acetylacetonate solvent-extraction process previously described. A 30-g batch made in the most recent test was examined spectrographically for impurities. All impurity elements were reported to be below the present limits of detection. Specifically, the report indicated the following:

< 20 ppm Ti

< 10 ppm Al, Ba, Ca, Cr, Fe, K, Na, Ni, Si, Sr, V

< 5 ppm Co, Cu, Li, Mg, Mn, Mo, Pb, Sn

This material was made available to the analytical chemists for use in the preparation of improved analytical standards which will be useful in characterizing future products. Larger quantities of beryllium hydroxide were then prepared by this process for conversion to the oxide by ignition. Laboratory studies of the distribution of boron between the aqueous and organic phases in the extraction process gave results which showed that if boron were present initially it should be removed in the process. Spectrographic analyses have shown less than 1 ppm boron in the beryllium hydroxide products.

Measurements made with the use of the porous-collector method led to the conclusions that the BeO-CeO_2 system is a simple eutectic, that the eutectic temperature is 1890 ± 20°C, and that the eutectic mixture contains 63 ± 3 mole % BeO.

Postirradiation analysis of the specimens from the beryllium oxide irradiation experiments was continued. Samples with various irradiation dosages at selected temperatures are being examined metallographically and by petrographic microscopy and are being analyzed for gas content. A corresponding group of samples is being studied by x-ray diffraction analysis and by electron microscopy at Battelle Memorial Institute and at the Oak Ridge National Laboratory. Preliminary reports on the x-ray and petrographic analyses indicate a significant increase in the c lattice parameter and gross lattice defects in the hexagonal BeO crystal structure. The evidence for these defects is being studied to construct a model representing an altered crystal structure.

Power Plant Preliminary Design Studies. – Estimates were made of the radiator weights for a gas-cooled reactor in a gas-turbine cycle for a space power plant operating with the best conceivable presently obtainable conditions. These estimates indicated that on the basis of specific weight the gas-turbine cycle cannot compete favorably with a reactor using the Rankine cycle. A Rankine-cycle reactor operating at a peak temperature of 1600°F would have a radiator with a specific weight of 3 to 4

lb per electrical kilowatt for a 1-Mw (electrical) power plant. Pushing the gas-turbine cycle to an operating point which appears to be the best possible in the foreseeable future, such as 3000°F with a system pressure of 750 psi, the estimates indicate that the radiator weight would still be 8 lb/kw for a 1-Mw power plant.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Reactor Evaluation Studies: Heat Transfer from Spent Fuels During Shipping. – An experimental device was designed and assembled for studying the temperature rise in spent reactor fuels during shipping. Stainless steel tubes containing Nichrome resistance wire for internal heating were assembled into bundles to simulate fuel elements and the bundles placed in a 12-in.-ID stainless steel cylinder to simulate a shipping cask. Different layers of insulation around the cylinder simulate various shield thicknesses. The thermal emissivity of unpolished type 304 stainless steel was found to be ~0.55 in a very simple experiment in which two concentric tubes of different diameters were separated by a near vacuum.

Release of Fission Products on In-Pile Melting of Reactor Fuels. – The experimental unit of the first meltdown experiment in the In-Pile Fuel Element Catastrophe Program is 100% assembled and tested. During the last shutdown the effect of the facility tube on the reactivity of the ORR was measured and found to be 0.035%. The experiment position indicator was installed, completing assembly of the experiment facilities in the reactor pool. Installation at poolside of the automatic liquid-nitrogen filling system is now in progress.

Basic Reactor Research: Pulsed-Neutron Measurements. – During the last few years the pulsed-neutron-source method has been applied to measure various neutron-diffusion parameters at the Bulk Shielding Facility. A group of such measurements involved the determination of the neutron-diffusion coefficient and the diffusion cooling constant for thermal neutrons in beryllium. A comparison of the results with data from similar experiments at another laboratory showed reasonable agreement in the diffusion coefficient, but disagreement in the diffusion cooling constant. In an effort to determine the cause of this lack of agreement, additional experiments have been performed with samples of beryllium obtained from the other laboratory. The results of these experiments are in agreement with previous results at the BSF, thus indicating that differences in the beryllium samples are not responsible for the disagreement. Other possible causes are being investigated.

Basic Reactor Research: Calculation of Scattering Cross Sections. – The accurate description of the processes of elastic and inelastic scattering within the nucleus is of fundamental importance to both reactor and shield design. A program intended to extend the detailed knowledge of the cross sections for these processes is in progress at the Bulk Shielding Facility. Both theoretical and experimental approaches are being made, so coordinated as to provide mutual reinforcement of the results obtained. The theoretical attack, presently somewhat more advanced, is for the

purpose of calculating the elastic and inelastic cross sections from first principles. The basic model being employed is an optical one, in which the nuclear interaction is replaced by a complex nonlocal potential well for the case of elastic scattering.

The conventional scheme for the computation of inelastic scattering involves two steps. The elastic wave functions are first calculated according to the usual local optical model, then utilized for the calculation of the inelastic scattering cross section. A program utilizing this conventional scheme is already in operation at the Laboratory.

A somewhat more sophisticated approach to the problem is being employed in the present work. A system of coupled differential equations has been constructed, the solution of which results in the simultaneous calculation of both the elastic and nonelastic scattering cross sections. The methods are being coded for use with the IBM 7090 computer, and individual results will be obtained with a very small investment in computer time.

The experimental portion of the program is at present essentially in the "make-ready" stage.

Basic Reactor Research: Measurement of α. – An experiment intended to establish the value of α, the capture-to-fission ratio, to a statistical accuracy of ~1% to 10% over a broad range of energies is in progress at the Bulk Shielding Facility. Materials studied will be U^{235}, U^{233}, and possibly Pu^{239}. A special multiplate fission chamber, embodying a special preamplifier and amplifier, has been designed and is nearing completion. For measurements in the region of 30 kev, this special chamber will be centered in a large aluminum tank of liquid scintillator. The electronic system, which will enable proper identification and counting of the various events occurring in the chamber and scintillator, has been developed. The neutron source is expected to be the 3-Mev accelerator in Building 5500, and the general experimental arrangement will resemble that used in previous measurements of capture cross sections.

Power Reactor Fuel Processing: Solvent Extraction Studies. – About 99.9% of the uranium was extracted with 5% tributyl phosphate (TBP) in Amsco from PWR fuel solutions prepared by the Modified Zirflex process; zirconium extraction was less than 0.001 g/liter, and there was no difficulty with emulsion or precipitation.

In preliminary experiments on extraction of Nb^{95} from 4 M HNO_3 with 30% TBP in dodecane containing 10% chemically degraded TBP-Amsco, only 80% of the niobium present was in an extractable form. The distribution coefficient of this extractable form was 0.90. The distribution coefficients of Nb^{95} between the organic phase and fresh 4 M HNO_3 ranged from 0.4 to 2.0.

Power Reactor Fuel Processing: Mechanical Processing. – In further batch leaching tests in glass equipment, 4 M HNO_3 at an HNO_3/UO_2 mole ratio of 4.06 was insufficient to completely dissolve the UO_2 in 4 hr, whereas 5.5 M HNO_3 at a mole ratio of 5 was barely adequate to dissolve it in 4 hr. When the dissolution temperature was decreased from 102 to 92°C the maximum uranium loading was decreased from 350 to 300 g/liter and the dissolution time increased from 1.5 to 2.5 hr.

Crushing of UO_2 pellets increased slightly the spread between the maximum and the minimum uranium loading in the product of leaching with 4 M HNO_3: at apparent steady state, the maximum and the minimum loading were 400 and 150 g of uranium per liter for crushed pellets, as compared with 375 and 225 g/liter for whole pellets. The dissolution time for crushed UO_2 pellets was 25% less than for whole pellets.

Power Reactor Fuel Processing: Modified Zirflex Process. – Engineering-scale tests were made with unirradiated TRIGA (8% U–ZrH) and PWR-1C (2.5% U–Zr) fuel samples to determine the effect of omitting the NO_3^- in the standard Modified Zirflex dissolvent, 6 M NH_4F–1 M NH_4NO_3–H_2O_2. Foaming, though not serious, was more pronounced than with NO_3^- present; dissolution rates were the same with or without NO_3^- in the dissolvent. Other TRIGA fuel dissolution tests with standard dissolvent were conducted in a 1-in.-diam recirculating dissolver. The minimum F/Zr ratio for the total dissolution was 7.5; the final ratio of free fluoride to uranium was 45. An insoluble surface layer formed on the fuel when the F/Zr ratio was < 7.5.

Corrosion rates of types 304L and 309SCb stainless steel in the 0.68 M zirconium solution resulting from dissolution of zirconium in 5.4 M NH_4F–0.33 M NH_4NO_3–0.003 M H_2O_2 increased from 14.3 to 24.0 and from 3.7 to 3.9 mils/month, respectively, when exposure times were increased from 24 to 48 hr. These rates are about double those obtained in the dissolver solution before dissolution.

Power Reactor Fuel Processing: Graphite-Containing and Uranium Carbide Fuels. – A sample of graphite containing 125-μ UO_2 particles coated with a 17-μ-thick layer of Al_2O_3 was subjected to the standard flowsheet treatment (two leachings with 90% HNO_3). Essentially no uranium dissolved, 99.18% of it remaining in the graphite residue. Combustion of a sample of this material in oxygen at 700 to 900°C and digestion of the ash in boiling 10 M HNO_3 for 8 hr recovered only 9.93% of the uranium.

PBR samples (Si-SiC-coated UO_2) were burned under similar conditions. The uranium loss to the HNO_3-insoluble residue was about 0.35% when the coating was 3 mils thick and about 2% when the coating was 30 mils thick. The uranium-silicon weight ratios in the respective samples were 4.5 and 0.33.

In preliminary tests to determine the explosive hazard associated with the use of 90% HNO_3 in processing graphited fuel, a mixture of disintegrated graphite and fresh 90% HNO_3 did not detonate when primed by a No. 6 cap and a 5-g pentaerythritol tetranitrate (PETN) booster.

Ion exchange resins separated uranyl ion from an unidentified dark-red species produced in the reaction of uranium monocarbide with nitric acid. Uranyl ion was sorbed on Dowex 50 cation resin; the dark-red species was not sorbed on Dowex 50 but was sorbed on Dowex 1 anion resin, leaving a yellow uranyl solution. The uranium on the Dowex 50 was satisfactorily eluted with 6 M HCl and the dark-red species on Dowex 1 with 12 M HCl.

Power Reactor Fuel Processing: Beryllium-Containing Fuel. – Dissolution rates of high-fired BeO were measured in 30% fluosilicic acid,

in 30% hypophosphorous acid, in 2.5 M trichloroacetic acid, and in 7.4 M phosphoric acid with and without added 0.25 M NaF. The rate of solution was 0.70 mg cm^{-2} min^{-1} in fluosilicic acid and considerably less in the other solutions.

In dissolution of GCRE fuel pellets (70% UO_2–30% BeO), the following solutions were no more effective than 8 M HNO_3: (1) boiling 2 M HNO_3 containing 2 to 6 M HCl and (2) boiling 8 M HNO_3 solutions containing 0 to 0.2 M NaF.

Power Reactor Fuel Processing: Corrosion Studies. – Unwelded INOR-8 was corroded at rates of 0.26 and 0.17 mil/month in a mixture of 90 vol % 21 N HNO_3 with 10 vol % 40 N H_2SO_4 at 50°C in 168 and 336 hr respectively. No local attack was observed. The corrosion rate of LCNA in this solution was decreased from 0.9 to 0.3 mil/month by passing dry air above the solution.

LCNA was corroded at a maximum rate of 0.08 mil/month in 288 hr at 50°C in a solvent extraction feed solution of the composition 1 M H^+–4.2 M NO_3^-–1 M Al^{3+}–0.4 M Zr^{4+}–2.7 M F^-. No local attack was observed.

Treatment and Disposal of Radioactive Wastes: High-Activity Wastes. – Ruthenium volatility during batch evaporation-calcination of TBP-25 process waste was decreased to 0.03% by the addition of 1.52 M phosphorous acid to the initial waste solution. With simulated Darex process waste, 0.02, 0.18, and 1.47% of the original ruthenium was in the condensate when the waste contained 1.80, 0.90, and 0.45 moles of H_3PO_3 per liter. A semicontinuous evaporation-fixation experiment was run in stainless steel equipment with ~20 liters of Purex process waste to which 1.5 M H_3PO_3, 0.8 M $Ca(OH)_2$, 1.3 M NaOH, and 0.34 M borax were added to suppress ruthenium and SO_3 volatility and to produce a melt that would solidify to a product with low void volume. The maximum ruthenium volatilized was 2% (background for stable ruthenium), and the solid produced had a density of 2.6 g/cc, representing a solid waste volume of ~6.1 gal per ton of uranium processed.

The oxides, including SO_3, from Purex waste (40 gal per ton of uranium) were incorporated into magnesium borophosphate glasses with softening points of about 850°C. These solids had densities of about 2.7 g/cc and contained 38 to 54% waste oxides, representing 4.8 to 6.9 gal of glass per ton of uranium processed.

The control and operability of a 9-liter/min evaporator close-coupled to the 8-in.-diam by 84-in.-long pot calciner were demonstrated with simulated Purex waste as feed. The condensate from the calciner was returned to the evaporator for acid removal by steam-stripping and de-entrainment of carry-over from the calciner. The acid was steam-stripped by adding water at a controlled rate to maintain the evaporator temperature at a predetermined point while keeping the acid concentration below 6 M.

The average feed rate to the calciner for the test was 20.5 liters/hr. The bulk density of the resulting calcined solid was 1.3 g/cc. The volume of noncondensable off-gas was 0.18 ft^3 per liter of calciner feed. All the nitrate, within analytical accuracy, was recovered in the evaporator condensate.

Treatment and Disposal of Radioactive Wastes: Low-Activity Wastes. – Preliminary drawings have been issued for the design of a 10-gpm pilot plant to demonstrate removal of fission product cesium and strontium from ORNL low-activity waste by ion exchange following a precipitation-flocculation step.

In studies of clinoptilolite, a natural zeolite mineral, as an adsorbent for decontaminating ORNL low-activity waste, cesium broke through (1% level) a clinoptilolite bed at 6900 bed volumes of waste. The distribution coefficient determined from the 50% breakthrough point was 8700.

Treatment and Disposal of Radioactive Wastes: Engineering and Hazards Evaluation. – The costs for interim liquid storage of high-activity wastes with 4% interest and the "present worth" method of financing future capital outlays ranged from about 0.002 mil/kwhr (electrical) for 0.5-yr storage to 0.005 to 0.01 mil/kwhr for 30-yr storage. Details of this study are contained in ORNL-3128, which is now in preparation.

The second phase of the study, pot calcination, is being conducted. In the pot calcination plant (for the purposes of the study, to take waste from a separations plant that would process a combined load of 1500 tons of uranium and 270 tons of thorium fuel per year), the number of pots filled per year and the number of pot lines required range from 925 6-in.-diam pots per year and three pot lines for acid Purex waste to 1540 12-in.-diam pots per year and seven pot lines for neutralized Thorex waste. From British data for fixation of aluminum waste in glass by pot calcination, 1100 pots per year would be filled and ten pot lines would be required for acid Thorex glass. The total plant floor area for various combinations of wastes and pot sizes ranged from 1500 to 3500 ft^2.

Waste Disposal Research and Engineering: Geochemical Studies. – By treating biotite and hydrobiotite with sodium tetraphenylboron, the cation exchange capacity was increased from 1.5 to 95 meq per 100 g and from 65 to 140 meq per 100 g respectively. X-ray diffraction patterns revealed that the treatment caused an increase in the c-axis dimension of the minerals. This observation is consistent with the view that the potassium ion was removed by the tetraphenylboron and the more hydrated sodium ion substituted in the vacant position. Analysis showed that the reduction in potassium in the minerals was equal to the exchange capacity generated. Treatment of muscovite in a similar manner produced very little change; this observation suggests that only trioctahedral micas will be affected by the sodium tetraphenylboron treatment. As expected, tests with treated and natural trioctahedral minerals showed that the former have much higher saturation capacities for cesium sorption.

Waste Disposal Research and Engineering: Disposal in Natural Salt Formations. – The Laboratory has entered into a contract with the Carey Salt Company for use of their inactive mine at Lyons, Kansas, as a location for waste disposal experiments using actual radioactive waste materials. In preparation for field experiments, an investigation of the

salt structure both above and below the existing mine opening is in progress. Two core holes drilled 40 ft below the floor of the mine show salt to a depth of more than 35 ft. One core hole in the ceiling and one additional hole in the floor remain to be drilled. The data from the completed drilling indicate that experiments at the existing mine level (1020 ft) will be surrounded by a minimum of 35 ft of salt. The total thickness of the salt bed above the mine is more than 300 ft.

The erosion of salt cavities by waste solution was found to be a physical process brought about by changes in the salt concentration at the liquid-gas interface. Changes in concentration result from evaporation and condensation of water. The rate of erosion is directly related to solution temperature.

Waste Disposal Research and Engineering: Process Waste Water and Treatment Plant Studies. – The physical characteristics of vermiculite are likely to change as a result of bed compaction or chemical treatment. A 2-in.-diam × 12-ft-long glass column was charged with 9 ft of vermiculite (BO-4 grade), and the loss of head was determined with fluid typical of process waste. The resistance to flow during upflow operation agreed with results obtained with a 1-1/2-ft column of vermiculite; channeling began at a flow rate of 2.5 ml min^{-1} cm^{-2}. However, during downflow operation the loss of head (37.5 cm of water per foot of vermiculite at 4 ml min^{-1} cm^{-2}) was twice as large as that obtained with shorter columns. After the vermiculite was treated with NaCl (80 liters of 12% solution) and NaOH (35 liters of 0.2 M solution) the loss of head increased to 120 cm of water per foot of vermiculite at 2 ml min^{-1} cm^{-2}, primarily because of breakdown of the particles by the caustic treatment. Although the head loss was reduced by removing the excess fine material with backwashing, full recovery did not occur; the head loss remained 1.6 times as large as before treatment. Since mechanically cleaned vermiculite does not require a caustic treatment, its use should obviate this difficulty.

Waste Disposal Research and Engineering: Clinch River Studies. – Radiochemical analyses of the core samples of bottom sediments collected between Clinch River miles 4.7 and 22.5 were completed. The predominant radionuclides identified were Cs^{137}, Ru^{106}, Co^{60}, and Sr^{90}. Calculations show that Cs^{137} and Sr^{90} make up 74 and 1.5%, respectively, of the identified radionuclides. The distribution of radionuclides along each cross section will be used to determine the total quantity of activity in the sediments of the river. Details of the investigation have been reported to the Clinch River Steering Committee.

PHYSICAL RESEARCH PROGRAM

PHYSICS AND MATHEMATICS RESEARCH

High-Voltage Experimental Program: The Reaction $O^{18}(\alpha,n)Ne^{21}$. – The total, absolute (α,n) reaction cross section of O^{18} has been measured for E_α = 2.4 to 5 Mev, using the large 1.5-m-diam graphite sphere [J. Nuclear Instr. 1, 335 (1957)] as the neutron detector. Targets consisted of tantalum with a surface layer anodized in high-purity H_2O^{18}, with thicknesses less than 2 kev (for 2.5-Mev alpha particles) for some measurements. Absolute cross sections were obtained by using O^{18} gas. The efficiency of the graphite sphere was determined with the aid of a radium-beryllium neutron source calibrated by the Bureau of Standards. Approximately 25 resonances, ranging in width from 5 to 150 kev, have been found. A preliminary value of the absolute cross section at the peak of the resonance at 3.19 Mev (Γ = 80 kev) is 109 ± 25 millibarns. As a check on the energy calibration the $B^{10}(\alpha,n)N^{13}$ resonance near 1.5 Mev was found at 1.507 ± 0.004 Mev. This value is slightly lower than the previous value [Phys. Rev. 105, 957 (1957)] obtained from the $B^{10}(\alpha,p\gamma)C^{13}$ reaction.

Heavy-Particle Physics. – The angular distribution of the products of the reaction $B^{10}(N^{14},C^{13})C^{11}$ was measured, and an interaction radius of ~11 fermis was deduced. Excited-state and ground-state transfer cross sections were measured as a function of energy for the reaction $B^{10}(N^{14},N^{13})B^{11}$; the results show that excited-state transfer seems to be favored at lower incident energies, and ground-state transfer is more probable at higher energies. The proton and alpha-particle spectra from the nitrogen bombardment of C^{12} were measured from 0 to 150°, and the results are being analyzed.

The 86-Inch Cyclotron: Nuclear Physics. – The investigation of nuclear structure with (p,d) reactions is continuing. The angular distributions of the prominent peaks in the deuteron spectrum from an Ni^{64} target show that those levels in Ni^{63} below 3 Mev excitation which are strongly excited by a (p,d) reaction have spins of 1/2 or 3/2. The lowest level that shows an ℓ = 3 angular distribution occurs at an excitation of approximately 3.4 Mev. The spin of that level must be 5/2 or 7/2. Comparison of the spectrum with (p,d) spectra from iron isotopes suggests that the 3.4-Mev level is reached by removing an $f_{7/2}$ neutron from the ground state of Ni^{64}. The absence of lower-lying ℓ = 3 peaks in the deuteron spectrum is surprising, since the spherical-potential shell model requires that at least two neutrons be in the $f_{5/2}$ shell in Ni^{64}. This result may be interpreted as an indication that Ni^{64} has a prolate deformation, which would displace the states so that the 5/2 spin state would be empty for a nucleus containing 36 neutrons (Ni^{64}).

Since the level structure of Ni^{63} is not known from any other work, these results constitute new information. The ground-state spin of Ni^{63}, known from beta decay to be either 3/2 or 5/2, and from this work to be either 1/2 or 3/2, must be 3/2. The energies and spins as determined are shown below:

Energy (Mev)	Spin
0	3/2
0.9	1/2 or 3/2
2.0	1/2 or 3/2
3.4	5/2 or 7/2

Electronuclear Machines. – In an investigation of the applicability of superconducting solenoids to electronuclear devices, preliminary tests of superconductors were made in the 200-kilogauss magnet at Vanderbilt University. Suitable techniques were developed for measuring the properties of superconductors in the pulsed magnetic field. Some results of this investigation are given under "Alloy Theory Research," this report.

The Isochronous Cyclotron. – Installation of the main magnet was completed, and the pole bases were aligned to exact specifications. The pole tips are now in the shop and are being precisely positioned on the pole disk before being placed in the magnet; this shop work is about 50% completed. The valley and harmonic coils were formed and are being vacuum-canned before being fitted to the pole tips. The regulators for the main coils and the valley coils were installed and checked out; the magnetic field was excited in preliminary tests. The AVF power supplies, the console, the control panel, and the relay cabinets were put in place and are being interconnected. Equipment was installed for the cyclotron machine shop in Building 6000.

The 86-Inch Cyclotron: Applied Physics. – The demand for high-specific-activity Co^{57} for use in Mössbauer studies has continued. Nickel salts purified by ion exchange are used in preparing nickel platings of extremely low cobalt content as targets for proton irradiation. Specific activities of 4.2 curies per mg of cobalt are achieved. This represents an almost 1:1 ratio of active to inactive cobalt. Over 2 curies was produced during this period. The following radioisotopes were produced: Be^{7}, Na^{22}, Ti^{44}, Mn^{52}, Co^{57}, Ge^{68}, As^{74}, Sr^{85}, Y^{87}, Y^{88}, $Y^{87,88}$, Cd^{107}, I^{124}, I^{125}, Cd^{149}, and Bi^{207}. A stack of molybdenum foils was also exposed to the external proton beam as a service irradiation. These irradiations totaled 240 hr of beam time, ranging from a 15-min irradiation to a 48.5-hr irradiation, and represent ~47% of the beam time normally available to this program.

Stable Isotope Development. – Further studies and experimentation associated with target preparation have continued on the decelerating calutron receiver. A vertical grounded plate with a defining slit was placed at the focal point of the incoming beam. Behind this plate an electric field was maintained to slow the positive ions. The effect of this electric field combined with the magnetic field causes the ions to travel in a cycloidal trajectory. A collecting plate was placed at the point of lowest energy, intercepting the beam. The fringing of the electric field through the opening in the grounded defining plate caused some radial defocusing; however, decelerating by full source potential caused only approximately a 10% loss in beam current. Targets of Sn^{120}, Ca^{40}, Mg^{24},

Eu^{151}, and B^{10} have been prepared with varying amounts of success. Generally the results are encouraging, and in the future this procedure may provide a good source for isotopic targets for nuclear studies.

Preparations necessary to construct and operate the new inhomogeneous-field isotope separator are being made. The components of a magnetic field measuring system have been purchased and plans made for their assembly. A large wheel that will permit accurate radial and azimuthal positioning of the magnetic field probe is being built. Construction of a modified source and receiver, necessary because of the 255° focal angle, is also in progress.

In order to begin with the best possible theoretical shape for the pole pieces, a numerical calculation of the field produced by various trial pole surfaces is being made. The field produced by the first trial surface is compared with the desired field shape. Corrections are then applied to this surface so that it will produce the desired field. Since the pole surfaces are limited by the tank volume of the calutron, this type of calculation will allow correction for disturbances produced by physical restrictions imposed on the pole pieces. Effects due to composition irregularities, saturation effects, skewness of the main field, and the unsymmetrical influence of the calutron tank will be measured experimentally. Final corrections to the field of the new separator will be made on the basis of these measurements.

Special Separations. – Stripping and decontamination operations in preparation for the new plutonium chemical processing area were completed by the H. K. Ferguson Company on March 27, 1961. The area was given a thorough cleaning following the stripping operations.

Approximately 9.7 kg of purified UO_3 was converted to UF_4 and reduced to metal. The metal was cast into a billet weighing 6654 g and transferred to Y-12 to be rolled into foil. This foil will be issued for civilian application.

A special request involving the preparation of 450 alpha sources for the Army Signal Corps was completed. Uranium-233 was adsorbed on Decalso and fused onto 3 × 7 in. stainless steel plates. The fused film appeared uniform and adherent, with a film thickness of about 1 μ. These sources were prepared at specified activity levels ranging from 2.0×10^5 to 1.9×10^6 counts/min.

The conversion of 80 g of $U_3^{235}O_8$ to UF_4 was completed. This material will be used to prepare U^{235} targets.

Approximately 35 g of Np^{237} has been purified to be used in the fabrication of 300 detector foils for a California company.

CHEMISTRY RESEARCH

Radiation Chemistry of Organic Materials: High Polymers. – It was found that the mixed radiation of the ORNL Graphite Reactor is 3.3 times as effective as pure gamma radiation alone in producing intermolecular cross links in polystyrene. The radiation intensity of the reactor (Hole 19) was measured with graphite and nylon calorimeters to determine the amount of energy deposited in hydrocarbon materials by the gamma and fast-neutron components respectively. A Co^{60} source, monitored with $FeSO_4$ solutions, was used for the gamma irradiations.

The number of cross links produced in soluble polystyrene samples, which had received low doses, was determined by measurements of average molecular weight. The cross links produced in samples irradiated for longer periods were determined by the fraction of sample remaining soluble after irradiation and by the degree of swelling of the insoluble portion when immersed in a solvent.

The polystyrene, which was from two different sources, was analyzed by spectrographic and radioactivation methods. These analyses showed that there were no trace impurities which had high neutron absorption cross sections. Therefore, the neutron field of the reactor could not have produced nuclear reactions which would have increased the dose in the polystyrene over that indicated by the calorimeters.

The measurements show that fast neutrons having the energy spectrum of the Graphite Reactor produce one cross link for every 300 ± 25 ev deposited in the sample, while gamma radiation yields one cross link for every 1300 ± 50 ev deposited.

Thorium Oxide Studies. – A slurry of Th–0.4% U^{235} oxide in D_2O irradiated for five weeks in the LITR at 280°C showed considerable particle damage. Twelve per cent of the recovered solids was in a dispersed form with an average size of 2500 A. Material that had settled to the bottom of the autoclave had an average particle size of 1.6 μ, but a very steep size distribution curve, indicating that nearly all the oxide had suffered particle damage with some reagglomeration occurring. The original preparation had been carefully classified to remove fines and contained only 0.5 wt % of particles less than 1 μ. At least 58% of the recovered solids (the dispersed plus the settled fraction) was less than 1 μ, as measured by sedimentation particle size analysis.

Gas recombination (280°C) tests with a slurry of Th–13% U oxide and a palladium catalyst, at constant initial hydrogen partial pressure (100 psi) in which the initial oxygen partial pressure was varied from 800 to 200 psi, showed initial reaction rates consistent with a 0.5 order of dependence of the recombination rate on the oxygen partial pressure. The apparent reaction orders for the individual experiments based on electrolytic gas (i.e., $H_2 + 1/2\ O_2$) pressure changed from first order to orders < 1 as the excess oxygen partial pressure was decreased. Previous work indicated a first-power dependence of the recombination rate on hydrogen partial pressure. The 0.5-power dependence on oxygen and the decrease to apparent orders < 1 as the oxygen partial pressure was decreased suggest that a kinetic expression for the recombination must take into account the adsorption of at least one of the gases (O_2) and possibly both at the low partial pressures.

Equipment Decontamination. – In oxalate-peroxide decontamination mixtures, stability was improved at 95°C by decreasing the peroxide concentration to 0.3 M, lowering the pH to 4.0, and adding 0.1 M ammonium citrate. In the presence of 100 ppm Fe^{3+} and with iron surfaces, both of which shorten the life of peroxide mixtures, the useful life without periodic adjustment of pH was extended to 6 hr. Several reagents normally used in industry for peroxide stabilization had little effect. Copper alloys must be excluded from all contact with the solutions. The oxalate solution without peroxide was too corrosive for use on carbon steels, but peroxide acted as a corrosion inhibitor for both carbon and stainless

steels. For example, at 95°C and pH 4 in the presence and absence of 0.3 M H_2O_2, corrosion rates of high-carbon ball bearings were 0.010 vs 1.6 mils/hr and of type 440C stainless steel ball bearings were 0.001 vs 0.32 mil/hr respectively. Several commercial corrosion inhibitors were without effect in peroxide solutions. There is a likely application of peroxide for corrosion inhibition in the second step of the widely used alkaline permanganate-ammonium citrate process for defilming and decontaminating aqueous heterogeneous reactors. Spray decontamination was simulated in a modified household dishwasher with very good results.

Analytical Chemistry Research. – An apparatus has been assembled for continuously monitoring the oxygen content of gas streams. The sensing element of this apparatus is a Hersch cell which consists of a cadmium anode submerged in 30 w/v per cent KOH and a silver cathode which is partially immersed in the electrolyte. Associated equipment is housed in a standard rack cabinet.

The gas stream is analyzed continuously for oxygen by flowing it through the cell above the electrolyte and measuring the cell current. Oxygen in the gas is reduced at the silver electrode, thus causing a current to flow which is proportional to the concentration of oxygen. It is estimated that concentrations of the order of 1 ppm can be measured by this means.

Although the cell responds very well to small changes in concentration of oxygen in the gas stream, it has been found to be very sensitive to changes in temperature. Efforts are being made to eliminate the thermal effect by controlling the temperature of the cell housing. The apparatus will be calibrated by the measurement of coulometrically generated oxygen and also by the analysis of gas streams to which known amounts of oxygen have been added.

Inorganic Solution Chemistry. – Equilibrium ultracentrifugation of hydrolyzed uranium(VI) chloride solutions indicates a weight average degree of polymerization of less than three, up to an average hydroxyl number of 1.4, the highest measured. A few ultracentrifugations in perchlorate and nitrate media give a similar picture. These results conflict with hydrolytic schemes in the literature based on acidity measurements; reinterpretation of such pH data indicates, however, that agreement within the probable experimental error can be obtained by postulating two hydrolyzed species, $(UO_2)_2(OH)_2^{2+}$ and $(UO_2)_3(OH)_5^{+}$, which are also consistent with the ultracentrifugal results. The species UO_2OH^+ or $(UO_2)_2OH^{3+}$ may also be present, but do not seem to be required by the data examined. These conclusions agree in general with those reported by others at the Laboratory on the basis of pH measurements in nitrate systems (ORNL-3120).

Organic Chemistry. – Isotopic tracer studies with C^{14} demonstrated that in the acid- and base-catalyzed rearrangements in aqueous solution of alloxan, monomethylalloxan, dimethylalloxan, and monophenylalloxan the nitrogen shift takes place to the exclusion of a carbon shift. The migration ratio of the N-methyl fragment with respect to the N-hydrogen fragment in the rearrangement of monomethylalloxan is about 3:1. Migration of the N-phenyl fragment takes place to the exclusion of the N-hydrogen fragment during the rearrangements of monophenylalloxan.

A continuation of studies on small, secondary C^{14} isotope effects with the differential method first used at this Laboratory led to the following results:

1. In the hydrolysis of p-benzoylbenzoic ester, labeled with C^{14} in the carbonyl group between the two benzene rings, $k^*/k = 1.0025 \pm 0.0006$.

2. In the formation of the 2,4-dinitrophenylhydrazone of phenyl t-butyl-1-C^{14} ketone, $k^*/k = 1.0029 \pm 0.0007$.

3. In the formation of the 2,4-dinitrophenylhydrazone of phenyl-1-C^{14} methyl ketone, $k^*/k = 1.0035 \pm 0.0003$.

The indicated uncertainties are standard deviations given by a non-linear least-squares treatment, carried out on the IBM 7090.

Radiation Chemistry. – Binary mixtures of C_2H_2 with the noble gases were studied in a research mass spectrometer at high pressures (> 0.1 mm in the ionization chamber). The results indicate that mixtures of C_2H_2 and xenon under electron bombardment produce two complex ions, $[XeC_2H]^+$ and $[XeC_2H_2]^+$. On the basis of pressure dependence measurements, it is assumed that both these complex ions result from the excited complex ion $[XeC_2H_2^*]^+$, the former (which is the more abundant one) by dissociation and the latter by collision with a neutral molecule.

High-Temperature and Structural Chemistry. – Solutions of lanthanum in molten lanthanum trichloride were found to exhibit an electronic conductance very similar in extent and concentration dependence to that observed for solutions of cerium in cerium trichloride. Saturation at 906°C was reached at 10.3 mole % lanthanum metal, in agreement with a very recent revised solubility value reported by others.

The electrical resistivity, ρ, of solutions of potassium fluoride in potassium metal increases linearly with the mole fraction, N, of the salt according to the equation $\rho = 420N + 58$ microhm-cm. Thus the increase with mole fraction is less rapid than for potassium iodide solutions. The difference is believed to be due to the smaller cross section of F^- compared with I^- for scattering electrons, modified by the difference in polarizability between the two anions, which counteracts the effect of their size.

Chemical Separation of Isotopes. – The solubility of BF_3 in diethyl sulfide as a function of temperature and pressure was found to be as follows:

Temperature (°C)	Moles of BF_3 per Mole of $(C_2H_5)_2S$	
	400 mm Hg	760 mm Hg
−25	1.116	1.204
0	1.032	1.074
30	0.895	0.958

Transuranium Elements. – A radial gang disconnect design using spring loading appears to be good for transuranium facility process disconnects. Test of a prototype with a helium leak detector showed no leaks at 200 psig of helium in 50 thermal cycles between 40 and 160°C and in 15 makes and breaks.

METALLURGY AND MATERIALS RESEARCH

Preparation of Pure Single Crystals. – The new apparatus for growing single crystals of Li^7F has been assembled and tested. The characteristics of the new apparatus provide (1) a 7- to 8-in. region at the top long enough to maintain the entire sample above the melting point, (2) a maximum thermal gradient at the melting point (844°C) to aid impurity exclusion, (3) a region of zero axial gradient below 844°C (this region will be used for annealing purposes – 700°C for one week and 500°C for one week), (4) a vacuum of 5×10^{-4} mm at the capsule entrance to remove volatile impurities. A 500-g batch of the purest available Li^7F, from which traces of magnesium had been removed by liquid-liquid extraction, has been dehydrated with anhydrous hydrogen fluoride gas and hydrogen. The first crystal-growth study with this material is in progress.

Several melts in the systems MoO_3-BeO, MoO_3-MgO, and MoO_3-ThO_2 have been made in an effort to find a low-melting flux suitable for growing crystals of refractory oxides. Intermediate compounds were observed in each system. Tentative optical and x-ray diffraction properties were obtained for these compounds.

Alloy Theory Research. – Studies of the critical currents in superconducting niobium-clad Nb_3Sn show currents as high as 100 amp at 100 kilogauss with the field parallel to the current. The critical currents in the transverse direction are much smaller, suggesting that domain structure plays an important role in Nb-Nb_3Sn in spite of its ultrahard nature. Interesting hysteresis effects were found on passage of successive current pulses near the threshold value. It is thought that thermal spikes develop whenever superconducting "filaments" are broken and that the thermal spikes reduce the current-carrying capacity thereafter. The superconducting wires can be conditioned for maximum current-carrying capacity again by passage of numbers of pulses just below the threshold current. In this superconductor, the twisted "filaments" are apparently made more stable by the subcritical currents themselves. For 30- to 80-μsec pulses at zero external field, the critical current is 210 to 260 amp in a 0.038-cm wire at 4.2°K. These values are one-third larger than the dc value and are believed to show that heating occurs in joints in the cladding around the Nb_3Sn core during a dc test with present joint design.

Experiments with coil preparation have shown that niobium-clad Nb_3Sn coils can be successfully brazed with silver to give high current-carrying capacity. Fields of 25,000 gauss have been reached in a superconducting niobium-clad Nb_3Sn electromagnet of the iron-core type at 4.2°K.

X-Ray Diffraction and Metallographic Research. – Measurements of short-range-order diffuse scattering in the h_1h_20 plane and the h_1, h_2, $h_1 + h_2$ plane of reciprocal space for the alloy Cu–16% Al have been completed. The measurements were made at –190°C to suppress temperature diffuse scattering. Fourier inversion of these data, now in progress, will yield a description of the short-range structure of the alloy.

X-ray powder patterns have been obtained from BeO specimens irradiated to 10^{21} fast neutrons/cm^2. Analyses of these patterns have shown (1) an average expansion of the hexagonal BeO lattice ($\Delta c/c \div \Delta a/a \cong 7$) and (2) interesting symmetric and asymmetric broadening of certain reflections.

The latter effect may be ascribed to a gross damage of the BeO lattice, and a detailed model of this damaged structure is being evolved.

The high-temperature diffraction study of Cu-32 at. % Au filings has continued, with the specimen temperature lowered from 340 to 315°C during the report period. In this temperature interval, indication of a two-phase region between the disordered and the antiphased ordered structure has been obtained, and the crystallographic features of the antiphased ordered structure have been verified.

Collection of three-dimensional intensity data for the diffraction of Cu Kα x rays from a $Ca_2Be_3O_5$ crystal has begun. A suitable $LuMnO_3$ crystal has been found for observations which may distinguish between the possible space groups $P6_3cm$ and P3c1 and between the two possible orientations of the polar axis.

CONTROLLED THERMONUCLEAR RESEARCH

Arc Research. – Measurements of ion drift velocities in the deuterium arc show a mean value in good accord with the estimate based on ion current and density. The value is greater near the anode than near the cathode. It changes only slightly with arc current and magnetic field, indicating that previously observed large changes in 2- and 4-mm microwave transmission do not reflect large density changes.

Measurements of the performance of the deuterium arc as an ion pump for various gases give anomalous results which indicate that other processes are comparable in importance with simple ionization, for example, charge exchange.

DCX-1 Facility. – Several gas dissociation runs have been made with the low base pressures now available with evaporated titanium films. In these runs the proton containment times (τ) were determined as functions of pressure from base pressure to about 1×10^{-5} mm Hg with argon, helium, and hydrogen gas leaks to the inner vacuum region. The injected H_2^+ current was 1 to 1.5 ma. At a base pressure of 5×10^{-8} mm Hg, the τ values were about 0.8 sec. By way of comparison, the calculated charge-exchange time for 300-kev protons in a pure hydrogen background at this same pressure is 3.7 sec. As the pressure was increased with a bleed gas, the ratio of the measured τ value to the calculated charge-exchange value approached a constant. For argon, this constant ratio was 1/1.9; for helium, 1/1.25; and for hydrogen, 1/1.7. The time decay of the neutral particle detector signals was exponential.

In one run a lower base pressure was obtained by replacing the water cooling on the high-vacuum liner with liquid nitrogen. At an indicated ion gage pressure of 1.4×10^{-8} mm, containment times of 3.0 sec were recorded. The time decay of the neutral particle detector signal was again exponential.

Larger Magnetic Facility. – Construction of DCX-2 is proceeding nearly on schedule. Industrial and architectural-structural design and specification is essentially complete. The control-room walls are being installed. Vacuum pumping is being installed.

Mirror coils are under construction at the General Electric Company. Rebids are being obtained for the inboard booster coils. The outboard booster coils are under test.

Vacuum. – A series of 20 runs in the large getter test pump facility have consistently given notable increases in the pumping speed of evaporated titanium for hydrogen. The technique has been to evaporate titanium in an atmosphere of an inert gas. Both argon and helium have been used at pressures between 0.1 and 500 μ. Pumping speeds as high as 10 liters sec^{-1} cm^{-2} are obtained. This corresponds to a sticking factor of 0.23.

Several experiments have shown that similar results can be obtained without the use of the inert-gas atmosphere by evaporating the titanium onto a copper liner at liquid-nitrogen temperatures. One experiment has shown that a combination of the low temperature and the inert-gas atmosphere during evaporation may produce substantial improvement in sticking factors over the value recorded above.

BIOLOGY AND MEDICINE PROGRAM

BIOMEDICAL PROBLEMS IN ATOMIC ENERGY OPERATIONS

Waste Disposal Research and Engineering: Evaluation of Soil Disposal. – As a part of the study of the White Oak Creek basin, an investigation is being made of the contaminated bed of former White Oak Lake to determine the extent of deposition and loss of sediment since the lake was drained in 1955. Measurements of the depth of sediment at seven cross sections show that (1) the channel of White Oak Creek, which was filled with sediments before the lake was drained, has for the most part eroded to its preimpoundment depth, (2) the depth of sediments over the lower one-third of the bed, with the exception of the stream channel, is approximately the same as that reported in 1953, and (3) the depth of sediments over the middle and upper two-thirds of the lake is slightly less than that reported in 1953. Approximately 150,000 ft^3 of silt has left the lake bed through erosion of the channel.

Ecology and Marine Studies: Soil Arthropod Investigations. – Weekly samples of bagged leaf litter have related the numbers of soil arthropods to the loss of weight and the release of incorporated radioisotopes. Pine, oak, and dogwood leaves were followed in pine and oak forests. The percentages of the initial weights remaining after one year were: pine, 63%; oak, 60%; dogwood, 52%. Each species decayed slightly faster in the oak forest than in the pine forest. All showed an initial period of rapid weight loss (probably reflecting the loss of readily soluble materials), a lower rate during the winter months, and an accelerated rate during the moist, warm summer months and the early fall period. Losses of Sr^{85}, Ru^{106}, and Co^{60} from the leaf litter corresponded to the losses of weight, but showed more variation than weight loss because of difficulties in counting at similar geometries. The numbers of arthropods in the litter bags were higher during periods of rapid weight loss. During the initial period the high numbers represented only a few species, but during the summer months more than 50 species, both predators and litter feeders,

were present. Immature stages of arthropods were more frequent during the periods of rapid breakdown, suggesting active feeding and population growth. The numbers were higher in the more rapidly decaying dogwood leaves than in pine and oak leaves. Total numbers were higher in the oak forest, where decay was more rapid, but the numbers of oribatid mites were somewhat higher in the pine forest.

Ecology and Marine Studies: Release of Isotopes During Leaf Decomposition. – Release of isotopes during the breakdown of dead organic matter serves to measure the rate of completion of the ecological cycle of chemical elements from soil to plants and back to soil. Cesium-134 was found to be released more rapidly than Sr^{90} and Co^{60} from the leaves of redbud trees during the early stages of their natural breakdown, which was more rapid than the breakdown of pine and oak leaves. This release was measured by scintillation counting of the radioactivity of samples placed in a standardized position over a 3-in. crystal. In the first method, samples of leaves tagged with the radioactive tracers were measured in 10 × 10 cm bags of glass fiber curtain material, before the leaves were placed outdoors and at intervals later. In a second method, the isotopes were detected after they were leached from these bags, by repeated counting of slabs of natural soil material which had been placed in screen-bottomed plastic sandwich boxes underneath the tagged litter bags at the time these were placed in the field.

Both methods showed that the leaching of all the isotopes began within one week of the time the leaves were exposed (October 17) – mostly after a single rain the night of October 18. Averages of four replicates in each of the five habitats showed progressive leaching during the initial 24 weeks. At the end of this time, approximately 77% of the cesium, 37% of the strontium, and 32% of the cobalt had been released from the organic matter. As expected, the underlying soil slabs generally failed to account for all these losses because of self-absorption and leaching through the surface soil mat. Rates of isotope release from bagged redbud litter did not vary greatly between the shortleaf and Virginia pine, white oak, red maple, and tulip tree forests and a grassy field, but the underlying organic mat in the pine forest retained less Cs^{134} and Sr^{90} than did the predominantly mineral soils of the other four locations.

Ecology and Marine Studies: Clinch River Studies. – The content of stable strontium in fresh-water clams will provide a basis for interpreting the deposition of Sr^{90} in clams of the Tennessee River system. The content of stable strontium and calcium in the shells of 15 species of clams from seven locations on the Clinch and Tennessee Rivers has been determined chemically. The shells contained an average of 3.9% organic matter, as determined from the loss on ashing at 500°C for 2 hr. The calcium content of the ashed shells was constant at 40%. The average strontium content for all species was 219 ppm. The mean strontium content for different species varied from 155 to 380 ppm, and the individual variation for all shells analyzed ranged from 146 to 518 ppm. In addition to the significant differences in the strontium content among the species, there were also differences within the same species collected at different locations on the rivers. The variation in strontium content is attributed partially to the growth rate, which was determined from the age and weight of the shell. The species containing 380 ppm deposited shell at an average

rate of 7.3 g/yr, whereas the species containing 155 ppm grew at an average rate of 2.2 g/yr. The uptake of strontium in the biological deposition of the calcium carbonate shell of clams is proportional to growth rate and from this standpoint is similar to the uptake in the growth of crystals in inorganic media.

Basic Instrumentation: Semiconductor Radiation Detector Research. – A diffusion furnace has been set up for making phosphorus-diffused p-n junction diode detectors. Studies of the diffusion coefficient for phosphorus into silicon in the temperature range 600 to 1000°C have been made. High-pressure steam oxidation of the silicon as a means of providing edge protection is being investigated. It appears that a layer of silica or quartz, grown on the silicon at high temperatures and pressures, may make both surface-barrier and p-n junction diodes more reliable and relatively independent of gas ambients. A new construction technique for surface-barrier diodes has yielded detectors which will survive 4000 v bias. The diode noise at this bias is too high to permit operation as a detector. However, good operation is obtained at 1000 v. This new technique involves the use of two casting resins (epoxy): one resin contains an amine hardener which leaves the surface n-type; the other resin contains an acid-type hardener and is doped with I_2, leaving the surface p-type. This approach was suggested as a result of studies, under this program, of the surface states on silicon and the effects of gas ambients and edge-protection materials.

ISOTOPE DEVELOPMENT PROGRAM

Recovery of Fission Products and Transuranics from Waste. – In preliminary tests with acid simulated Purex waste ~85% of the zirconium was batch-extracted (one stage) in 1 hr with 0.3 M di-2-ethylhexylphosphoric acid (D2EHPA) and 0.15 M tributyl phosphate (TBP) in Amsco 125-82. Oxalic acid, ~1 M, stripped the zirconium from the organic phase.

Radioisotope Research and Development. – An improved radiation source has been developed by utilizing the ion exchange and glaze-forming characteristics of Decalso, a commercial aluminosilicate ion exchange compound. The source is fabricated by first adsorbing a radioactive cation on finely ground Decalso from a neutral or basic solution, drying the loaded Decalso, adding calcium oxide as a fluxing agent, and firing at 1100°C. The resulting glaze will adhere firmly to ceramic and metallic backings and is resistant to flaking, to leaching by the common acids and bases with the exception of hydrofluoric acid, and to abrasion. A controlled film thickness as thin as 1.0 μ has been applied to a metallic backing, and much thicker sections can be applied. Such thin films allow a high efficiency of emission of beta or alpha particles from the source material. Several hundred flat, rectangular U^{233} alpha sources have been fabricated by this technique. Radioisotope sources of this type will have application at ORNL as standard or instrument check sources in place of existing sources fabricated by the evaporative technique.

The first shipment of fission product fractions adsorbed on Decalso in shielded casks was made from Hanford Atomic Products Operation to ORNL.

A total of 120,000 curies of Cs^{137} and 20,000 curies of Sr^{90} was shipped in five casks without incident. En route, observations of accelerative forces on the casks and conditions pertinent to future shipments were made by two ORNL engineers.

Design is in progress on a semicontinuous loop process for the production of I^{125} by irradiating normal xenon in the LITR. A test irradiation of a batch of pressurized xenon in the ORR yielded 135 mc of I^{125}.

Radioisotope and Stable Isotope Production. – Suitable charge materials were prepared for the separation of the isotopes of Ca, Dy, Eu, Mg, K, Sm, Sn, Yb, and Zr. Tungsten and uranium materials were also furnished for exploratory work.

The chemical recovery of Dy, Yb, K, and Sm from calutron components used in the recent isotopic separations of these elements is continuing. Recovery operations on europium have been completed; 94% of the starting material has been recovered and returned to inventory. Some 600 g of gallium as metal has been recovered from salvage operations.

Collections of separated isotopes, as estimated from ion current readings, totaled 2284 g. Elements processed included Ca, Cl, Dy, Mg, K, Sn, Yb, and Zn. Separations were performed in the four calutrons in Building 9731 and in the 24 calutrons in Building 9204-3.

Chemical recovery and refinement were completed on 22 new inventory lots of Ca^{48}, Cd^{110}, Cd^{112}, Cl^{35}, Cl^{37}, Fe^{54}, Fe^{57}, Fe^{58}, Ir^{191}, K^{39}, K^{40}, K^{41}, Pb^{208}, Zr^{94}, and Zr^{96} and on current returned samples of Cr^{54}, Te^{128}, Ti^{46}, Ti^{50}, and W^{186}. Sixteen new inventory lots and eighteen current returned samples were prepared for inventory following refinement and spectrochemical and mass analyses.

The inventory lots of Cl^{35} and Cl^{37} were products of a special effort to obtain gram quantities of these isotopes having an isotopic purity above 99%. This is the highest purity yet obtained for chlorine in a single-pass calutron separation.

As part of the refinement and development program, a large number of individual samples were recovered on a preliminary basis in order to obtain retention, charge contamination, or mass assay data.

Currently in varying stages of recovery and refinement are new enriched isotope lots of Ca, Cd, Cl, Ga, Fe, Ni, K, Ta, Yb, Dy, and Zr.

Conversions of inventory forms to more suitable forms for customer use included: Li^6 metal → LiF, Li^6 metal → Li_2CO_3, Li^6 metal → Li_2SO_4, $Sn^{120}O_2$ → Sn, $Ge^{70}O_2$ → Ge, $Zn^{64}O$ → Zn, $Fe_2^{54}O_3$ → Fe, B^{10} element → H_3BO_3, B^{10} element → B_2O_3, $NaCl^{35}$ → RbCl, NH_4Br^{79} → $PbBr_2$, $Ti^{48}O_2$ → Ti, $Sr^{87}(NO_3)_2$ → SrO, $Cd^{112}O$ → Cd, and $Pb^{208}O$ → Pb.

One special target was prepared for Los Alamos from 27.382 g of enriched Pb^{204} by casting the metal into a cylinder with an outside diameter of 0.625 ± 0.01 in. and a height of 0.48 in.

Inventory samples of $Zr^{91,92,94}O_2$ and $Ti^{48}O_2$ were reduced to finely divided metals, formed into crystal bars, and rolled into thin foils for accelerator studies.

Silver tellurate was prepared from several isotopically enriched samples of tellurium and used for the first time as an assay material. This compound, although not ideal, appears to be superior to either the element or the oxide used previously.

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