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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

DECEMBER 1965

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December 1965

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

DECEMBER 1965

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

REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

EGCR Fuel Irradiations. — The EGCR-vendor fuel material in poolside capsule 04-8 reached an estimated burnup of 7263 Mwd per metric ton of UO_2. The capsule continued to operate satisfactorily at a maximum measured cladding temperature of 1540°F (838°C).

Compatibility of Oxide Fuel and Graphite Under Irradiation. — We are conducting experiment 05-10 in the ORR poolside facility to evaluate the compatibility of $(U,Th)O_2$ fuel pellets with graphite under high-temperature gas-cooled reactor conditions. Irradiation continued with the fuel-graphite interface maintained near the design value of 1370°C and swept with an inert gas containing 230 ppm of CO. The indicated maximum UO_2 temperature is 1650°C. The infrared CO analyzer indicates a slight increase in CO concentration of the outlet sweep gas over that of the inlet. However, it is known that the analyzer is affected by the radioactivity in the outlet gas. We are trying various techniques to obtain a better estimate of the change in CO concentration. We have experienced difficulty in measuring fission-gas release rates because of the high activity level of the samples. Measured values (expressed as R/B, ratio of release rate to birth rate) are approximately 1.5×10^{-2} for ^{135}Xe and 5.3×10^{-2} for ^{133}Xe.

BeO-Graphite Compatibility Experiment. — The capsule for studying the compatibility of BeO with graphite in the presence of irradiation was removed from the reactor at the end of ORR cycle 62 (December 12). Irradiation was started March 12, 1965. The temperature of the BeO-graphite interface was maintained near the design value of 1500°C during the first half of the irradiation period. The indicated temperature then gradually declined, reaching approximately 1260°C at the time of removal. The total fast-neutron exposure is estimated to have been 1.3×10^{21} neutrons/cm^2 ($E > 0.18$ Mev).

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Prior to removing the capsule from the reactor, gas lines leading into the specimen region were sealed by injecting epoxy resin to fill traps provided for this purpose. We are making special efforts to exclude air and moisture from the specimens until the capsule is opened in a special hot-cell dry box for postirradiation examination.

High-Burnup Coated-Particle Irradiation Experiment. — We are conducting an experiment to evaluate the performance of loose pyrolytic-carbon-coated fuel particles in a graphite container at high burnup (50 at. % of the heavy metal) under conditions of interest to high-temperature gas-cooled reactors. The capsule was temporarily removed from the ORR F-9 facility on November 30, 1965, so that the facility could be used for fuel meltdown tests. The capsule was stored in the ORR pool in a holder provided for this purpose and was reinstalled in the reactor on December 21. The estimated burnup as of November 30 was 32.5 at. % of the heavy metal in the UC_2 particles in the lower fuel compartment and 30.5 at. % of the heavy metal in the sol-gel UO_2 particles in the upper compartment.

Fast Gas-Cooled Reactor Irradiation Tests. — We are performing a series of capsule irradiation tests in the ORR poolside facility to support the development of fuel elements for a fast gas-cooled reactor. These tests in a predominantly thermal-neutron spectrum are intended principally for investigating fuel-cladding interactions. The General Atomic fast gas-cooled reactor design (GCFR) is used as a reference in the selection of test parameters.

Irradiation of the second capsule, designated P4B-2, which began on October 21, was terminated as scheduled on December 12, 1965. This capsule contained two fuel specimens (prepared by General Atomic) consisting of UO_2 pellets of special shape encapsulated in 0.375-in.-OD Hastelloy X tubing. The tubing wall thickness is 0.015 in. in one of the specimens and is tapered from 0.010 to 0.020 in. in the other. The external pressure in this test was 940 psia. The maximum cladding temperature was maintained at 1400°F (757°C) during the first two days of irradiation and was then reduced to 1250°F (678°C) for the balance of the irradiation period. This reduction was accomplished by moving the capsule to reduce the neutron flux and, hence, the power from approximately 20 to 17 kw/ft, as estimated from the thermal design. The irradiation proceeded satisfactorily. We applied a total of 26 thermal cycles and saw no evidence of leakage from the fuel cladding. Postirradiation examination is in progress.

Strength of Porous-Carbon Coatings. — We studied the strength and deformation properties of porous coatings deposited from acetylene. When we plotted the average crushing load per particle vs the density, only a wide scatter band of data was obtained. However, the data selected were from batches containing large (460-μ) or small (200-μ) microspheres and varied widely in total coating thickness. When the data were expressed as the crushing load per unit of coating thickness, the correlation with density was much more evident. A plot of these data vs the density could be fitted to a least-squares line having a base intercept (showing no significant strength) at a density of approximately 0.5 g/cm^3.

In an attempt to confirm the compressibility of porous coatings we determined stress-strain relationships of coatings of different densities by measuring the change in diameter of a single coated particle as a function of the applied load. We found that low-density porous coatings (0.6 g/cm^3) deformed as much as 35% before crushing, whereas coatings with intermediate or high densities showed maximum deformations of approximately 15%. Furthermore, the deformation of such porous coatings was observed to be almost a linear function of the load, but the deformation rate of high-density coatings decreased greatly at higher loads. This behavior certainly contributes to the demonstrated capability of porous coatings for protecting high-density outer coatings from mechanical damage during irradiation.

Effects of Heat Treatment on Pyrolytic-Carbon-Coated Oxide Particles. — We extended the out-of-pile heat treatment of pyrolytic-carbon-coated UO_2 particles to 680 hr at 1900°C and 500 hr at 2000°C in a vacuum of 10^{-5} torr. Fuel migration into the coatings of all batches, as determined by microradiography and metallography, was slight or even undetectable, except for one batch that had an outer coating deposited at 1800°C over a porous inner layer. Particles in this batch developed a layer of graphite at the UO_2-carbon interface, and as much as 8 to 10 μ of fuel migration into the porous coating could be detected. This phenomenon must be attributed to a slight permeability of the outer coating or to the formation of small amounts of UC_2 during initial stages of deposition of the outer coating at 1800°C.

Operation of EGCR Main Blowers. — We investigated analytically the ability of the main blowers of the EGCR to function following a failure of the blower seal-water system. From an analysis based on determining a time-dependent temperature distribution in the blower end-bell housing region, we concluded that if the main blowers are operated for any appreciable length of time following the seal-water failure, the blower bearings will fail because of a reduction in the fluid film thickness.

REACTOR FUELS AND MATERIALS

Materials Compatibility. — Our continuing study of the compatibility of refractory metals with molten alkali metals now centers on the corrosion reactions stemming from the presence of oxygen in the system of potassium and Nb—1% Zr. We heated two groups of Nb—0.86% Zr alloy specimens, differing in oxygen content, for 100 hr at 815°C in Nb—1.05% Zr capsules with potassium containing 1500 ppm dissolved oxygen. Specimens containing 2100 ppm oxygen (less than two oxygen atoms per zirconium atom) gained weight by taking up oxygen. Those with 4100 to 4700 ppm oxygen (more than two atoms per zirconium atom) lost oxygen. A pretest heat treatment at 1200°C given some specimens did not affect oxygen exchange. After the test, all specimens were partially covered by a dark, tightly adherent corrosion product less than 0.001 in. thick, identified tentatively by electron diffraction as NbO_2. The oxygen content significantly increased in the area of the capsules that contacted the liquid. Hardness

measurements showed the oxygen to be restricted to the first 0.010-in. layer of the capsule wall. Interaction with the capsule and the specimen reduced the oxygen level of the potassium to less than 50 ppm.

Behavior of High-Temperature Materials Under Irradiation. — The status of the program is summarized in the table below. The irradiation facility used is the Engineering Test Reactor J-12 position, which contains a four-hole filler piece that permits simultaneous irradiation of as many as four capsule assemblies.

Status of Irradiation Experiments

Capsule Assembly	Specimens	Expected Maximum Fast-Neutron Dose (neutrons/cm^2, >1 Mev)	Irrad. Temp. (°C)	Status
		$\times 10^{21}$		
41-23	1/2-in. × 1/2-in. cylinders: Al_2O_3, Y_2O_3, MgO, hot-pressed carbides of Ti, W, Ta, Zr, and Nb (total = 60)	7	~250	Undergoing irradiation
41-24	1/2-in. × 1/2-in. cylinders: Al_2O_3, Y_2O_3, MgO, both hot-pressed and slip-cast carbides of Ti, W, Ta, Zr, and Nb (total = 64)	4	~250	Irradiation completed
41-25	1/2-in. × 1/2-in. cylinders: explosion-pressed carbides of Ti, Ta, W, Zr, and Nb (total = 60)	4	~250	Undergoing irradiation
41-26	1/2-in. × 1/2-in. cylinders: nitrides of Ti, Ta, Zr, Y, and Nb (total = 60)	4	< 100	Still in design stage
41-27	1/2-in. × 1/2-in. cylinders: Al_2O_3, Y_2O_3, graphite, both hot-pressed and slip-cast carbides of Ti, W, Ta, Zr, and Nb (total = 52)	4	~1100	Irradiation scheduled to start in January 1966
41-28	1/2-in. × 1/2-in. cylinders: explosion-pressed carbides of Ti, W, Ta, Zr, and Nb, graphite, coated particles (total = 42)	>1	1100–1400	Undergoing assembly; irradiation scheduled to start in February 1966
41-30	Tensile test specimens (total = 126)	0.6	~150	Postirradiation examinations in progress
41-31	Tensile test specimens (total = 126)	0.6	~600	Being fabricated; irradiation to begin in January 1966
41-32	Tensile test specimens (total = 126)	0.6	~150	Undergoing irradiation

Electron-Beam Floating-Zone Refining of Niobium. — Niobium metal from two different sources was floating-zone refined using electron-beam heating. The impurity content along the length of the zoned rods was evaluated as a function of total pressure during zoning and number of zoning passes. This was done using resistance ratios; vacuum fusion analyses for O_2, N_2, and H_2; Leco combustion analyses for carbon; neutron activation analyses for tantalum and tungsten; and spark mass spectrometric analyses for other metallic impurities.

The results indicated that most of the removal of metallic impurities was by evaporation, since there was no significant variation of metallic impurity content along the zoned length, with the possible exception of zirconium and copper. However, the metallic impurity content generally decreased with increasing number of passes. Tungsten and tantalum were exceptions to this. The amounts of these two metallic impurities did not vary along the zoned length and did not decrease with increasing number of passes.

There was no statistically significant variation of C, O_2, H_2, or N_2 content along the zoned length. Also, C, O_2, and H_2 exhibited little change in content for increasing number of passes after the first pass. However, the N_2 content decreased with increasing number of passes.

The resistance ratios, as measured along the zoned rods, showed a slight variation, with the higher ratios at the finish end of the zoned length. The first pass resulted in the greatest change per pass in overall resistance ratio of the zoned rods. Subsequent passes had a continual decreasing effect upon the amount of increase in the resistance ratio.

It is concluded that most of the purification of niobium occurred by evaporation and perhaps some gas and gas-metal reactions.

Tensile tests were made at room temperature on specimens taken from zoned niobium single-crystal rods that were seeded to a tensile axis orientation near the center of the unit triangle. Slip-line morphology and rotation of the tensile axis were recorded as a function of strain. The primary slip system was (101)[111]. Extensive latent hardening on secondary slip systems was indicated by the rotation of the tensile axis; the rotation overshot the (001)-(011) symmetry boundary by 8°. The appearance of secondary slip lines and the deviation of the tensile axis rotation from the path predicted by primary slip both occurred at about 35 to 40% tensile strain. This also was the end of stage I on the stress-strain curve.

Fuel Element Development. — Studies of the thermochemical deposition of refractory metals and alloys have continued. Deposition of tungsten-rhenium alloys in both a moving and a stationary hot zone is being investigated. The moving-hot-zone technique, wherein the hot zone is provided by an induction coil, shows promise for depositing alloys of uniform composition and thickness except for circumferential ridges that vary in height, width, and spacing. For example, in a nominally 20-mil-thick deposit the ridges are about 20 mils high, 20 to 40 mils wide, and spaced 1/16 to 1/4 in. apart. The ridges have been characteristic of the moving-hot-zone technique; their cause is not yet known.

Another technique for preparing tungsten-rhenium deposits consists in deposition on the outer surface of an internally heated mandrel in a

relatively large mixing chamber. A deposit prepared at 600°C, a pressure of 10 torrs, and gas flow rates of 2000, 60, and 20 cm^3/min of H_2, WF_6, and ReF_6, respectively, had a rhenium content of $\left(25.2 \begin{smallmatrix} +1.2 \\ -1.9 \end{smallmatrix}\right)\%$ over an 8-in. length.

Molybdenum has been deposited by hydrogen reduction of MoF_6 at 600, 700, and 800°C. Smooth deposits were obtained at 800°C, while at lower temperatures we obtained whiskers (needles) and an amount of MoF_3 that increased with decreasing temperature.

FUNDAMENTAL HEAT TRANSFER AND FLUID DYNAMICS

Boiling in Multirod Geometries. — We resumed critical heat flux experiments with a steam-heated tube mounted in the central position of a seven-rod cluster in which the six peripheral rods were electrically heated. A thermocouple probe consisting of four couples at 90° circumferential spacing was located within the steam-heated tube at a position 1/2 in. below the top of the heated section. With a saturated water flow rate of 1 gpm and a power level of 3.5 kw per rod, the steam pressure inside the heater was raised progressively until a transition was indicated by the sudden increase in reading of one of the thermocouples. The precise pressure level (temperature) at which transition occurred was observed to be dependent on the rate at which the data were taken. Subsequent experiments, very carefully performed, failed to reproduce the transition at steam pressures up to 20 psig, and we concluded that it was probably due not to inception of film boiling but to some other effect, such as activation of a nucleation site or overcrowding of sites.

Hydromagnetic Stabilization of Vortex Flow: Experimental. — We measured the effect of bleeding off a portion of the boundary layer through slits in the vortex-tube wall with a 1-in.-diam, 6-in.-long tube in which the vortex flow field was generated by 142 0.052-in.-diam round nozzles arranged in three rows. The boundary layer was bled off through thirty 0.006-in. slits uniformly spaced around the circumference and extending the full length of the tube. These were hydrodynamic experiments with water at 60°F for preliminary evaluation of the technique.

We observed first that without wall bleed, the boundary layer along the concave wall became unstable at a tangential Reynolds modulus of 230 (corresponding inward radial Reynolds modulus of 7.4). Second, the tangential Reynolds modulus at transition to instability increased approximately linearly with increasing bleed flow; with all-bleed flow (outward radial Reynolds modulus of 14.0) the tangential Reynolds modulus increased to 500. However, the radial Reynolds modulus based on through flow (i.e., inward flow leaving the center of the tube) had to be decreased as the bleed flow was increased to effect stabilization, and hence we are not certain whether the observed increase in Reynolds modulus with increasing bleed flow is attributable to a stabilizing effect of the latter or to the effect of the change in velocity profile with decreasing inward flow. Third, the recovery of injection velocity as tangential velocity is essentially independent of bleed flow under stabilized conditions. We interpret this as possibly indicating that the increased

laminar wall shear resulting from reduction in boundary-layer thickness offsets the increased momentum input as the jet velocity and mass flow are increased. We anticipate repeating these experiments with an electrically conducting liquid in a strong magnetic field.

Hydromagnetic Stabilization of Vortex Flow: Theoretical. — We extended the analysis of nonlinear hydromagnetic stability for flow between insulating walls to include the second-order nonlinear approximation. The difference between the two results was much larger than expected — from about 10% to as much as 500%.

Comparison of the first- and second-order results indicates that the effect of including the second-order terms is to reduce the rate of energy dissipation in the flow. The reduction in dissipation apparently occurs because the smaller-wavelength second-order component of the disturbance derives most of its energy from the first-order component rather than from the main flow and thus reduces the overall level of disturbance. We found, for the first-order calculations, that when the results are correlated by plotting the percentage increase in energy dissipation produced by the disturbance vs the percentage increase in Taylor number above the critical value, the resulting curves are nearly independent of the Hartmann modulus; they change at most by about 15% over the entire range of Hartmann modulus. With the second-order results, on the other hand, the sensitivity to Hartmann modulus is much greater: for a radius ratio of 0.25, increasing the Hartmann modulus from 0.0 to 100 decreases the energy dissipation by about 57%.

Vortex Flow near a Stationary Disk. — We continued our theoretical studies and achieved stable (nonoscillating) solutions of the complete Navier-Stokes equation for values of the tangential Reynolds modulus up to 2.5. However, we are unable to obtain nonoscillating solutions at $N_{Re,\theta}$ greater than about 5; this indicates that the boundary conditions used are no longer physically meaningful and that some of the assumptions made in deriving the equations are no longer valid.

We also continued our experimental measurements with a detailed study of the flow in the free stream (away from the walls). Our data show qualitative agreement with the theoretical study of Donaldson, and we find that at high rotations and low through flows as much as 80% of the flow bypasses the free stream and goes directly into the end-wall boundary layers.

We have made an extensive analysis of data from which the ratio of radial-to-tangential velocities as a function of axial distance from the stationary disk can be determined. Since our results did not exhibit radial similarity when the "classic" dimensionless axial distance ($\eta = \sqrt{N_{Re,\theta}}\,\frac{z}{r}$, where $N_{Re,\theta}$ is the tangential Reynolds modulus of the flow, z is the axial distance from the disk, and r is the local radius) was used as the independent variable, the data were reanalyzed using both $\eta = \sqrt{N_{Re,\theta}}\,z$ and $\eta = \sqrt{N_{Re,\theta}}\,zr$ as the independent variable. Neither of these cases exhibited radial similarity, however, and we have concluded that the flow cannot be described in terms of ordinary similarity variables in the Reynolds modulus range studied (100 to 1000).

POWER REACTOR FUEL PROCESSING

Close-Coupled Process Development. — Since refabrication of thorium fuels will require shielded facilities, coupling the reprocessing plant to the refabrication plant (and possibly to the reactor) may decrease fuel-cycle costs to a minimum. This goal seems especially attainable if a simplified, low-decontamination process can be developed to provide feed for an improved sol-gel process. Thus, we are studying new approaches to remotely operable methods for decontaminating and recycling thorium fuel and are preparing an engineering cost evaluation of these new concepts.

Further tests with the batch differential contactor confirmed the previously reported coextraction of zirconium and fluoride with the uranium and thorium. About 60% of the zirconium introduced into the shear-leach dissolver product precipitated during adjustment of the feed to acid-deficient conditions, but it did not interfere with solvent extraction. Most of the soluble zirconium and 25% of the fluoride accompanied the uranium and thorium into the tributyl phosphate (TBP). Zirconium and fluoride extract because of the highly concentrated feeds required to saturate the solvent in order to ensure good separation of the fission products. If the zirconium and fluoride interfere with the sol-gel process or the refabrication of the fuel, we may have to include a scrubbing step in the solvent extraction procedure.

Unfortunately, a high-density, second organic phase forms when the thorium concentration exceeds 0.2 M in 30% TBP. We observed that the dense phase which separated from the first 0.1 volume of solvent that had been equilibrated with a synthetic Thorex feed solution was 0.67 M in Th and 0.48 M in U. This third phase thus presents problems related to criticality and to the design of equipment. Accordingly, we have begun to study other solvents to find one that has all the desirable features of 30% TBP—70% n-dodecane without this undesirable one. Tri-n-pentyl phosphate is being synthesized and will be tested.

We are investigating what may be a simpler approach to fuel reconstitution. The present ORNL sol-gel process produces sols by thermal denitration of thorium nitrate and suspension of the resulting ThO_2 powder in water. We are now looking into a one-step method in which the nitrate in the uranium-thorium solutions from the stripping column is removed by extraction with long-chain amines. The fact that both uranium and thorium can be denitrated this way represents a distinct advantage over the present sol-gel process. So far, Amberlite LA-2 is the best amine. It is diluted with n-dodecane.

Thorium solutions up to 0.4 M (U:Th ratio, 0.1) were denitrated by contacting them with the amine (1.5 moles of amine per mole of nitrate) for 30 min at 95 to 100°C. However, solutions that were 0.6 M in thorium, or more, formed solids during the first 3 to 5 min of contact with the amine but liquefied after another 5 to 10 min of contact. The resulting sols had a nitrate-to-metal ratio of about 0.3. This is much higher than permissible with the present sol-gel process, but it still results in apparently acceptable gels and fired products. A sol 0.16 M in thorium, 0.015 M in uranium, and 0.06 M in carbon (from retained amine), with a nitrate-to-metal ratio of 0.32 was evaporated at 100 to 105°C to

produce a fluid, stable concentrate. The concentrate was 4.54 M in thorium, 0.42 M in uranium, and 0.2 M in carbon, and the nitrate-to-metal ratio was 0.27. Next, we will try to make microspheres from the concentrate.

FUEL-CYCLE STUDIES

Pressing and Sintering, and Extruding to Form Sol-Gel-Derived Fuels. — We are investigating the fabrication of ceramic nuclear fuels by extrusion of sol-gel materials. This is being done to find more economical ways of making small-diameter fuel rods from radioactive materials where remote fabrication is necessary.

To determine the effect of moisture content of the sol-gel "clay" on shrinkage and cracking during drying and firing, a series of air-dried clay samples were extruded. These had moisture contents of 72.5, 66.5, 65.1, and 63.6 vol % when extruded. The shrinkage on drying decreased with decreasing moisture content, as expected. Further evaluation of the characteristics of these samples is under way.

We have obtained a 2-kg batch of ThO_2–5% UO_2, 10 M colloidal sol-gel clay. It will be used for initial evaluation of the effect of this concentration of UO_2 on clay behavior. Results of the first tests indicate that it cannot be extruded at as low a moisture content as the pure ThO_2 clay and that it may be fired in air or vacuum at 1200°C. No effect of this treatment on sample characteristics could be found.

Conversion of $(Th,U)O_2$ to $(Th,U)C_2$. — We made further studies of the conversion of sol-gel $(Th,U)O_2$ microspheres (25% UO_2) to $(Th,U)C_2$ by heating the solid-solution oxides with lampblack in a graphite crucible rotating in a flowing argon atmosphere. The oxide particles were 177 to 250 μ in diameter, the carbon-to-oxide weight ratio was 1:1, and the speed of rotation of the crucible was about 40 rpm.

About 10% of the particles examined metallographically contained small unconverted spots near the centers of the particles after heating for 5 hr at 2000°C. Only about 1% of the particles was not completely converted after heating at 2100°C for 3 hr.

Vibrational Packing of Microspheres. — During the past month a laboratory investigation of the packing of ThO_2 microspheres by relatively low-energy vibration was started. Although the results to date are preliminary, it is apparent that packed densities in excess of 80% of theoretical can easily be achieved. Two size fractions of thoria microspheres were used in the initial tests. The coarse material was −35 +40 mesh (diameter, 500 to 420 μ) produced by the sol-gel column method, while the fines were all smaller than 325-mesh (<44 μ) material produced by the sol-gel "stirred-pot" method.

All attempts to mechanically blend the two sizes failed. Simultaneous, separate feeding of the two fractions also resulted in considerable segregated layering. The best results were achieved by first packing the coarse particles with a heavy follower rod to produce a density nearly 60% of theoretical. The fines were then poured on top of the packed column, the rod inserted, and, in a relatively short time under

vibration, the fines had filtered down into the packed bed. With a 70% coarse, 30% fine fraction mixture, densities up to 81% have been achieved in glass tubes. Next month, tests will be conducted in metal tubes in which additional impact energy can be used, and the optimum percentages of the two size fractions can be determined.

Sintering of Sol-Gel Oxides. — We have made and sintered a series of isostatically pressed pellets from calcined ThO_2–8 wt % UO_2 to determine the effect of powder-calcining temperature, isostatic pressure, and sintering temperature on the final density of the pellets.

The data have not been fully analyzed; however, we see that green density at any one isostatic pressure decreases with decreasing powder-calcining temperature from 900 to 650°C, but then increases again as the calcining temperature is lowered to 400°C and below. Also, sintered density increases with increase in isostatic pressure for any one calcining temperature, and sintered density increases with a decrease in powder-calcining temperature for any one isostatic pressure. We feel that the best approach to producing high sintered densities is to calcine the powder at a low temperature and isostatically press at a high pressure. Of course, sintering temperature and other sintering conditions could also affect the final density. It is clear that high-density pellets can be made from sol-gel-derived ThO_2–8 wt % UO_2 powders.

Sol-Preparation Studies: Thoria-Urania. — One objective of these studies is to determine whether sol-gel technology can be extended to include mixtures of thoria and urania in all ratios. We are concentrating our efforts on an approach in which sols are mixed in the desired ratios for the variety of fuel compositions that we anticipate will be studied in the development program in the Thorium-Uranium Recycle Facility. The original sol-gel process is limited to mixtures containing less than 10% uranium, which is added to thoria sol as uranyl nitrate.

In the present mixed-sol approach, a thoria sol is mixed directly with a urania sol. Other possible methods for preparing mixed sols, such as solvent extraction of nitrate ion from mixed thorium-uranyl nitrate solutions, dispersal of precipitated thorium hydroxide with uranyl nitrate, and coprecipitation of uranium and thorium hydroxides followed by peptization in nitric acid, are also being investigated.

The thoria sols have been prepared by several methods, each sol having a different thoria crystallite size and nitrate ion concentration. These sols have been blended with urania sols, and in each case conditions have been found under which useful mixtures with a thoria-to-urania ratio of ~3.5 can be made. In this way we are obtaining insight into the fundamental nature of stable thoria-urania sol mixtures. Firing studies on microspheres prepared from mixed sols having a thoria-to-urania ratio of 3.5 have shown that some method of carbon removal must be used if dense microspheres are to be produced. The carbon is introduced in the form of alcohol and surfactant during sphere formation and can be removed by oxidation with air or CO_2.

Sol-Preparation Studies: Urania-Zirconia. — The increased strength of urania microspheres containing up to 20 vol % zirconia provides an incentive to study ways of making mixed urania-zirconia sols. The method being studied includes the addition of zirconium as zirconyl nitrate or

as zirconia sol to a U^{4+} solution. The mixture is converted to a mixed-sol system by the precipitation-peptization method developed for urania sols (ORNL-3874). When zirconia sol is used, it is prepared by titrating a zirconyl nitrate solution to pH 1.5 to 1.7 with ammonium hydroxide, and boiling to produce an opalescent ~0.5 M zirconia sol. Mixed sols prepared with zirconia sol are less viscous than those prepared with zirconyl nitrate. Several batches of microspheres prepared from the mixed sols have been fired, and the expected threefold to fivefold increase in crushing strength over that of pure urania has been obtained. However, it may be necessary to increase the firing temperature to above 1150°C to produce near-theoretical density.

Sol-Preparation Studies: Thoria-Carbon. — Thorium dicarbide microspheres can be prepared from mixed thoria-carbon sols. This method leads to a low-density (<90% of theoretical) product, and in the relatively few experiments performed so far, small amounts of unreacted ThO_2 have been present in the product. Present studies are designed to determine what control can be achieved over density and residual oxide (or free carbon) content. In addition to their use for carbide preparation, mixed oxide-carbon spherules are used in some methods for nitride and sulfide synthesis.

Of the five carbons and channel blacks tested in the current series of tests, the material having the lowest specific surface area has given the mixed sol of lowest viscosity. If viscosity is too high (above about 60 centipoises) the sols are not manageable. It appears that about 3.5 M is as high a ThO_2 concentration as is practical when the carbon-to-thoria mole ratio is about 4. This is the ratio required to form ThC_2. The first results of sphere-forming attempts with the mixed sols indicate that only a narrow range of successful forming conditions exists. Conversion of some of these spheres to the dicarbide appears to have been successful when a maximum temperature of 1785°C and vacuum were used.

Sol-Preparation Studies: Urania-Molybdena. — A method for producing controlled porosity in urania microspheres is being studied. The method is based on forming a urania-molybdena sol which is gelled and then fired to remove the volatile MoO_3. So far, several grams of ~200-μ-diam UO_2 spherules containing 1- to 10-μ-diam pores have been prepared. If the firing is done in a reducing instead of an oxidizing atmosphere, a UO_2-Mo cermet results.

Coated-Particle Development Laboratory. — This laboratory was set up to develop methods for making carbon-coated microspheres of thorium and uranium oxides and carbides and their mixtures. Equipment and procedures developed here will be used in the shielded Thorium-Uranium Recycle Facility now under construction.

The design of the microsphere-forming column being studied is based on the fact that sols of thoria and urania can be set to gels when sufficient water is extracted from them. In the column an immiscible alcohol is passed countercurrent to sol droplets, extracting water from the droplets and setting them to spheres of gel. These spheres settle to the bottom of the column as they shrink and densify. The development of dispersers to form sol droplets of closely controlled diameter is important.

The column was operated with thoria sol during the day shift for five days at a rate of about 500 g of ThO_2 microspheres per hour. A shear nozzle was used as the sol disperser. The size distribution of the product was approximately the same as that obtained under the same conditions in the smaller column used for laboratory development studies. Two rotary dispersers, one with eight 0.016-in.-diam orifices and one with twelve 0.010-in.-diam orifices, were designed and made. The disperser with the 0.010-in. orifices should produce microspheres in the diametral range 210 to 250 μ.

During the past month a pneumatically operated particle-loading and -unloading device was added to the coating system. The fluidized-bed coating furnace was loaded with microspheres while at temperatures between 1100 and 1300°C, and was unloaded while at temperatures up to 1700°C. Such a means of remotely withdrawing samples from the fluidized-bed coater is necessary if the coater is to be used in a remotely operated facility such as the Thorium-Uranium Recycle Facility. Also, such a system will enable us to load and unload the coater without extended cooling periods, thus using the equipment more efficiently.

The pneumatically operated loading-unloading device was also used with the entrained-bed coater. The important result of this work was that the spherules were easily unloaded without putting the removal tube down into the entrained bed where the coating takes place, but could be removed from the gas stream at a fixed point. This obviates the need for a moving-particle pickup system, and avoids problems from soot accumulation on the pickup system.

We are developing an in-line monitor for determining the average coating thickness of pyrolytic carbon on ThO_2 microspheres. The monitor uses x-ray attenuation to determine the thickness.

Thorium-Uranium Recycle Facility. — The Thorium-Uranium Recycle Facility is a shielded facility being built to provide for development of methods of remote processing and fabrication of Th-^{233}U fuels. The building is made up of four 20-ft-wide cells having a combined length of 106 ft.

Construction is now nearly 35% complete. Almost half of the exterior concrete block walls are now in place, as well as most of the high-bay-area roof and the second-floor slabs. Stainless steel liner plates are ready for installation in one of the cells. Procurement is proceeding satisfactorily.

Equipment design effort is limited to checking and revising drawings pertaining to the liquid-handling equipment and to studying preliminary chemical and equipment flowsheets for high-uranium-content urania-thoria sols. Development designs were made, and some tests were performed on several components.

^{233}U Storage and Distribution. — Installation of the ^{233}U solution storage system, with a capacity of 400 kg of ^{233}U in nitrate solution at a concentration of 180 g/liter, is now complete. Borosilicate-glass Raschig rings were added to the required depth on the floor, and unit shielding of the tanks was completed. The safety circuits, which function to prevent a nuclear excursion in the event of human error, were tested, and operation was satisfactory. Acceptance by the ORNL Criticality Review Committee was received.

The glove-box facility for processing 1 kg of ^{233}U per day was equipped with gloves and sealed, and the ventilation system was placed in service. Determination of efficiencies of the filters is the final requirement before the introduction of ^{233}U into the system.

Fabrication of a geometrically safe ^{233}U dissolver was completed during the month and installation started. The dissolver will be capable of dissolving 5 kg of ^{233}U (as UO_2) daily.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — After extensive work on sealing penetrations, the leakage rate from the secondary containment (reactor and fuel drain cells) was well below the maximum acceptable. Extrapolation of leakage rates measured at 10, 20, and 30 to 39 psig gave less than 0.4%/day (<200 scf/day), compared with 1.0%/day assumed in the safety analysis.

Modifications on the radiator-door lifting and guiding mechanism were completed, and the radiator was heated to about 1100°F for hot testing of the door action. A satisfactory temperature distribution was attained in the radiator, indicating adequate sealing with the doors closed, and the doors worked freely in a brief test with the radiator hot. The test was terminated, however, when the area on top of the enclosure became too hot for the heater lead insulation and terminal blocks. After addition of more thermal insulation, the radiator was reheated and the enclosure top again became too warm. The new radiator door hoods interfere with the flow of ventilation air across the top of the radiator, and modifications are under way to install higher-temperature electrical insulation and redirect the flow of air in the area above the radiator.

Tests with the main blowers in operation showed that air leakage from the radiator duct will at times exceed the capacity of the coolant cell exhaust to the ventilation stack. Ducting is being installed to use an annulus blower to return air from the coolant cell into the radiator duct at the base of the coolant stack.

Startup of the reactor (without the coolant system) proceeded through checkouts, heating of the equipment in the reactor cell, flush salt circulation, fuel salt fill, and criticality.

The flush salt was circulated for 55 hr while nine samples were taken to determine the condition of the salt. The oxide content was proved to be quite low (about 100 ppm) by a newly developed analytical technique. Mechanical performance of all components was also good during flush salt operation.

Fuel salt was charged on December 19, and the reactor was made critical the next day. The critical rod position agreed closely with the predictions (+0.08% $\delta k/k$ difference), showing there had been essentially no dilution by residual flush salt. The first operation, at 1 kw, was movement of the nuclear chambers to approximately the operating locations. A flux traverse along the servo-driven fission chamber tubes showed that the newly installed, partial sleeves of cadmium were successful in increasing the attenuation to the value necessary for operation of the wide-range counting channels.

While the radiator modifications are delaying the filling of the coolant salt system, the fuel system is being operated subcritical or at very low power. Low-power experiments include rod-shadowing experiments and measurement of fluxes at the source tube and at the external reactor vessel surveillance specimens. Meanwhile, samples are being taken daily to establish the fuel salt composition at the beginning of power operation.

The computer is in routine operation, although portions of the reactivity balance calculation are not in their final form. Since the computer was accepted on October 1, there have been six downtimes for a total of 62 hr. (The lease contract allows only six times and 88 hr down in the first 12 months without penalty.)

Reactor Analysis. — The analysis of control rod differential worth measurements made during run 3 was completed. Stable periods measured during steady-state circulation were converted to differential reactivity worths, by means of an inhour-type formula developed to include the effects of circulation on the delayed-neutron precursors. The results were in good agreement with differential worths obtained from conventional analysis of period measurements made with the fuel stationary.

A mathematical curve fitting procedure was applied to the curves of experimental rod worth vs position, with the ultimate purpose of on-line computation of control rod reactivity with the TRW 340 data logger. The conventional first-order perturbation theory formula, which utilizes the flux distribution corresponding to all control rods fully withdrawn, proved to be inadequate for this purpose. By use of a simple theoretical model, this formula was modified to allow the axial flux distribution to be perturbed by the insertion of the shim rod bank. The resulting functional expression for the total reactivity worth of a given rod configuration depended on the positions of the regulating rod and the shim rod bank, and also contained several parameters which depended only on the separate poisoning effects of the fully inserted rods. Numerical values for these parameters were then obtained by a linear least-squares fit of this expression to the experimentally measured curves of rod worth vs position.

Development of Components and Systems. — Difficulty with the operation of the insulated doors in the salt-to-air radiator enclosure was traced to insufficient operating clearance between the door and the enclosure seal. The guide tracks for the radiator door were moved to provide a 1-in. clearance when the doors are off the closed position. It was necessary to increase the stroke of the cam-action lock which applies a sealing force in the closed position. The doors have operated successfully with the radiator at 1100°F, and no further trouble is expected from this source.

Analysis of the results of the ^{85}Kr experiment is essentially complete. Based on rate constants derived from this experiment and other sources, the predicted xenon poison fraction for the MSRE will be about 1.7%. This is higher than the original estimate of 0.9%, primarily because the pump bowl stripping efficiency was found to be about 10%, rather than 50% as originally estimated. Potential errors in this poison fraction calculation involve the assumptions that the circulating bubble concentration is zero and that the elements involved behave chemically as thermodynamically predicted.

A system is being designed to enable collection of samples from the reactor off-gas stream. The unit will include provisions for continuous analysis by thermal conductivity or gas chromatography, plus a cold trap for collection and concentration of samples which will be transferred to a hot cell for further analysis. Information as to the nature and concentration of relatively long-lived contaminants will be obtained. An estimate of the xenon poison fraction will be obtained by comparing relative concentration of xenon isotopes. The design is about 80% completed, and procurement of some components has been started.

MSRE Pump Program. — The modifications to the molten-salt prototype pump test facility were completed. It is ready for pump testing, and the prototype pump rotary element is being installed in it.

The design details for the MK-2 fuel pump tank were completed, and they are undergoing review by MSRE project personnel.

The modifications to the spare rotary assembly for the fuel pump to obtain positive sealing against oil leakage along the outside surface of the shield plug are being reviewed by project personnel. As soon as the drawings are approved, work will begin on modifying the spare rotary assembly for the fuel pump.

The PK-P molten-salt pump continued on endurance operation, circulating salt at 1200°F and 800 gpm, and has operated continuously for a total of 3984 hr.

NUCLEAR SAFETY

Behavior of Fission Products During In-Pile Destruction of Reactor Fuels. — The alterations of the in-pile fuel destruction facility (position F-9 in the ORR) to accommodate a simulated containment vessel are complete. The modifications to the facility were described in an earlier report (ORNL-3886, p. 14). Current emphasis is being given to fabrication of the secondary container for the vessel.

The experimental program to study the behavior of fission products released during simulated reactor accidents is being continued. A Zircaloy-clad UO_2 fuel sample was melted in-pile for a 5-min period in moist air. This experiment was performed to compare the behavior of iodine released from Zircaloy-clad UO_2 with the anomalous behavior of iodine observed when iodine was released in moist air from stainless-steel-clad UO_2 (ORNL-3788, p. 17). The apparatus has been segmented and will be analyzed for fission products. The apparatus for an experiment with a stainless-steel-clad UO_2 fuel sample has been assembled and is ready for installation in the reactor facility.

Characterization of Accident-Released Fission Products. — When composite diffusion tubes are used for the characterization of radioiodine species, high relative humidity interferes with the deposition of organic iodides on the charcoal-lined section. Earlier results showed that the moisture influence probably could be avoided by drying the air sample with Drierite (anhydrous $CaSO_4$) and by substituting a specially activated and impregnated charcoal (MSA 85851) lining for the regular charcoal (PCB) lining. Additional tests were made at 90°C and 75% relative

humidity, using varying amounts of Drierite, with both types of charcoal linings. When adequate amounts of Drierite were used, good characterization of the "A" fraction of an iodine source (mainly CH_3I) was obtained with both types of charcoal. If depletion of the desiccant occurred and some moisture penetrated, then the impregnated charcoal gave better results than the unimpregnated charcoal. Barium oxide was also tested as a potential desiccant; but it was found to adsorb more than 50% of the "A" fraction, whereas Drierite has shown an average retention of less than 1%. These results provide the basis for a method for the characterization of methyl iodide under various conditions up to 90°C and 75% relative humidity using composite diffusion tubes equipped with Drierite and an impregnated charcoal lining.

Investigation of iodine behavior in May packs was continued. A May pack of the type used in the Nuclear Safety Pilot Plant was tested with an air-steam atmosphere at 100°C, 2 atm absolute pressure, and 90% relative humidity. In general, the behavior was as predicted and similar to that of composite diffusion tubes at the same conditions, but before more precise comparisons can be made, further experimentation will be required. Construction of a more versatile facility for investigating the behavior of May packs under elevated temperatures and high humidities is almost complete.

Application of the fibrous filter analyzer to study the behavior of simulated accident-released particulate material in moist atmospheres and at elevated temperatures is being evaluated. Two problems are under concurrent study: (1) the physical response of the analyzer to an aerosol under conditions of high humidity, and (2) the production of a radioactive, monodisperse aerosol of known size with which to calibrate the response of the analyzer under various combinations of operating parameters. At low sampling velocities, 3 to 11 fpm, the fibrous filter analyzer shows promise as an analytical sampler in a wet atmosphere. The fiber configuration and filtration processes are not significantly affected by the moisture. A satisfactory monodisperse aerosol of known size has been prepared from a Dow polystyrene latex solution; however, efforts to tag it radiochemically have not been wholly successful. Other methods for generation of a monodisperse aerosol are being considered.

Removal of Methyl Iodide from Gases Under Reactor Accident Conditions. — In a previous report it was noted that a form of commercially available coconut charcoal (Mine Safety Appliances Co. No. 85851), specially treated and containing iodine, was found to be highly effective in trapping radioactive methyl iodide from humid air streams. Also, in some cases, it was demonstrated that even higher methyl iodide efficiencies could be obtained by experimentally impregnating various charcoals with elemental iodine, elemental bromine, or various salts of iodine and bromine. These tests were conducted at relative humidities of 60 to 70%. Of the impregnants tested, potassium iodide seemed to be the most efficient.

Results of recent experiments at 24°C and 90% relative humidity indicate that some loss of efficiency occurs as the humidity increases, from an efficiency of 99.99% to as low as 98.5%, in some cases, for 2 in. of charcoal impregnated with potassium iodide. The MSA 85851 charcoal under these conditions produced an efficiency of 89.8% for a depth of 2 in. Additional experiments are under way.

Noncombustible adsorbents such as activated alumina and molecular sieves have been impregnated with materials such as elemental bromine, potassium iodide, and triethylenediamine and tested for methyl iodide removal from air streams at 70% relative humidity. While charcoal impregnated with these materials gave excellent efficiencies for methyl iodide removal, the efficiency of impregnated alumina or molecular sieves for methyl iodide was essentially zero.

Ignition of Charcoal Adsorbers by Fission Product Decay Heat. — Out-of-pile charcoal ignition experiments are being continued to study and standardize experimental methods of performing in-pile ignitions. An apparatus has been made to cycle the ignition of charcoal beds 1.2 cm in diameter, 5.5 cm deep, and weighing 3 g. Temperatures are measured at the front and at one-third of the depth of the bed. Inlet air is preheated to approximately 15°C below the temperature of the bed. Several ignition tests have been made on different charcoals with this apparatus to determine in a preliminary manner the usefulness of the apparatus and the reproducibility of the data. Some interesting preliminary results are: (1) ignition temperatures are fairly reproducible; (2) in most cases moisture in the air reduced the ignition temperature below that observed in dry air; (3) the ignition temperature of commercially available "high ignition" charcoals is approximately 150°C above that of other charcoals and is not affected by humidity or gas velocity.

Additional experiments are being conducted to provide an understanding of the temperature profile in the charcoal bed as ignition occurs. These out-of-pile experiments are designed to provide the background to interpret data produced by in-pile experiments in which the decay heat of iodine contributes to the ignition of charcoal.

Particulate Materials from Reactor Accidents: Simulation and Filtration. — Additional experiments have confirmed the preliminary observation that moist conditions cause a drop in the efficiency of fibrous filters for trapping particulate aerosols of the type produced by the melting of UO_2 fuels. With ~95% relative humidity at room temperature, efficiencies as low as 93% have been noted, but, on the average, efficiencies of 98 to 99% have been obtained. These efficiencies are to be compared with >99.9% with a relative humidity of <3%. Investigations are being conducted to identify the mechanisms for this reduction in efficiency.

Photomicrographs of the aerosol produced with ~95% relative humidity reveal that the particles are coated with a thin film of unknown composition and that agglomerates are in the form of chains, rather than clusters as observed under dry conditions. Additional tests are planned to study this change in aerosol behavior.

Nuclear Safety Pilot Plant. — NSPP run 12 was made on November 12, 1965. The fuel material melted in this run was stainless-steel-clad simulated UO_2, containing Ru, Te, I, Cs, Ba-La, and Ce as the simulated fission products. Reducing conditions were maintained in the furnace by permitting steam to be present and by introducing some free hydrogen in the gas surrounding the plasma jet. The run was intended to duplicate run 10, and the results are similar to those obtained in run 10, as would be expected. However, removal of iodine from the model containment

vessel (MCV) atmosphere was appreciably slower, most probably as a result of the difference in condensate holdup. In contrast to run 11, which had an oxidizing furnace atmosphere, no indication of desorption was seen and no measurable quantity of activity was removed while purging.

The filter loop was started up 2 hr after the start of the run and was operated for 6 hr. The concentration in the MCV atmosphere was reduced to background during the first 2 hr of filter operation. Most of the activity was collected on the absolute filter.

Two remote sampling devices proposed for use in the LOFT experiment were tested: a plateout sampler and a gas sampler. Both functioned properly.

Nuclear Safety Information Center. — NSIC's selective dissemination of information (SDI) experiment was initiated in October. To date, six runs have been made in which the 50 interest profiles of the participating personnel have been matched against bibliographic entries in the Center's computer storage files. Initial response from the participants indicates that the experiment is going well. The program will be expanded to a much wider audience if the response continues to indicate that this is warranted.

Technical personnel in government service or with the nuclear industry who feel that they would like to participate in the SDI program may apply by advising NSIC of this desire in writing. The request should include a brief summary of the applicant's particular interest or speciality in the field of nuclear safety.

NSIC's third computer-sorted bibliography, Indexed Bibliography of Current Nuclear Safety Literature — 3, ORNL-NSIC-12, has been printed and distributed. Final review and editing of a state-of-the-art study that collects and evaluates design, cost, and operating data on secondary poison-injection systems have been completed. The report has been submitted for publication and will be issued in January 1966 as ORNL-NSIC-7.

During the report period NSIC received and answered 40 requests for information. In the same interval the staff conferred with 14 visitors.

Off-Shore Siting of Power Reactors. — At the end of the report period, the Laboratory contracted to conduct a study of the off-shore siting of boiling-water and pressurized-water reactors, using combinations of artificial islands and caissons located below mean sea level. It is expected that ORNL will act as the prime contractor on the study, subcontracting specific studies on engineering, oceanography, and water reactor systems as appropriate. The study is envisioned as having three phases:

1. The possibility of gaining basic "internal" safety advantages (those exclusive of the particular site environment) by the use of the caisson concept will be evaluated as a function of reactor power level and type.

2. Assuming positive results from the first phase, the basic conceptual arrangement and structural design for the reactor size and type selected in phase 1 will be developed.

3. A detailed structural design and cost estimate will be obtained for the basic concept and will be used in an attempt to assess the economic effect of variables such as site and reactor size, as well as the costs of alternate types of "improved" land-based containment.

To date we have undertaken a literature search on the task and are in the process of talking to a number of reactor manufacturers and architectural engineers who are competent in this area.

RADIOACTIVE WASTE DISPOSAL

Injection into Permeable Formations. — Recalculation of the activation energy of desorption of iodine by air from sediment on the Clinch River flood plain was made after a numerical error was found in the original calculations. The new value for the activation energy increased from 14.2 kcal/mole at a loading of 10^{-10} mole of I_2 per square centimeter to 54 kcal/mole at a loading of 10^{-11} mole of I_2 per square centimeter. These values of the activation energy of desorption are consistent with a system containing condensed I_2, plus some chemisorbed iodine on the mineral surfaces. Previously, it had been necessary to postulate that some of the I_2 vapor had been converted to HI, because the activation energy for desorption was lower than the heat of sublimation of I_2.

Intermediate-Level Radioactive Wastes. — Incorporation of intermediate-level radioactive waste in asphalt for storage or burial of the product is being studied as a general method for treating all types of waste in this category. The incorporation of inert solids in asphalt does not appear to present any additional hazard to that normally present when processing, storing, or shipping plain asphalt. However, the hazards involved in incorporating nitrate salts, such as those from ORNL evaporator concentrates, are being studied.

Atlantic Research Corporation (ARC), of Alexandria, Virginia, has been selected as a subcontractor competent to conduct tests to determine the safety characteristics of asphalt products that contain various amounts of nitrate salts.

Over 200 small samples of asphalt product in various configurations containing 0, 20, 40, and 60 wt % salts (0, 12, 23, and 35 wt % nitrate) from simulated ORNL evaporator concentrates were prepared and shipped to ARC for standard laboratory tests of stability, including drop weight, autoignition, shock sensitivity, and flame propagation. Eight large samples, 30-gal drums, were prepared and shipped for large-scale fire tests. The results of these tests will permit a better evaluation of whether or not asphalt plus nitrate salts from ORNL waste might be dangerous under some set of conditions.

Low-Level Radioactive Wastes. — The Water Recycle Process for treating low-level radioactive waste is being developed as a replacement for the traditional "treat and discharge" method of waste-water treatment. The process is based on the demineralization of all new water entering a nuclear installation, and recycle and reuse of used water that has been completely decontaminated and demineralized. A micro pilot-plant system was constructed which includes (1) continuous clarification using zeta-potential-controlled additions of coagulant (alum) and coagulant aid (activated silica) and (2) continuous demineralization by cation-anion exchange followed by sorption on activated carbon. Flowsheet variations are being tested while a large inventory of demineralized and decontaminated water is accumulated from the treatment of ORNL low-level

radioactive waste. This will be used later to demonstrate the recycle portion of the flowsheet.

Overall decontamination factors (DF's) ranged from 100 to 1000 for ^{144}Ce, ^{131}I, ^{106}Ru, ^{125}Sb, and ^{95}Zr-Nb; from 1000 to 10,000 for ^{137}Cs and ^{90}Sr; and above 10,000 for ^{60}Co and total rare earths. Abnormally low pH (4.9 vs 7 to 8) of the waste prevented proper flocculation in the clarifier and thereby decreased the DF for ^{60}Co by a factor of 35. Use of a second column of activated carbon after the cation column and before the anion column (solution pH = 2.5 to 3.0) also decreased the DF for ^{60}Co, presumably by changing the ionic species present.

These results demonstrate the operability of the recycle approach but indicate the necessity of maintaining the pH in the range 7 to 8 for both flocculation and activated-carbon steps.

High-Level Radioactive Waste Calcination. — Work on the solidification of high-level waste in glass and ceramics was continued with the development of melt compositions for a newly defined high-sulfate waste, designated Purex 2. Solutions simulating this waste with fission products resulting from a fuel burnup of 20,000 Mwd/metric ton were fixed in ceramic solids which contained up to 45 wt % waste oxides. The process was tested in both semi-engineering-scale and full engineering-scale equipment. Operating temperatures were between 800 and 850°C. The volume reduction (volume of liquid waste/volume of solid) was about 7. Corrosion of the melting-storage vessel ("pot") was small to moderate.

These tests indicate that the compositions developed can be used in a practical fixation process but that further work is desirable to decrease container corrosion. Also, more work is needed to define and solve the problems resulting from mixing fluxing agents with the waste by proportional feeding to the pot instead of in a tank prior to feeding.

Disposal in Natural Salt Formations. — On November 17–19, 1965, the seven cans containing about 10^6 curies of fission products (14 irradiated ETR fuel assemblies) were placed in the Lyons, Kansas, mine. The maximum radiation dosage received by personnel involved in the transfers was about 200 millirads. Radiation levels immediately above the array holes are generally less than 10 mr/hr, and unlimited access is allowable everywhere except immediately over the holes.

Both salt temperature rises and fuel element temperatures are in reasonable agreement with theoretical calculations, though generally running somewhat below the predictions. Thermal expansion of the floor had reached about 1/2 in. in the center of the arrays at the end of the second week. Thermal stresses (but not yet temperature rises) have also been transmitted to the supporting pillars, as indicated by the increased flow rates of the salt in the pillars and in the roof. There have been no indications of any differences in behavior between the radioactive and control arrays, and no indications of chlorine release.

Safety Evaluation. — Low-level contaminated waste water, generated by the operations at ORNL, has been released to the Clinch River near Oak Ridge for the past 20 years, under controls consistent with the recommendations of the ICRP and the NCRP. Evaluation of radiation exposure doses to populations downstream, on the basis of long-term monitoring data and the identification of critical exposure pathways and critical

population groups, has been one of the principal objectives of the recently concluded, comprehensive Clinch River study.

By selecting reasonable values for periods of occupancy and dietary habits, an estimate was made of the cumulative dose (20-year period) received by the skeleton and total body of males working and residing in the Clinch River—Tennessee River environment (see table below). The fractions of maximum permissible dose received by the thyroid and GI tract were smaller than that received by the total body and were not included. Since the Clinch River does not serve as a source of municipal water, children do not consume water taken directly from this river. Therefore it was assumed that the youngest age group at the beginning of exposure was the group of 18-year-olds employed at the Oak Ridge Gaseous Diffusion Plant. Tennessee River water does serve as a municipal water supply, and it was found that the 14-year-olds constituted the critical population group.

Consumption of contaminated water and fish was the most important exposure pathway, and ^{90}Sr was responsible for more than 99% of the skeleton and total body dose. The estimated cumulative dose to the skeleton of the 18-year-old male utilizing the Clinch River was largest, at 3.2 rems. The estimated dose to the skeleton of the 14-year-old residing along the Tennessee River is 0.45 rem. In both cases the dose estimate is less than about one-twentieth of the maximum permissible dose. These estimated doses are believed to be high as a result of the conservative assumptions made in their estimation.

Estimated Cumulative Dose Received by Critical Organs of Males from Use of Clinch River and Tennessee River[a]
(rems)

Critical Pathway	Clinch River		Tennessee River	
	Skeleton	Total Body	Skeleton	Total Body
Drinking water	1.4	0.11	0.38	0.030
Recreation	0.018	0.019	0.003	0.003
Fish	1.8	0.14	0.070	0.0057
Total	3.2	0.27	0.45	0.039
Maximum permissible dose[b]	60	10	20	1.0

[a]Aggregate exposure for the period 1944 to 1963.

[b]As recommended by ICRP, the annual dose rates for continuous occupational exposure are reduced to 1/10 and applied to the Clinch River and are reduced to 1/30 for bone as critical organ and to 1/100 for total body as critical organ and applied to the Tennessee River.

[redacted] [redacted]

PHYSICAL RESEARCH

PHYSICS AND MATHEMATICS

Medium-Energy Nuclear Physics: Experimental. — The differential cross section for elastic and inelastic scattering of 40-Mev protons was measured for ^{28}Si, ^{58}Ni, ^{60}Ni, and ^{54}Fe. These data supplement the polarization data obtained earlier. Analyses of inelastic-scattering polarization indicate that distorted real and imaginary potentials are not sufficient to explain the data. Work is now in progress which will include a distorted spin orbit potential in the calculations. Preparations are in progress to measure the energy spectrum of tritons from the reaction $^{3}He + d \rightarrow t + 2p$ with the broad range spectrograph at 0° to the incident beam.

The reaction $^{16}O(d,^{3}He)^{15}N$ was studied; in addition to the ground and third excited states, the unresolved 5.28- and 5.31-Mev doublet was observed. Observation of this doublet implies that two-particle, two-hole configurations are present in the ^{16}O ground-state wave function, as indicated by G. Brown and A. E. S. Green (unpublished). The general shapes of the first and third excited states are reproduced by distorted-wave calculations, and the doublet shows evidence of containing a summation of both 1d and 2s components. Some difficulties were encountered in using the same ^{3}He optical-model parameters for the ground and third excited states; this is most probably due to the low atomic number of the target. To obtain better agreement between theory and experiment for these states, it is necessary to interpolate between ^{3}He elastic-scattering parameters, and to choose the values corresponding to the exit channel energies. Difficulty is also encountered in attempting to extract reliable spectroscopic information for the two-particle, two-hole states, since wave functions, and thus the binding energies, for these states are not known.

Sources of ^{157}Eu, produced by the $^{160}Gd(p,\alpha)$ reaction, were analyzed with conversion electron spectrographs. The 50 transitions observed in the spectra include confirmation of the 9.4-kev (E1) radiation connecting 64-kev ($5/2^{+}$) and 54-kev ($5/2^{-}$) states in ^{157}Gd. A $5/2^{-}$ [523] assignment for the excited level at 434 kev is supported by the relative intensity of the E1 de-excitation to members of the $5/2^{+}$ [642] rotational band. A 474-kev state is depopulated by intense dipoles to single-particle states at 64 kev ($5/2^{+}$) and at ground ($3/2^{-}$). Since no radiation to the low-lying rotational structures was detected, the 474-kev state is designated as the $3/2^{+}$ [651] configuration. Excited states at 686- and at 683-kev energies are classified as even parity on the basis of M1 feeding to the 64-kev ($5/2^{+}$) level. A number of weakly populated states are proposed at energies of 449, 630, 811, and 1137 kev.

Medium-Energy Nuclear Physics: Theoretical. — In many nuclear shell-model calculations in the past, a conventional type of the phenomenological two-body interaction has been used, which is everywhere attractive and of moderate strength. In most of the cases the nuclear spectra are calculated by ordinary perturbation theory. Recently, some efforts have been made to calculate the nuclear spectra from the first principle, using the free nucleon-nucleon potential.

[redacted] [redacted]

The first difficulty encountered in such a calculation is that the free nucleon-nucleon potential is known to have very strong short-range correlations, invalidating the ordinary perturbation theory. This difficulty can be avoided, however, if one defines an effective operator, which takes into account the short-range correlation effectively. Such an effective operator can be regarded as the effective interaction to be diagonalized in a shell-model calculation.

The short-range correlations can be taken into account by calculating the reaction matrix from the free nucleon-nucleon potentials in the Brueckner approximation to obtain the effective interaction. The reaction matrix was calculated for light nuclei from the Brueckner-Gammel-Thaler and the Hamada-Johnston potentials by solving a Schroedinger equation after neglecting the Pauli principle and other many-body effects. For both potentials the calculated reaction matrix compares well with other results which do take into account the Pauli principle and other effects approximately; this indicates that these effects are small for light nuclei. Calculations of the nuclear spectra using the reaction matrix as an effective interaction are in progress.

Electronuclear Systems Development. — The ORIC beam extraction system was modified by the installation of a new power supply for the inside coil of the iron channel and the installation of a remote-control mechanism for optimizing the position of the channel. This has resulted in greatly improved operation of the cyclotron; extraction efficiencies of 60% are now obtained for most particles. Also, a wider range of energies can be extracted because of the greater flexibility of the system.

Compressing the dee so that the dee aperture is reduced about 0.5 in. has caused the limit of available radio frequency to be transferred from the resonator to the power amplifier system. The system will now operate at up to 22.4 megahertz; the former limit was 21.9 megahertz. This increased the energy of the extracted proton beam up to 64.5 Mev.

A new device for measuring the time width of the extracted beam was built and tested. The system uses a constant-impedance 50-ohm Faraday cup connected directly to a sampling oscilloscope. The results of measurements with this device confirm earlier measurements and indicate a beam phase width of 20° full width and 10° full width at half maximum.

An amplifier for the spinning-wire beam scanners was built to improve the operation and to increase the range of usefulness of these scanners. The amplifier is expected to allow beams as low as 10 nanoamperes to be observed; the former threshold was 1 μa.

Advanced Accelerator Studies. — In a separated-orbit cyclotron, random errors in construction and alignment of the pole tips and in adjustment of the magnetic field lead to an uncertainty in the position of the beam and, thus, affect the specification of the aperture of the beam pipe. The uncertainty in beam location is proportional to the field gradient and to the amplitude of the envelope of betatron oscillations. For axial motion, it is found that reducing the gradient at low energies does not affect beam position uncertainty for a given set of tolerances. For radial motion in this machine, however, reducing the gradient reduces both the betatron oscillation amplitude and the beam position

uncertainty due to errors. This advantage is partially canceled by the increased radial oscillations caused by coupling to the phase-energy oscillations. For a net saving in radial aperture and for simplified construction, we are designing a machine with the gradient the same in all sectors. For the 12-sector, 11-turn, 10- to 50-Mev machine under development, the aperture is about 1.5 × 3.0 in. for an FDO focusing structure having a gradient of 0.8 kilogauss/in. A set of 11 pole tips for one magnet sector of this cyclotron is being machined according to this plan.

Isochronous Cyclotron Operations. — The ORIC was scheduled for a total of 1128 hr during the past two-month period. The distribution of the time was as follows: 57% to nuclear research bombardments; 12% to ORIC beam and machine development; 13% to installation of new equipment; 3% to scheduled shutdowns for routine maintenance; 9% to component failure; and the remaining 6% to downtime due to miscellaneous operation outages, such as rf and vacuum troubles.

Extracted beams of alpha particles at 68 and 80 Mev, deuterons at 34 Mev, $^{3}He^{2+}$ at 50 Mev, $^{3}He^{+}$ at 25 Mev, and protons at from 25 to 63 Mev were provided for the experimenters. Beam "on target" time averaged ~48% of the total 1128 hr.

Physics of Fission. — A multicomponent ^{127}I beam (30 to 80 Mev) from the tandem accelerator was used for continued studies of channeling in gold single crystals (approximately 2500 A thick). A new arrangement involving time-of-flight techniques was used to obtain improved energy and angular resolution. Detailed angular scans were made across planar channels. At the center of the channels the characteristic normal energy loss was almost completely replaced by a lower energy loss characteristic of the plane. Slightly off planar-channel directions (about 0.2°), additional sharp groups with intermediate energy loss were found. Characteristic energy losses for several planar directions were obtained. With the high angular resolution used, no difference was observed between the (111) planar energy loss and the [110] or the [112] ("pipe") energy losses.

High-Voltage Experimental Program: Lack of Structure in ^{235}U Fission Cross Section in the Neutron Range 150 to 335 kev. — The ratio of ^{235}U fission cross section to the $^{10}B(n,\alpha\gamma)$ cross section in the energy range 150 to 335 kev has been determined using the 3-Mv pulsed, bunched Van de Graaff accelerator. This ratio was reduced to the relative fission cross section by using a boron cross section obtained from a combination of the $^{10}B(n,\alpha_0)^{7}Li$ cross section (obtained by reciprocity from the inverse reaction) and the $^{10}B(n,\alpha\gamma)/^{10}B(n,\alpha_0)$ ratio. The results show no tendency to deviate from a smooth monotonic decreasing fission cross section as a function of energy. This result is in contrast with recent results of Albert which show strong (about 30%) fluctuations in this energy region.

High-Voltage Experimental Program: Elastic and Inelastic Proton Scattering from ^{106}Pd and ^{108}Pd. — Angular distributions of elastically and inelastically scattered 12- and 13-Mev protons from ^{106}Pd and ^{108}Pd have been measured for angles between 24 and 165°. Quadrupole and

octupole deformation parameters, β_2 and β_3, were extracted from the experimental cross sections by means of the distorted-wave direct-interaction theory. They are: $\beta_2 = 0.25$ and $\beta_3 = 0.15$ for ^{106}Pd, and $\beta_2 = 0.23$ and $\beta_3 = 0.14$ for ^{108}Pd. These are consistent with values obtained through Coulomb excitation studies. The octupole states were observed at 2.07 Mev in ^{106}Pd and at 2.03 Mev in ^{108}Pd. Differential cross sections of the two quadrupole phonon states have been compared with the predictions of the coupled channels theory as extended by Tamura. Best agreement between experiment and theory for excitation of the second 2^+ state was achieved when about 5% of the one-phonon state was admixed with the two-phonon state. Energies and cross sections at one angle for a number of other levels below 3.2 Mev were also measured.

High-Resolution Electron Microscopy. — A survey has been made of some possible methods for producing the thin film required for the proposed film lens. It is important to recognize that the very thin film (allowing essentially complete electron transmission) is required to span an aperture only about 1 mil in diameter. Beyond this diameter the film can be smoothly brought up to a mechanically usable thickness of roughly 1 mil in a transition distance of about 10 mils. We thereby enormously reduce the magnitude of the film problem. We plan to explore several schemes which utilize electropolishing techniques to produce small, smooth walled pits in thin metal stock. The polishing process can be finely controlled by monitoring the optical transmission of the film, to bring the center of the pit to the desired thickness. Central thicknesses of about 50 A for aluminum or 150 A for beryllium should be permissible. Another related technique involves pyrolytic deposition of a graphite film on one side of a stainless steel foil about 1 mil thick. This foil can then be electropolished until a hole appears in the stainless steel about 1 mil in diameter, choosing conditions so that the graphite is relatively untouched. The required electropolishing and monitoring equipment is being planned. Further planning is being done to facilitate a reasonably complete test of the film lens idea. It is hoped that a realistic test can be made in a conventional microscope, by using a commercial image intensifier to sharply reduce the contamination problem, and by rebuilding a single section of the microscope column to incorporate the film lens in place of the conventional objective.

CHEMISTRY

Gas Transport Phenomena. — Previous theoretical treatments of gas transport through porous media, in which the dusty-gas model had been employed, were noticeably deficient in the description of flow as the result of gradients in pressure. This shortcoming has recently been removed by the realization that the strict separation of viscous and diffusive fluxes is theoretically justified; that is, the two modes of transport may be (mentally) treated independently of one another; the only "cross-term" of consequence can be included in the pressure diffusion coefficient.

The most fruitful aspect of this realization to date has been the development of an expression for diffusion under a pressure gradient

completely on a theoretical basis, with an explicit expression for what had previously been an empirical coefficient. In addition, the new results lead directly to the prediction of the Kramers-Kistemaker effect, wherein a pressure drop is generated within a diffusing gas mixture when the net molecular flux is zero.

Analysis of Molten Salts by Electrochemical Methods: Voltammetric Measurements in Molten $LiF-BeF_2$. – Controlled-potential electrolysis experiments have been continued on the molten $LiF-BeF_2$. It is now believed that the anodic wave observed in the molten $LiF-BeF_2$ at approximately +1.5 v is an oxide wave ($O^{2-} - 2e \rightarrow 1/2\ O_2$), and recent attention has been focused on controlled-potential electrolysis as a possible means of determining oxide or at least purifying the melt with respect to this constituent. The major difficulties encountered on electrolyzing the melt so far have been the shifting of the potential of the platinum quasi-reference electrode during the electrolysis experiment, low current efficiencies, and the necessity of conducting the electrolysis under reduced pressure in order to draw appreciable cell current (5 to 10 ma) at a stationary indicator electrode.

In an effort to achieve a more stable reference potential for the electrolysis, a melt of $LiF-BeF_2$ was prepared in which the porous graphite inner compartment of the graphite cell was filled with a mixture of $LiF-BeF_2$ and NiO so as to form a saturated solution with respect to nickel oxide. Into this compartment we have placed a nickel electrode. The platinum counter electrode, which was previously located in the porous graphite compartment, is now placed in the main portion of the melt along with the indicator electrodes.

If the anode is vibrated during electrolysis, cell currents comparable to or larger than those obtained under reduced pressure can be realized without evacuating the cell. A vibrating electrode was prepared by coupling the stem of the electrode (1/8-in. nickel rod) to a small 60-cps electric buzzer. Flexibility of the electrode was achieved by means of a nickel metal bellows joint. With this arrangement it is planned to electrolyze the melt in a protective atmosphere of helium and allow the gaseous products from the electrode reaction to be swept from the cell in the flowing helium stream. A trap to isolate the products from the helium stream was designed and fabricated.

For this experiment the vibrating pyrolytic graphite anode was utilized. This electrode was coupled with a platinum counter electrode and a nickel–nickel oxide reference. Two indicator electrodes, a platinum and a platinum–10% rhodium, were utilized to record current-voltage curves of the oxide wave at approximately +1.5 v. The controlled-potential electrolysis was carried out in a helium atmosphere (~2 lb overpressure), and the gaseous products were swept at a flow rate of ~80 ml/min into a 13X Molecular Sieve trap maintained at the temperature of liquid nitrogen. Unfortunately, the trap developed a leak at the end of the experiment, and it was therefore not possible to obtain a gas chromatographic analysis of the equivalents of oxygen collected, from which it was hoped to gain some idea concerning the current efficiency of the electrode process.

Of importance, however, is the fact that the wave height of the anodic wave did decrease this time after the electrolysis period. For the passage of 456 coulombs of electricity through the melt (average

electrolysis current, 5 to 10 ma), the height of the "oxide" wave decreased from ~2000 μa to 600 μa at the platinum–10% rhodium indicator electrode and from ~2000 μa to 1060 μa at the platinum microelectrode. This is the first time that a definite decrease in wave height has been observed following controlled-potential electrolysis. From these observations it appears that both electrodes are useful indicator electrodes; however, concentration vs limiting current exhibits greater linearity at the platinum electrode. Additional work is required, therefore, to evaluate more fully the two electrode materials for anodic indicator electrodes.

In a prior controlled-potential electrolysis experiment the oxide wave did not change appreciably after the electrolysis. It was believed at one time that a nickel oxide may be diffusing into the melt through the porous graphite inner compartment of the cell. Although this may be happening to some extent, it is now believed that the melt might have been contaminated by a trace of moisture in the helium stream, because helium was bubbled through the melt in the former experiment; whereas for this run the helium flowed over the surface of the melt only.

To check this out, the moisture content of the helium stream is to be determined with a moisture monitor, and necessary precautions are to be taken to assure a low moisture content of the helium as well as the service lines. It is our intention to continue the controlled-potential electrolysis experiments.

Properties of Aqueous Solutions: Species and Thermodynamic Properties. — The isopiestic apparatus, constructed primarily to measure activity coefficients of multicomponent solutions, is complete. Our initial objective is to establish in representative cases the best procedures for computation of free energies of multicomponent systems from free energies of the limiting two-component systems. Although we are particularly interested in perchlorate salts, because of their extensive use in the study of complexing reactions, the first systems undertaken have the solute composition of seawater and its concentrates. We selected these solutions because practical implications have led to a recent computation by Lietzke and Stoughton of their thermodynamic properties from NaCl data.

In effect they obtained a semiempirical equation for osmotic coefficients, Φ_{NaCl}, of NaCl in terms of a Debye-Hückel term and a power series in molality $\underline{m}$; their equations also covered a range of temperature. They then tried to compute seawater osmotic coefficients, Φ_{SW}, by inserting into the NaCl equation seawater concentrations expressed in different ways. From comparison of boiling-point elevations of seawater computed in this way with what they took to be the best values in the literature, they concluded that the best seawater osmotic coefficients were obtained by expressing the concentration as "osmolality," $\frac{1}{2}\sum_i m_i$, the summation being over all ionic species. There is, however, considerable disagreement between values of seawater boiling-point elevations measured in different laboratories, and a direct experimental determination of Φ_{SW} seemed desirable.

We have made measurements of isopiestic ratios at 25°C of NaCl and seawater for seawater ionic strengths between 0.5 and 7. Although the results are still preliminary, it seems clear that use of ionic strength $(\sum_i \frac{1}{2} m_i z_i^2)$ of seawater in the power-series concentration terms of the

equation for Φ_{NaCl} leads to much better agreement with experiment than use of osmolality.

Chemical Physics: Molecular Beam Studies. — A computer program has been written which may be useful in the interpretation (in terms of classical mechanics) of angular distributions measured in experiments on molecular beam scattering. The program is written in two parts: the first part is used to adjust the minimum value of the interatomic potential to any desired equilibrium internuclear distance by adjusting the strength of the repulsive portion of the potential, while the second part computes the classical deflection function, the corresponding phase shift, and the angular spacing between the principal rainbow and the second-order rainbow.

TRANSURANIUM-ELEMENT PROCESSING

Process Development: Irradiated Plutonium Dissolution. — Dissolution experiments with irradiated HFIR prototype targets have continued with segments of prototype target TRU-3 (nearly 62% of the plutonium fissioned). Results were intermediate between those for TRU-1 (where nearly 32% of the plutonium fissioned) and TRU-2 (where about 64% of the plutonium had fissioned) target elements. All samples had been dissolved in each of the following three: HCl, HNO_3, and HNO_3-HF. Three molar HCl, which dissolved 63 to 65% of the plutonium in TRU-1 targets and 99.85% of that in TRU-2 targets, dissolved 87.2% of the TRU-3 target in 5 hr at reflux; an additional 3.28% dissolved after 10 hr of refluxing in 6 M HCl. The residue was dissolved by refluxing for 5 hr in 6 M HCl—0.1 M CuF_2. Concentrated HNO_3 dissolved 68.0% of the plutonium in a second TRU-3 target segment after the aluminum matrix was first dissolved with 1.2 M NaOH—1.5 M $NaNO_3$. Nearly all this dissolution occurred in the first half hour. A comparable set of conditions resulted in dissolving 45% of the plutonium in a TRU-1 target. A third TRU-3 target dissolution was made with concentrated HNO_3—0.1 M HF, and 99.5% of the plutonium dissolved within 1 hr at reflux after the aluminum matrix was first dissolved in 1.2 M NaOH—1.5 M $NaNO_3$. This is comparable to the 99.95% dissolution achieved with TRU-1 and TRU-2 targets.

Process Development: ^{243}Am-^{244}Cm Purification. — Sixteen grams of ^{244}Cm and about 17 g of ^{243}Am, recovered by cell 4 solvent extraction processing, were further purified by LiCl anion exchange. The product solution contained no detectable rare-earth fission products, and ionic impurities amounted to less than 1 wt %. The material is in storage, and the ^{243}Am and ^{244}Cm will be separated soon.

Process Development: Analyses and Processing of Savannah River HFIR Prototype Rod. — The prototype HFIR target rod, which had been irradiated in the Savannah River High Flux Reactor for more than a year, was dissolved and processed to recover the transplutonium elements. Before irradiation the rod contained 10 g of plutonium, of which 95.7% was ^{242}Pu. The target received an estimated time-integrated neutron flux of 3.5×10^{22}. After the aluminum matrix was dissolved with NaOH-$NaNO_3$, plutonium was recovered by HCl anion exchange, and the transplutonium elements were recovered and separated from rare-earth fission products by LiCl anion exchange. Preliminary results indicate that the rod contained 5.6 g of Pu, 1.5 g of

Am, 2.6 g of Cm, and 5 μg of Cf. An intensive study is under way here and at Savannah River to gain an understanding of the low production of californium, which had been expected to be in the 45- to 60-μg range.

TRU Operations. — The operating group, consisting of 10 technical and 20 nontechnical people, moved into the TRU facility and began prestartup testing of the facility and the equipment. Calibrations of chemical-makeup-area equipment, and repairs to numerous leaks found in this piping, have been mostly completed. Damage to roughing filters in the cell-ventilation system was noted. More-rugged filters were installed, and the system was given the standard dioctyl phosphate efficiency tests. All four units passed after one leaking flange was repaired.

Transuranium Processing Plant Design and Construction. — ORNL craft forces began final installation of in-cell equipment and piping on November 1. This work will be completed by January 1, except for the target-fabrication equipment and the shielding windows. All underground piping, footings, and grade beams have been completed for the office addition, and service lines have been run from the existing equipment room to the new addition. AEC approval to proceed with the emergency power system revisions was granted.

Vibration problems with the centrifuge were solved by installing an 8-in. rigid spindle between the motor and the bowl. The electrical drive and control system for the centrifuge was received and is being installed.

HFIR Target Fabrication Development. — Testing of the remote target-fabrication equipment has continued. The status of the testing, by cubicles, follows:

In cubicle 3 the galling of the shaft of the pellet magazine was solved by nickel-plating the shaft. We still have a problem with the magazine, however, in that the pellets cock and jam the rotation of the magazine in the discharge position. This condition can probably be corrected either by restricting the cocking of the pellets or by vibrating the magazine during rotation. Operation of the blending station indicates that some additional refinements are required in the vibrator trough to contain the powder, and that a motorized device for opening the blending container is required. All other equipment appears to be satisfactory. Considerable time was spent in setting up the die-and-batch scales to operate with the new radiation-resistant transducers.

In cubicle 2, problem areas in the operation of equipment appear to be few. The welder appears to be satisfactory; a number of welds were made using the remote setup of the assembly-machine transfer arm. The new transducer for the void-measurement station has been received and is being installed.

In cubicle 1, mockup operation has indicated problems that arise from crowding. For example, the manipulators cannot reach all the positive horizontal stops on the transfer arm; a special tool is being designed to overcome this problem. The transfer arm interferes with the cell crane track at the dimensional inspection station, electrical vertical stops on the transfer arm are not positive, and differences in equipment elevations are causing trouble in placing the target rod in some stations. None of these problems is insurmountable. The dimensional inspection unit has given undue trouble and is being modified.

A 14-min movie of the mockup was prepared.

METALLURGY AND MATERIALS

Deformation of Crystalline Solids. – Previously (ORNL-3835, p. 27), some preliminary results on the work-hardening characteristics of tantalum and a tantalum-niobium alloy were discussed. This investigation was continued with a study of other variables that affect the work-hardening characteristics.

A Ta–9.9 at. % W alloy showed a parabolic stress-strain curve, unlike the tantalum and tantalum-niobium, which had a comparable concentration of interstitial impurities. An alloy of Ta–9 at. % W of lower interstitial concentration also had a parabolic stress-strain curve. The interstitial concentration has a large effect on the type of stress-strain curve obtained; for example, doubling the interstitial content completely eliminates the three stages of work hardening in tantalum.

The extent of secondary slip in all cases was limited, resulting in an overshoot of 20 to 30°; however, some secondary slip is necessary for the existence of stage II. This large degree of overshoot can be explained in terms of dislocation dynamics. The density of mobile dislocations on the secondary slip system is less than that on the primary. For the secondary slip system to have the same strain rate (i.e., the same strain) as the primary, the resolved shear stress has to be larger. This difference in stress is related to both the difference in the density of mobile dislocations and the ratio of the temperature (T) to the activation volume (v). However, T/v goes to zero at higher temperatures.

Previously, we explained the temperature dependence of the work hardening (i.e., the existence of stage II) by the conclusion that there are more edge and mixed dislocations at the lower effective stresses (higher temperatures), and these would react with screw dislocations to form sessile dislocations, which would be the nuclei of tangles. Theoretical work by Y. T. Chou [J. Appl. Phys. 36, 1435 (1965)] on the anisotropy of the line energy of dislocations has shown that a screw-screw dislocation reaction is unstable and that the most stable reaction is between two edge dislocations. Therefore the two conditions for the existence of stage II are met at higher temperatures.

Examination of electron transmission micrographs from sections parallel and perpendicular to the primary slip plane shows that profuse tangling occurs only when the sample has been deformed into stage II.

Reactions at Metal Surfaces. – Our previous work on the corrosion of aluminum alloys in water indicated that the presence of a second phase in the alloy might have a beneficial effect on its corrosion properties. To investigate this phenomenon more thoroughly, we exposed a series of aluminum-zinc alloys to water at 200°C and 170 psi. The second phase in the aluminum-zinc system appears at approximately 13% Zn at 200°C, and the corrosion rate definitely decreased as the zinc concentration was increased from 13% to 15%. However, the greatest relative improvement in corrosion properties occurred between alloys containing 2 and 5% Zn, that is, for single-phase alloys. This improvement is no doubt related to the fact that 5% Zn was adequate to prevent the severe intergranular attack suffered by pure aluminum in high-temperature water. Thus the presence of a second phase is important in reducing the corrosion rate

of aluminum-zinc alloys, but other factors (i.e., the formation of a protective $ZnAlO_4$ film) must also be considered.

Theoretical Research. – A collaborative effort on the crystal field theory of p electrons has been finished. Theoretical studies of electronic states in disordered solids and of magnetism are continuing. Efforts have been made to predict molecular configurations theoretically.

Spectroscopy of Ionic Media. – We have prepared a bismuth impurity center that contains five bismuth atoms with a total oxidation number of +3, that is, $(Bi_5)^{3+}$. This center was prepared by a reaction between bismuth metal and $BiCl_3$ in a molten mixture of $ZnCl_2$ and KCl as solvent. The center has an intense absorption band at 390 mμ with a molar extinction coefficient of about 6.5×10^3 and two much weaker bands at 770 and 875 mμ. Additional weak bands were observed between 390 and 770 mμ, but these may be due to other bismuth centers. It appears likely that this center involves direct bismuth-bismuth bonds in which pπ electrons play a role of importance.

Fundamental Research in X-Ray Diffraction. – Our optimization of the various parameters associated with the counting of x rays with a proportional counter, preamplifier, and amplifier has been concluded. The theoretical limit of resolution of the counter has been achieved, and we have succeeded in eliminating deleterious space-charge effects.

We have continued assembly of the low-angle x-ray scattering apparatus with which the optimized counting system will be used. A cryostat for work at liquid-nitrogen temperatures has been designed, and a single-axis step-scanner was purchased. A theoretical method for correction of collimation effects in small-angle scattering experiments has been developed and will be incorporated in a computer program for processing data from these experiments.

Statistical analysis of the new x-ray diffraction data from a crystal of yttrium beryllium oxide has been completed, and Patterson functions were synthesized. From these functions we determined the 8 yttrium atom positions in the unit cell; then we located 16 oxygen and 4 beryllium atoms by Fourier methods. Least-squares refinement of the trial structure will be pursued with particular attention to the possible relaxation of the restraint of the mirror symmetry of the crystal's apparent space group (Pmcn).

We have developed a description of the anomalous transmission of x rays (the Borrmann effect) in terms of the Darwin formulation of the dynamical theory of x-ray diffraction rather than the Ewald–von Laue formulation. Our development predicts all observed features of the Borrmann effect in terms that are perhaps more physically tractable than those encountered in the alternate Ewald–von Laue approach.

Transition Metals and Alloys. – We found the resistivity of zirconium single crystals at low temperatures in transverse magnetic fields to be nearly isotropic for different field and current directions. For every direction investigated the resistivity varied nearly quadratically with the magnetic field strength. This would indicate that zirconium has a closed and compensated Fermi surface with equal numbers of holes and electrons.

The low-temperature electronic specific heat of scandium-zirconium alloys decreased rapidly from a high value for scandium to a low minimum for alloys containing large amounts of zirconium. Since the Pauli susceptibility decreases with temperature in scandium and increases with temperature in zirconium, the Fermi surface of scandium is near a maximum and that of zirconium is near a minimum in the density of electronic states. The results for zirconium may be compared qualitatively with energy—wave number curves calculated by Mattheiss [Phys. Rev. 134, A970 (1964)] for a similar metal, titanium. An electron band structure with slightly overlapping bands in zirconium, similar to that calculated for titanium, would agree well with our results.

Preparation of Pure Materials. — One of the most effective and simple means currently employed for removing traces of impurities from materials which are stable in the molten state is zone-melting purification. The efficiency of separation is governed by the distribution coefficient, k, the ratio of solid and liquid solubilities of the impurity, and the host solvent. We have determined that k for the lanthanide trifluorides, CeF_3, GdF_3, and LuF_3, in LiF is 0.3 to 0.5, which indicates that the rare-earth trifluorides can be effectively removed from LiF by zone melting. We have now begun to examine the change in LiF-based solvents on increasing the number of components in the solvent. In recent experiments we have found that the distribution coefficient of SmF_3 in $LiF-BeF_2-ZrF_4$ mixtures of compositions near that of $2LiF \cdot BeF_2$ is 0.9. A value of unity precludes separation. It appears therefore that effective removal of the lanthanide trifluorides from fluoride mixtures of the types currently employed in reactor development will require process control which involves multiple-pass melting by narrow molten zones.

X-Ray Topograph Examination of Vacancy Clusters in Nearly Perfect Copper Crystals. — Vacancies present at temperatures near the melting point in nearly perfect 99.999% copper crystals with dimensions of the order of centimeters cluster upon cooling the crystal, and the clusters are visible as "black spots" in Borrmann x-ray topographs. There is phenomenological evidence that the black spots correspond to some form of vacancy cluster. The diffraction contrast of the black-spot images has been examined in a series of 111, 220, 200, and 222 topographs taken with Ag, Mo, Cu, and Cr $K\alpha_1$ radiation. Some of the images show the proper contrast for prismatic dislocation loops. Many of the images appear in all topographs. Images of defects that intersect the exit surface for the x rays show a black-white contrast which can be interpreted in terms of vacancy loops. It is concluded that the evidence from diffraction contrast supports the assumption that the black spots correspond to vacancy clusters of various forms.

Thermal Migration of V_K Centers in KI. — This work was performed to supplement previous studies on the thermal reorientation of V_K centers, where it was shown that V_K centers reorient through an angle of 60° only. Jumps to first and second nearest neighbor (nn) positions are characterized by changes in orientation of 60 and 90° respectively. Therefore, the activation energy for reorientation (0.273 ± 0.004 ev) is required for a first nn jump. A possible motion along the <110> directions of a crystal, which would involve fourth nn jumps, prompted further studies.

The long-range migration of V_K centers through the lattice results in their annihilation at electron excess centers. Two independent methods used for measuring the activation energy for migration yielded 0.28 ± 0.01 and 0.26 ± 0.01 ev. Since the activation energies for migration and first nn jumps are the same within experimental error, these results provide evidence for the hypothesis that V_K centers move by a random walk involving first nn jumps only.

Recombination Luminescence in NaI and RbI. — Previous studies have shown that several nonactivated alkali halides exhibit a luminescence at low temperatures which arises from the recombination of an electron with a V_K center. These studies have been extended to NaI and RbI, and it is concluded that this recombination process is responsible for the 295-mμ emission in NaI and the 318-mμ emission in RbI. This process is demonstrated experimentally by the observation of a linear polarization in the emitted light if V_K centers are preferentially oriented along a chosen [110] direction prior to the recombination event. In these experiments it was first necessary to identify V_K centers in these two crystals. This was achieved by observing the optical anisotropy induced in an irradiated crystal by bleaching with linearly polarized light in the 400-mμ region. The anisotropic absorption spectrum is very similar to that of the V_K center in KI. The V_K centers are stable in NaI for temperatures below about 58°K, and in RbI below about 125°K.

A Method for Calculating Absolute Frequency Distribution Functions in Solids. — A new method to calculate absolute phonon frequency distribution functions, which is an extension of the extrapolation method developed by Gilat and Dolling, involves dividing the irreducible section of the first Brillouin zone into a cubic mesh and approximating the constant-frequency surfaces inside every small cube by a set of parallel planes. This method proves to be of high precision and resolution in obtaining fine details associated with a given model, and it requires relatively short computing time. Applications have been made to nickel, aluminum, and sodium, for which there exist satisfactory force-constant models. New critical points have been found for aluminum at ν = 7.104 ± 0.006 terahertz and for sodium at ν = 2.72 ± 0.01 terahertz. Certain critical points associated with the longitudinal phonon band have been resolved more sharply than in earlier calculations.

CONTROLLED THERMONUCLEAR RESEARCH

The DCX-2 Injection-Accumulation Experiment. — Most of this report period has been spent in continued efforts to improve the vacuum system. This has resulted, finally, in a complete dismantling of the vacuum system and re-cleaning all of the individual parts. The liner was modified so that now it is possible to flow liquid nitrogen through its water paths, which should also improve the base pressure. The emphasis on better vacuum requirements has followed, among other reasons, from a detailed study of gas dissociation experiments previously carried out. The data show that the intensification of the microinstability results in an increase in the density of trapped protons in the midplane of DCX-2, as compared with the

situation when the microinstability is only weakly present. This is exhibited in a peak in the curve of density vs pressure as the pressure in DCX-2 is raised either with nitrogen or hydrogen. The cause of the increased density can be ascribed to an effectively longer containment time of the trapped particles resulting from a reduction in the effective charge exchange cross section because of the increased energy spread of the trapped particles. Improvements in the base pressure are required to enable us to better interpret the energy distributions observed in DCX-2 and should lead to a better understanding of the nonlinear behavior of the instabilities. The poorer vacuum conditions result in the emission of a large flux of nonhydrogenic gases from the walls into the liner as the plasma develops. Since these gases have cross sections for ionization and charge exchange which are very different from those of hydrogen, they tend to complicate the interpretation of the results. In fact, although mass spectrometric information of the residual gases is continuously monitored and the partial pressure of hydrogen is extracted from the data, this information is limited by shortcomings in the speed of the analyzer and the difficulties in interpretation of the observed mass spectrum. Further, the effects of the outgassing on the cold plasma cannot easily be determined. If the pressure can be reduced by our cleaning procedures, it is hoped that the contribution of these gases can be made negligible.

The response of the radio-frequency antennas has been calibrated, and we observe electric fields at the probes as large as 200 v/cm for some of the harmonics. The fields acting on the particles are even larger. The variation in the character of the instability as a function of pressure has been examined but cannot, as yet, be explained, since a proper model of the instability is still to be developed.

Active and Passive Stabilization of Plasmas and Ion Beams. — The accompanying table shows qualitative comparisons of observed operating conditions for a standard calutron using the Ca^+ beam passing through a homogeneous magnetic field. The reference row is for the best beam focus which occurs at a pressure of 4×10^{-6} torr and a minimum depth of the positive potential well at the 90° point of 25 v.

Pressure	Depth of Positive Potential Well	Beam Imperfection	Ion Loss Rate
Higher	Deeper	Larger	Larger
Reference	Reference	Reference	Reference
Lower	Deeper	Larger	Smaller

Ion loss rate is a sample of the ion drain measured by a Faraday cup at the 90° point. Beam imperfection is defined as the ratio of the total beam current to the beam current that enters the collector slit.

The disturbance which perturbs the beam and gives rise to a large beam imperfection appears to be associated with the depth of the positive potential well. The problem posed by this table can be stated in several ways; for example, if, as shown in the top row, the deeper potential well is required to produce the larger ion loss rate, then why is the deeper potential well required in the bottom row?

A computer program is being developed which will simulate various individual aspects of the beam plasma interaction in the calutron. The "foundation" potential distribution has been completed for a lune-shaped charge distribution for various wall configurations and plausible perturbations. Work is in progress on a program which allows electrons to move in the foundation potential well.

Properties of Vacuum Arcs. — Studies have continued on the excitation heating process (ORNL-3886, pp. 31–32), previously suggested as a mechanism for generating very energetic carbon ions in the presence of relatively low-energy electrons. Attempts to ignite the excitation heating mode in nitrogen-fed arcs having metal electrodes continued to be unsuccessful. Using boron nitride insulators placed appropriately inside a hollow metal anode having a tungsten backstop and inside a water-cooled, copper cathode shield insulated from a tantalum cathode, energetic ions of boron, carbon, and nitrogen were observed. Attempts are being made to reduce the carbon impurity which may dominate the excitation heating process. However, the arc exhibits the largest spectral line slants (due to Doppler shifts) of any energetic arc studied to date. The shift is counter-Larmor ($E \times B$), and the uncorrected Doppler temperatures ($T_\perp$ from line half-widths only) indicate a mass dependence (B^+: 4.0×10^6 °K; C^{2+}: 5.0×10^6 °K; N^{2+}: 5.9×10^6 °K).

Experimental Plasma Physics. — A simple decay technique for measuring the average plasma density in discharge tubes has been successfully tested. The technique works by sampling the flow of plasma to the walls of the container after the ionizing power supply has been turned off. The decay technique supplements Langmuir probes, which one can show to be unreliable in small quiescent plasma systems.

The very sensitive technique for detecting ionic sound waves by having them perturb an electron-carrying sheath around a probe (ORNL-3886, p. 32) has been experimentally shown to apply to cold electron-absorbing probes as well as to hot electron-emitting probes. One must be careful to keep the cold probe small in size and the applied positive potential low, or oscillations or a glow discharge, respectively, develops around the probe.

BIOLOGY AND MEDICINE

TERRESTRIAL AND FRESHWATER ECOLOGY

Aquatic Ecology: Feeding Rate of Carp in White Oak Lake. — The daily food consumption by fish in White Oak Lake can be estimated by measuring their turnover of ^{137}Cs. Previous reports have given an estimate of the daily ration for maintenance consumed by yearling carp. Separate estimates of each diet item were made using graphic analysis. Now, equations have been developed allowing separate calculation of the amount of each diet item consumed for maintenance metabolism and growth. The equation for the maintenance ration R, in grams per day, is

$$R = \frac{Q_e k}{a_1 c_1 f_1 + a_2 c_2 f_2 + \ldots + a_n c_n f_n},$$

where Q_e is the equilibrium body burden of the fish in micromicrocuries; k is the daily rate of biological elimination of ^{137}Cs by the fish (radioactive decay is negligible); f_1, f_2, and f_n are the fractions of the first, second, and nth items, respectively, in the diet; the c's are the concentrations of ^{137}Cs in micromicrocuries per gram in or on each corresponding diet item; and the a's are the fractions of the ingested radioisotope that are assimilated by the fish for the diet items. The equation for the growth ration is

$$R_g = \frac{0.5Q_e k + \dot{Q}}{a_1c_1f_1 + a_2c_2f_2 + \ldots + a_nc_nf_n},$$

where $\dot{Q}$ is the change in body burden or the increase in ^{137}Cs due to growth in micromicrocuries per day, and Q_e is the body burden of the new tissue in micromicrocuries. Since Q_e is accumulated throughout the day, the average value of $0.5Q_e$ is used for the elimination correction. The value of $\dot{Q}$ is determined by estimating the growth of the fish by standard fishery methods.

The total food consumed per day by an average yearling carp was 5.36 g wet weight. The maintenance ration included 2.4 g of detritus and 1.6 g of algae, while the ration for growth included 0.82 g of detritus and 0.54 g of algae.

Aquatic Ecology: Vertical Distribution of Radionuclides in Periphyton of White Oak Lake. — The role of attached plants and animals in radionuclide cycling in aquatic environments has not been assessed. White Oak Lake offers a unique opportunity for study of this aspect because of its contamination with various radioisotopes. During the summer of 1965 a pilot study was undertaken to determine the vertical distribution of radionuclides in periphyton developed in artificial substrates. A series of continuous substrates (polyethylene tape) were suspended from surface to bottom in the lake, and periphyton was allowed to grow on them. At periodic intervals tapes were removed and returned to the laboratory for analysis of the vertical distribution of organisms and radioactivity. To date only biomass data are available, together with visual observations of changes in the distribution of the principal plant and animal components. These results indicate that the populations do not stabilize after a two-month interval, but there is continuing change and redistribution on the tapes. The dynamics of the biomass are expected to determine the dynamics of the radionuclides.

Radionuclide Cycling in Terrestrial Ecosystems: Distribution of ^{137}Cs in Terrestrial Microcosms. — The effects of various combinations of microflora, millipedes, snails, and green plants on the rate of transfer of ^{137}Cs from decomposing leaf litter were evaluated in microcosms for a period of six months. The microcosms were leached with rainwater, and the ^{137}Cs concentrations in the components of the various combinations of compartments, as well as in mineral soil, soil organic matter, and leachate seeping through the soil, were measured monthly. After six months, sterile leaves had lost 40% of their ^{137}Cs. Of the initial ^{137}Cs, 28% had been retained by the soil and 10% had leached through the soil. In the presence of a leaf-decomposing microflora, the leaves lost 10% of

their weight and 50% of their ^{137}Cs; the microflora and mineral soil retained 36 and 10%, respectively, and only 3% leached through. When leaf-eating millipedes were added to this system, the leaves lost 43% of their weight and 66% of the initial ^{137}Cs. The microflora, which, to a large extent, was consumed by the millipedes only, retained 11% of the ^{137}Cs, and the millipedes retained 0.4%. As in the microcosm with the sterile leaves, the soil retained 28%, but added organic matter in the soil (feces, leaf fragments) retained 22%, and 6% leached through. The presence of snails had a similar but less pronounced effect than the presence of millipedes. The presence of green plants mainly increased the ^{137}Cs retained by litter (through the addition of new litter). Surviving plants retained an average of 1% of the ^{137}Cs. The strongest single effect on the leaching of ^{137}Cs from leaf litter came from the microflora. The main effects of millipedes, snails, and plants were significant ($P < 5\%$), as was the interaction between millipedes and snails. The microcosms provide an excellent tool for studying the movement and distribution of mineral elements under the influence of various biotic as well as abiotic compartments.

Radionuclide Cycling in Terrestrial Ecosystems: Radiocesium Turnover Rates for Terrestrial Isopods Under Natural and Laboratory Conditions. — Ecological studies dealing with the turnover rates and behavior of radionuclides in natural environmental systems are often dependent upon laboratory determinations of biological half-lives for prediction of subsequent isotope movement in the field. Many environmental parameters interact to affect the biological retention of radiocesium in free-living species. Paramount among these factors is the influence of temperature. Recent studies have demonstrated that ^{134}Cs retention in terrestrial isopods is a function of the environmental temperature t°C, following a Q_{10} relationship ($P < 0.01$) in which the elimination constant k is given by

$$\log k = -2.22 + 0.033t \ .$$

Predictions based upon this model for ^{134}Cs retention by field populations of isopods under known temperature regimes closely approximated observed turnover rates. *Armadillidium vulgare* exhibited a turnover rate of 2.4% per day and differed by a factor of only 0.1% from the expected rate. *Cylisticus convexus* had a turnover rate of 3.3% per day, which was 1.1% per day greater than that predicted. All of these values were derived from linear exponential loss rates due to bodily excretion.

Since isopods are poikilothermic, it was not unexpected that a similar Q_{10} relationship ($P < 0.01$) was demonstrated for metabolic rate and temperature. The number of microliters of oxygen consumed, A, is given by

$$\log A = 1.68 + 0.027t \ .$$

The solution to these two simultaneous equations gives an expression describing the direct relationship between the elimination constant for radiocesium and oxygen consumption:

$$k = 5.25 \times 10^{-5} A^{1.22} \ .$$

Although a power function, the equation is approximately linear over the normal range of environmental temperatures. Because such relationships exist between radiocesium turnover rates and species metabolism, radioactive tags can be used to measure the metabolism of field populations of arthropods. Radioisotope methodology thus appears to offer the ecologist a new and valuable tool for the study of nutrient flow and energetics in various components of terrestrial ecosystems.

Radionuclide Cycling: Punch Card Inventory of Oak Ridge Regional Flora. — Over 1500 plant taxa contribute to the genetic diversity of woodland and herbaceous ecosystems in Oak Ridge and surrounding Morgan, Roane, Anderson, Knox, and Loudon counties in eastern Tennessee. Records from the ORNL ecology herbarium, the University of Tennessee herbarium, county maps, and observations have been recorded on a punch card file, which is updated as additional information is obtained from (or for) the radiation ecology research program on the Oak Ridge reservation. An IBM 1401 computer program created punched paper tape, recording information in a form suitable for printing with a Friden Justowriter. A preliminary alphabetic list of results through late 1965 has been issued. Numerical codes for order and family in the plant kingdom are adapted from the International Plant Index, so that the data can be sorted in taxonomic categories when desired. The data-processing methodology may be of broader interest in compiling regional floras and international records like those desired for either intensive ecosystem analyses or worldwide inventories of vegetation types for conservation. Examples covering the composition of *Liriodendron tulipifera* forests and the distribution of ^{137}Cs in the species of a tagged plot near ORNL will be reported later.

Environmental Radiation Botany: Dose Contribution to Plant Roots by Soil Activation Products. — The fast-neutron dose delivered to potted plants during radiation-effects experiments at the HPRR can be accurately estimated, but the additional dose of gamma and beta radiation delivered to the plant roots by the activation of elements in the potting soil is unknown. A study now in progress is designed to estimate the dose delivered by this secondary source of radiation after reactor shutdown.

Emory silt loam, a soil commonly used for growing experimental plant material, was placed in polyethylene bags and molded into 11 × 11 cm plastic frames 3 cm thick to form a monolithic soil sandwich. Seven such sandwiches were arranged back to back to form a package 21 cm thick which simulates the tangential cross section of a planting pot. The density of the reconstructed soil averaged 1.57 g/cc, and the moisture content was 32.4% of the oven-dry weight. Gold and cadmium-covered-gold fission foils were inserted between the soil layers to measure the thermalization of the fast neutrons as they passed through the soil mass.

The experimental package was exposed at 1.0 m from the HPRR core to 2056 rads (tissue dose in air) of fast neutrons for a duration of 10 min. The thermal-neutron fluence reached a maximum of 10.4×10^9 neutrons/cm^2 at 6 cm from the exposed surface. The major activation products identified by spectral analysis were ^{56}Mn, ^{51}Cr, ^{86}Rb, ^{181}Hf, and ^{95}Zr-Nb in decreasing order of their relative contribution to the total gamma activity. Manganese-56 contributed over 96% of the measured activity, while ^{51}Cr and ^{86}Rb collectively made up 3% of the activity in the total soil mass. The distribution of radionuclides within the experimental package

followed the trend of the thermal-neutron flux, the highest activities being contained in the soil layer extending from 3 to 6 cm. Infinite-dose estimates are being calculated for the gamma and beta radiation resulting from activation of soil minerals. Because of the short half-lives and the small quantity (less than 0.5 μc/g) of the nuclides involved, the dose to the roots from this secondary source of radiation will be extremely small compared with the primary dose received from reactor fast neutrons.

Environmental Radiation Botany: Changes in Nucleic Acid Characteristics in Mimosa During Postirradiation Senescence. — Nine-day mimosa seedlings (*Albizzia julibrissin* Durazzini) were given a total dose of 1200 rads (tissue dose in air) of fast neutrons delivered over a period of 9 min. Epicotyls of irradiated and control seedlings were excised at intervals between 4 hr and ten days after the irradiation. Total RNA, soluble RNA (sRNA), and total DNA were extracted. The total quantities of each and the base composition of the sRNA were determined. Total RNA as percent of dry weight decreased within 4 hr after the irradiation (from 2.92 to 1.25%). However, six days after the irradiation, the total RNA in the controls had dropped to levels comparable with the irradiated (0.44% of dry weight). This decrease in the controls is a normal phenomenon occurring with age in plants. Total DNA as percent of dry weight also decreased within 4 hr after the irradiation (from 1.29 to 0.26%), while the controls dropped to levels comparable with the irradiated within six days after the irradiation (0.21% of dry weight). Soluble RNA did not vary quantitatively between the control and the irradiated plants throughout the experiment, but it decreased significantly in both between six and ten days after the irradiation.

The base compositions of the sRNA showed some early shifts after the irradiation. Adenine and cytosine did not vary significantly from the controls, but guanine decreased and uracil increased within 4 hr after irradiation.

Such shifts in total RNA, but not in sRNA, suggest rapid decreases in fractions of RNA other than sRNA (primarily ribosomal RNA). The base-composition shifts of sRNA after irradiation could result in alterations in patterns of protein and enzyme synthesis and later differentiation and growth responses in the irradiated plants.

Dynamics of Herbaceous Ecosystems: Secondary Succession on Old Fields. — The combined influences of ionizing radiation, fire, etc., inflicted by possible broad-scale thermonuclear war would destroy many natural ecosystems and would lead to the abandonment of vast acreages of cultivated lands. Secondary succession would occur on these areas, but its rate, nature, and extent are uncertain. Experiments are being planned on secondary succession on old fields on the reservation.

Field observations on and literature study of secondary succession on old-field ecosystems of this area suggest that the following stages occur. After the abandonment of the land, there is a pioneer stage of vegetation which will invade and may persist for several years. Such pioneer plants include ragweed, aster, horseweed, and poverty grass. From as early as two years and lasting for several years, there is an intermediate stage in which the dominants are ragweed, sassafras, blackberry, and broom sedge. These plants change in relative abundance, and as early

as three years and lasting to as many as eight or ten, broom sedge becomes dominant. During this period there is an invasion of woody plants and pines. The pines and shrubs eventually displace the broom sedge by shading and persist for many years. Eventually they are displaced by hardwoods of the climax forest of the area. The rapidity of succession is a function of site fertility, the time of year of abandonment, the climatic conditions, effects of grazing, and chance.

These observable changes in the vegetation component of the ecosystem during secondary succession led to the formulation of hypotheses to be tested in the experiments. These tentative hypotheses concerning movement of nutrients and radionuclides are as follows: (1) The rate and amount of nutrients cycling through any trophic level during a given time increase as the ecosystem advances in secondary succession. (2) The depths from which nutrients are extracted from the soil solution by plants, and the depth to which nutrients percolate through the profile, increase as the ecosystem advances in secondary succession. (3) The physical and chemical processes controlling the fixation and exchange of radionuclides in soils are altered by the biological changes in the soil complex during secondary succession. (4) The rate and amount of nutrients cycling through any trophic level and the resistance of the system to environmental stresses are directly proportional to the diversity of the producers.

Dynamics of Herbaceous Ecosystems: Preliminary Soil Studies. — Experiments are being planned which involve the effects of radiation, fire, herbicides, grazing organisms, and original vegetation type on the rates of nutrient and radionuclide cycling and energy flow during secondary succession. The site selected for this research is an alluvial flood plain on the reservation, much of which is underlain by a fragipan or impervious layer from 18 to 30 in. deep. The experimental treatments described above will be applied in 12 × 12 m plots alone and in various combinations in a fractionally replicated design. Because these will be long-term investigations of ecosystem dynamics in secondary succession and cannot be replicated easily in time, the locations of the plots are especially critical. Because soil conditions are known to have a strong influence on the rate, amount, and type of plant growth, a detailed survey of the chemical and physical soil characteristics of the site is under way.

Initially, several pits were excavated in areas of expected soil extremes; the soil profiles were examined, and color photographs were taken. The soil horizons were determined and measured, and soil characteristics measurable in the field were recorded. Then a 50-ft grid was established on the site. At each grid point two intact soil cores were obtained with a hydraulic coring machine. The coring tube was inserted through the A and B horizons and into the C horizon. The depth of each horizon, the depth to mottling, and other characteristics of these soils were recorded, using the photographs from the pits as references. The information from these field surveys, as well as data from subsequent laboratory chemical and physical analyses on horizon samples, was fed into a computer program to plot out contour maps of soil properties with the Calcomp plotter. The accuracy of the contour maps generated in this manner was verified by comparison with a contour map obtained by conventional engineering surveying

procedures. The contour maps of various soil properties provide soil profile information, not readily determinable from the surface, which is necessary in selecting the plot locations.

Systems Ecology: Models for Intraseasonal Growth in Plants. — In experimental ecology it is desirable to be able to interpret the influence of various treatments on the rate, amount, and duration of growth of plants in the field. Three models are being investigated to express the height of a plant as a function of time, as follows:

$$H = \frac{1}{A + BC^t} ,$$

$$H = \frac{A}{1 + Be^{Ct}} ,$$

$$H = k_1 Ae^{Bt} + k_2(C + Dt) ,$$

where H is height, t is time after initiation of flowering, and A, B, C, and D are parameters. For the third equation, if $t > t_0$, then $k_1 = 0$ and $k_2 = 1$; and if $t < t_0$, then $k_1 = 0$ and $k_2 = 1$, where t_0 is the time of completion of flowering.

Data from a series of fertility experiments performed elsewhere are being utilized in studying these models. Nitrogen and phosphorus were applied factorially at 0, 50, and 100 lb/acre; the heights of randomly selected plants were measured in replicated plots for each treatment at weekly to fortnightly intervals during the growing season. The species investigated were *Agropyron spicatum*, *Stipa comata*, and *Oxytropis serecia*.

The first model, the classical logistic, was found not to have a sufficiently rapidly rising phase of the curve during the period of early growth. The second model, derived from integrating the equation $dH/dt = k_1 t - k_2 t^2$, provided a more satisfactory fit to the data but did not clearly represent the changes for some grasses under certain treatments. The third equation, a discontinuous regression fit, appeared to explain plant growth under these conditions. There was exponentially increasing growth until the time of setting of seed and of approaching soil drought; then there was a linear decrease in height as the plant matured and portions of the plant shattered from physical causes. Different properties of these equations, such as the upper and lower asymptotes and the date at and the height at inflection (where applicable), were utilized in interpreting the influence of the experimental treatments to provide additional insight into plant growth phenomena.

Systems Ecology: Prediction by Nonlinear Programming of Chemical Composition of Dietary Botanical Components. — In a previous report (ORNL-3814, p. 45), computer experiments were described in which least-squares and nonlinear programming routines were compared for various ecological analysis problems. Further development has been made in formulating ecological problems so that nonlinear programming methods may be used in their solution. These methods are being developed for use in analyzing dietary relations in proposed studies with grazing organisms in radioactively contaminated areas, such as the White Oak Lake bed. These methods,

if sufficiently precise, can be utilized to replace costly radionuclide analysis and tedious hand separation of mixed forage samples into species and plant-part components. This operations research technique also is being used to obtain more information about ecological samples than is feasible otherwise.

Relationships between the chemical and the botanical composition of forage samples obtained from esophageally fistulated animals are shown by the following formulations: C_{ik} = the percentage of chemical k in mixed forage sample i, W_{ij} = the proportion by weight of sample i that is contributed by plant j, and $\hat{P}_{jk}$ = the predicted percentage of chemical k in plant j. The concentration of a given chemical in a sample of forage is

$$C_{ik} = \sum_{j=1}^{m} W_{ij} \cdot \hat{P}_{jk} .$$

The predicted percentage of chemical k in plant j is being determined by using nonlinear programming to minimize the following function:

$$Q = \sum_{i=1}^{n} \sum_{k=1}^{p} \left[C_{ik} - \sum_{j=1}^{m} W_{in} \cdot \hat{P}_{jk} \right]^2 .$$

The solution matrix, $\hat{P}_{jk}$, has the following constraints: $\hat{P}_{jk} \geq 0$ for all j and k, and

$$\sum_{k=1}^{p} \hat{P}_{jk} = 100$$

for all j. Furthermore, knowledge of the minimum and maximum possible values for chemical constituents of individual plants or plant parts may be utilized in the solution. Similar studies are in progress to use these methods to predict the digestibility of different chemical constituents of individual plant parts from the above relationships and from knowledge of digestion of the mixed sample as measured by micro techniques.

Movement of Nuclides in Terrestrial Environment. — Following one year of experimentation (ORNL-3762, p. 41; ORNL-3835, p. 38) and 53 in. of rain, the ^{137}Cs loss amounted to 31% on the bare-soil plot, 19% on the clipped-meadow plot, and 6.7% on the tall-meadow plot. On the vegetation-covered plots more than 95% of the ^{137}Cs loss occurred during the winter and spring months, as compared with 67% loss from the bare-soil plot. More than four-fifths, and more than one-half, of the cesium loss on the tall-meadow and clipped-meadow plots, respectively, was due to the five high-energy and high-intensity rains that occurred during the winter and early spring seasons. The same five rains accounted for only one-third of the ^{137}Cs loss on the bare-soil plot.

After one year of experimentation, 6% of the applied ^{137}Cs was still found in the litter material, which constituted the 1964 vegetation, as

compared with 26% reported for the tall and the clipped meadow in the early spring. Less than 1×10^{-10} μc of ^{137}Cs per gram of dry material was found in the new 1965 vegetation, indicating that no significant uptake occurred during the 1965 growing season.

The average specific loss of ^{137}Cs from the bare-soil plot was 0.137 μc/g in the eroded material and 5.0×10^{-6} μc/cc in the liquid phase. The distribution coefficient for the A_p horizon of Captina soil was calculated to be 27,400 cc/g from field data, as compared with the 30,000-cc/g value obtained in the laboratory. The average specific activity of the eroded material from the vegetation-covered plots was 0.86 μc/g. The above values are indicative of the average ^{137}Cs concentration in the soil following surface application of 0.9 mc/m^2 of ^{137}Cs. For different rates of application, an appropriate factor should be incorporated in the calculations. The higher specific activity in the eroded material from the vegetation-covered plots reflects the leaching loss from the vegetation and litter as contrasted with the loss from soil alone.

A second series of replicate plots have been started. These plots are scheduled to be dosed with ^{137}Cs in the early part of April to determine the seasonal effect on the rate of radionuclide loss under natural conditions.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Radiation Physics: Almost Longitudinal Instabilities in a Plasma-Beam System. — A plasma-beam system immersed in a magnetic field B_0 has been investigated under the assumption that the plasma is stationary, cold, uniform, and extended and that the beam of charged particles is uniform in density and moving in a direction parallel to that of B_0. Instabilities that are almost longitudinal were investigated with respect to conditions of occurrence, strength and type of instabilities, and dependence on the angle between the direction of B_0 and the direction of the excited waves. Two logically distinct types of waves that occur were investigated. An almost longitudinal Cerenkov wave has a phase velocity equal to the projection of the beam velocity in the direction of the wave. An almost longitudinal cyclotron wave has a frequency equal to the Doppler-shifted gyrofrequency of the particles in the beam.

Radiation Physics: Variational Calculation for Bound States in an Electric Dipole Field. — The quantum mechanical problem of an electron moving in the field of a permanent electric dipole has been investigated in an attempt to determine whether negative-energy eigenvalues, that is, bound states, exist for small dipole moments. When the expectation value of the Hamiltonian is minimized in a variational calculation and then set equal to zero, the solution of the resulting equation gives a value D_{min} of the dipole moment which is sufficient to assure the existence of a bound state. The trial wave function $\psi = \exp(-\alpha r^t)(C_0Y_{00} + C_1Y_{10})$ gives $D_{min} = 1.65 \times 10^{-18}$ esu-cm for an electron in the field of a finite dipole.

Radiation Physics: Surface Plasmon Dispersion Relation for an Electron Gas. — The dispersion and damping of surface plasmons in a semi-infinite free-electron gas has been examined using a specular-reflection

model for electrons at the bounding surface. It was found that this dispersion relation agrees well with the predictions of the hydrodynamical model for small wave numbers. The damping rate, which is predicted to be zero on the hydrodynamical model, was found to be less than 1% of the plasmon frequency over a substantial range of wave numbers.

Radiation Physics: WKB Treatment of Bound States in an Electric Dipole Potential. — The Wentzel-Kramers-Brillouin method has been applied to the problem of the bound states of an electron in the field of a finite dipole. A necessary condition for binding was derived which suggests that bound states may exist only if the dipole moment is larger than a certain minimum value. It was shown that the number of bound states given by the WKB method is comparable with the exact number for the one- and three-dimensional square-well potentials.

Radiation Physics: Time-of-Flight Determinations of Electron Diffusion Coefficients and Electron Drift Velocities in Ethylene and in Water Vapor. — The method described by Hurst *et al.* [J. Chem. Phys. 39, 1341 (1963)] for the determination of electron diffusion coefficients, D, and drift velocities, w, from time-of-flight studies of the motions of individual electrons in gases has been improved in two essential ways. First, an accurate method of measuring all fluctuation in the time-of-flight distribution except that due to the diffusion process itself has been developed. Second, a data analysis procedure has been worked out in which the effects of instrumental fluctuations are removed and accurate values of the parameters D and w are found from a generalized least-squares procedure, making use of a digital computer. The previous assumption that the detection of single electrons follows Poisson statistics was examined experimentally and found not to be in error. Distortions in the electron time-of-flight distributions arising out of electron losses during detector dead times were calculated. Application of the improved method has been made to ethylene and to mixtures of ethylene with water vapor over the range of E/P (ratio of electric field E to pressure P) from 0.01 to 1.0 v cm^{-1} $torr^{-1}$. Independent measurements of the quantities D and w were made; the ratios D/w were compared with theory in the region of thermal electron energies. The ratio of the momentum transfer cross sections for H_2O and ethylene was the same whether use was made of D(E/P) or w(E/P) data in the region of thermal energies. It was found that D/w for thermal electrons was 12% higher than theory. After careful error analysis, it was suggested that infrequent long-duration collisions between electrons and ethylene may account for the larger D values.

Radiation Physics: Interaction of Low-Energy Electrons with Benzene Derivatives. — We have studied the interaction of low-energy electrons with a series of benzene derivatives using the combined swarm-beam method. For C_6H_5Cl, *o*-, *m*-, and *p*-$C_6H_4Cl_2$, *o*-$C_6H_4CH_3Cl$, C_6H_5Br, C_6D_5Br, and *o*-$C_6H_4CH_3Br$, two distinct electron capture processes have been observed: a process at thermal energies and a process at about 1 ev. Both processes are believed to be dissociative, since Cl^- and Br^- were the only negative ions detected with a time-of-flight mass spectrometer.

Internal Dose Estimation. — A method for computing the dose to a tissue phantom from a gamma-ray source has been developed using a Monte-Carlo-type computer program. The source subroutine can be rewritten to

suit the specific problem, and thus the source may be outside the phantom or within the phantom. Each photon is assigned a weight of 1 in starting, and the weight is reduced following each interaction by multiplying by the ratio of the cross section for Compton scattering to the total cross section. The cross sections are taken from National Bureau of Standards Circular 583 and its supplement and are programmed on a separate subroutine.

The code has been used to obtain preliminary estimates of dose for a particular case of some interest for clinical practice. Neohydrin, a compound of mercury, is known to concentrate in the cortex of the kidney, and thus when labeled with radioactive mercury is often used in scanning procedures. The code has been used to estimate the dose to the cortex, the medulla, and various other regions of the body from ^{203}Hg uniformly distributed in the cortex of the kidney. The body is taken as a homogeneous right circular cylinder with the composition of standard man, and the cortex and the medulla of the kidney are represented by geometrical configurations approximating the size and shape of these tissues.

The results now available are preliminary in that they estimate only the dose due to the presence of ^{203}Hg in the cortex. It is found that the ovaries receive about 7.5 millirads/hr per millicurie deposited in the cortex, and the dose rates to the cortex and the medulla are about 15 times as high. It is planned to use the code to estimate the dose received during the early period following administration while the neohydrin is circulating in the blood, and also the dose delivered after the ^{203}Hg has been eliminated from the kidney. The code offers considerable flexibility in programming the source and target regions and is intended for use in estimating the dose to a variety of organs for many exposure situations.

TRAINING, EDUCATION, AND INFORMATION

Professional Health Physics Training. — The summer field training program was completed. Nineteen students from Vanderbilt University and the University of Tennessee were trained in applied health physics and research health physics activities. A lecture and seminar program was included. The students were supported by the AEC, the U.S. Public Health Service, or both.

Physics 472 (General Health Physics) at the University of Tennessee was completed. Eleven students were enrolled. Physics 473, which is a continuation of Physics 472, will begin in January 1966. Chemistry 546 (Radiation Chemical Physics) was completed with an enrollment of 9. Physics 661 (Interaction of Electrons with Gases) was completed with an enrollment of 5. All of these courses were taught by Health Physics Division personnel.

The Division cooperated with ORINS in the presentation of a ten-week course in health physics for state health personnel. Nineteen students completed the course.

The Education and Training Section assisted ORINS by presenting lectures as a part of two courses in medical radioisotopes. There were 34 medical doctors enrolled in the two courses.

There were five Oak Ridge Graduate Fellows and six Special Fellowship students (AEC-USPHS) working on advanced degrees in the Division under the supervision of senior scientists of the Radiation Physics Section and the Radiation Ecology Section.

Staff members of the Division visited 19 colleges and universities to acquaint faculty and students with research opportunities at ORNL, the possibilities of careers in health physics, and the AEC health physics fellowship program.

ISOTOPE DEVELOPMENT

Fission Products Development. — In a hot-cell test demonstrating the recovery of cesium from Hanford sugar-treated waste (STW) with 4-*sec*-butyl-2-(α-methylbenzyl)phenol (BAMBP) in Amsco 125-82, there was no apparent degradation of the BAMBP in 80 extraction-strip cycles. The final solvent contained a negligible amount of nitrated BAMBP and showed no loss of cesium extraction power when compared with the original solvent in standard cesium extraction tests. About 92% of the cesium was recovered in the demonstration run. Phase separation was satisfactory; the decontamination factors for cesium were 1000 from strontium and iron; over 2000 from aluminum, cerium, zirconium-niobium, and ruthenium; over 100 from chromium, nickel, and molybdenum; and over 700 from sodium.

Stable Isotopes Development. — A double-arc system incorporating a set of 180° beam boundary plates is being studied to further the technology of two-arc operation with the long-range objective of lowering isotope costs. Preliminary comparative results of the two systems show that the double-arc system gives over 65% more ion output than the standard one-arc unit.

A survey on the collection of kilogram to multikilogram quantities of about 95% ^{170}Er has been made. This isotope is used as target material in the production of ^{171}Tm, which is being considered for application in a specialized nuclear heat source. The quantities requested are beyond the capabilities of present calutron facilities; however, kilogram quantities of ^{170}Er could be supplied for experimental purposes.

A special isotope collection is in progress using the new concepts for high-purity separations (recessed-pocket receiver, calcium pumping, beam boundary plates, and reduced baffle opening) to evaluate the feasibility of collecting milligram quantities of low-abundance rare-earth isotopes. A run is in progress with emphasis on collecting ^{136}Ce.

Attempts to increase the isotopic abundance of ^{33}S and ^{123}Te have resulted in the highest purities achieved to date (78.6% ^{33}S and 85.9% ^{123}Te). Since these values appear to be limited to some extent by the large angular divergence of the ion beam as it leaves the ion source, this effect must be minimized before maximum benefits can be derived from new receiver concepts. Equipment components (liner, source, and receiver) required for studying the relationships between angular divergence of the beam and ion source parameters have been assembled, and evaluations will be made in the near future.

Special Separations. — Gram quantities of ^{238}Pu have been separated for the first time in the ORNL calutrons. From a feed material of 48.5 g of 79% ^{238}Pu, about 5.4 g of ^{238}Pu was collected containing 99.45 ± 0.05% ^{238}Pu. Source and receiver components designed for the americium separation were used for processing this material during the two runs (one 78 hr and one 115 hr).

Target Preparation. — Polished, chromium-plated stainless steel and Pyrex were tried as substrates for the preparation of self-supported films of silicon and germanium, since their smooth surfaces had fewer surface imperfections (scratches, rolling lines, etc.) than other substrate materials. Because of the low ductility of both silicon and germanium, such imperfections caused the films to crack when removed from the substrate. Studies of appropriate combinations of substrate, parting agent, and substrate temperature during evaporation resulted in the use of evaporated NaCl as the parting agent on any substrate, with a temperature of 20°C or lower. This latter condition improved the ductility of both silicon and germanium films. However, both glass and stainless steel proved to be poor substrates, causing considerable strain in the condensed films. After a wide variety of metal substrates had been tried, tungsten sheet was found to yield adequate ductility of the film and the most reproducible results. When unpolished, rolled 0.010-in. tungsten sheet was used, even the rolling marks in the substrate caused only minimal rupture during film release and mounting.

A new type of tungsten evaporation crucible, similar to a Knudsen crucible, having a 0.020-in. effusion port at the top, was prepared to limit sparking of the evaporants. Sparks striking the condensing substrate caused pinholes in the films. The results to date indicate that this crucible is vastly superior to the graphite crucibles previously used with germanium and also to the open tungsten crucibles (or beryllium oxide crucibles) used for silicon evaporations. Self-supporting films having thicknesses between 10 and 1500 $\mu g/cm^2$ have been prepared using these improved crucibles and other techniques noted above. Polished tungsten substrates are now being fabricated which should tend to eliminate the discontinuities in the condensed films.

Samples of magnetically oriented thin films of elemental iron have been prepared for solid-state and neutron-physics experiments. The samples consist of a glass substrate onto which gold electrical contacts were deposited by condensation of gold vapor. Alternate layers of iron and MgF_2 or SiO were produced by vapor condensation in a fixed magnetic field having a strength of about 20 gauss. Permanent Alnico magnets were used for this purpose. The samples were prepared with a sandwich structure consisting of a layer of iron (about 800 A thick), a layer of MgF_2 or SiO (50 to 100 A), a second layer of iron (about 1600 A), and a cover layer of MgF_2 or SiO (50 to 100 A) over the the entire substrate and all condensed films. Measurements of film thickness were made during the evaporation of each material by measuring the frequency shift of an oscillating quartz crystal, f_0 = 6 Mc/sec. During the evaporation of all the components, the glass substrate was maintained at a temperature of 300°C to ensure proper adhesion and annealing of the condensate films.

Radioisotope Research and Development. — The thermal-neutron cross section and effective resonance integral for the $^{186}W(n,\gamma)^{187}W$ and

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$^{187}W(n,\gamma)^{188}W$ reactions were determined by irradiating 10-mg samples of WO_3 (containing 97.2% ^{186}W) with and without cadmium shields. The thermal-neutron flux and epithermal-neutron flux were monitored with cobalt (cross section, 37 barns) and gold (resonance integral, 1558 barns) respectively. The thermal-neutron capture cross sections determined for the $^{186}W(n,\gamma)^{187}W$ and $^{187}W(n,\gamma)^{188}W$ reactions were 33 and 64 barns respectively, and the effective resonance integrals were 318 and 2760 barns respectively.

Tungsten-188 (half-life, 69 days) decays to ^{188}Re (half-life, 17 hr), which in turn decays to stable ^{188}Os. Since rhenium behaves chemically like technetium, ^{188}Re may be useful in medicine in a manner similar to ^{99m}Tc [Nucleonics 23(8), 106 (1965)]. The major advantage of ^{188}W-^{188}Re over ^{99}Mo-^{99m}Tc is the longer half-life of the parent (69 days for ^{188}W, as compared with 66 hr for ^{99}Mo). An experimental isotope generator was prepared by sorbing $^{188}WO_3$ on an HZO-1 (hydrous zirconium oxide) column and eluting ^{188}Re with normal saline solution. When the eluted ^{188}Re was counted after decaying through seven half-lives, no ^{188}W was detected.

In our systematic investigation of gold radioisotopes, attention has now been directed to decreasing the ^{198}Au content in the ^{199}Au product. Gold-199 is routinely prepared in the ORR by the irradiation of natural platinum (7.23% ^{198}Pt and 25.4% ^{196}Pt). The ^{199}Au is formed by the reaction $^{198}Pt(n,\gamma)^{199}Pt$, followed by beta decay of ^{199}Pt to ^{199}Au. Trace amounts of ^{198}Au, usually 0.1 to 0.5%, have been found in the ^{199}Au product. Spectrographic analysis of the platinum target shows that the gold content is below 1 ppb, which indicates that the ^{198}Au is not produced from gold contamination. Another way by which ^{198}Au could be introduced into the ^{199}Au product is by the successive reactions $^{196}Pt(n,\gamma)^{197}Pt$, beta decay of ^{197}Pt to ^{197}Au, and $^{197}Au(n,\gamma)^{198}Au$. Studies were conducted to determine whether 0.1 to 0.5% ^{198}Au would be produced in the ^{199}Au product when platinum was irradiated for about 60 hr at a flux of 10^{14} neutrons cm^{-2} sec^{-1}, and if so, to find a method of eliminating the ^{198}Au.

Three 10-mg samples of natural platinum were irradiated in the ORR: one without purification, another purified by solvent extraction procedures to remove traces of gold, and a third purified by solvent extraction and shielded by 40 mils of cadmium. The results given below show that ^{199}Au can be prepared essentially free of ^{198}Au by using a 40-mil cadmium filter on the platinum target.

Conditions	Yield (mc)	
	^{199}Au	^{198}Au
Without purification of target	20.7	0.024
Target purified to remove ^{197}Au	20.6	0.024
Target purified to remove ^{197}Au and cadmium shielded	12.0	<0.002

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A separation procedure (generator type) for ^{131}Cs (half-life, 9.7 days) was developed by placing ^{131}Ba (half-life, 11.6 days) on a Bio-Rex-70 ion exchange column and eluting the cesium with 0.5 M ammonium acetate. The ^{131}Ba was obtained by irradiating 50 mg of natural barium carbonate at an average flux of 2×10^{14} neutrons cm^{-2} sec^{-1} for 60 hr in the hydraulic tube facility of the ORR. The first elution using 0.5 M ammonium acetate contained various gamma energies which were not identified but presumably were due to ^{24}Na and other impurities formed in the silica ampul during irradiation. Subsequent elutions contained ^{131}Cs with less than 0.009% ^{131}Ba.

The equipment for the enrichment of ^{85}Kr by thermal diffusion is now in partial operation. The first cascade of three columns has been loaded with about 2000 curies of 5% ^{85}Kr and has been operating satisfactorily for two months. The other cascade of three columns is still operating on nonradioactive krypton but will be loaded with radioactive gas in January 1966.

Several prototype beta source capsules were fabricated and tested for structural integrity. The capsules are stainless steel cylinders with a 10-mil-thick beryllium window in one end to transmit the radiation with minimum losses. The results of the structural tests show that the capsules are a substantial improvement over previously used beta source capsules, which use very thin stainless steel or aluminum windows to obtain equivalent transmission. Temperature resistance tests on the prototype capsules are in progress.

The release of tritium from tritiated paints during the drying period immediately following application on a watch or instrument dial was investigated. The tritium loss during a 4.5-hr drying period at 28°C varied from 0.14 to 2.56% among five different commercial paints.

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