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# OAK RIDGE NATIONAL LABORATORY

# STATUS AND PROGRESS REPORT

# DECEMBER 1961

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

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

DECEMBER 1961

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.

## SPECIAL NUCLEAR MATERIALS PROGRAM

Dissolver Solution Analyses: Coulometry of Uranium. — Studies on the use of an aqueous supporting medium of 0.1 M sulfamic acid and 0.5 M sulfuric acid in the controlled-potential coulometric titration of uranium reveal that (1) procedural details need not be changed when this electrolyte is used, except that the test portion must be transferred to the electrolyte before mercury is added; (2) sulfamic acid effectively eliminates the interference of nitrite; (3) uranium can be determined accurately in this electrolyte, even in the presence of as much as 5 M $HNO_3$, 1 M $Th(NO_3)_4$, 0.5 M $LiNO_3$, or 0.15 M $Al(NO_3)_3$. It has been observed, furthermore, that increasing the concentrations of sulfuric or sulfamic acid does not significantly alter the tolerance limits cited above.

Work has been continued on the procedure for the determination of uranium by extraction from tri-n-octylphosphine oxide—cyclohexane and coulometric titration. It has been established that uranium can be stripped quantitatively from TOPO-cyclohexane with an aqueous solution of 4 M $H_3PO_4$—1 M $K_3PO_4$—1 M $H_2SO_4$ in two passes and that the uranium can then be titrated coulometrically in this medium without the necessity for further treatment. Similarly, it has also been found that uranium can be titrated directly in the aqueous phase without further treatment after stripping it from the organic phase by three passes with 2 M $Na_2SO_4$ (pH of 1). The relative standard deviation of either procedure, including extraction, stripping, and titration, is indicated as being about 0.2% for samples containing 5 mg of uranium.

Dissolver Solution Analyses: Coulometry of Uranium(IV). — A method is now being developed for the determination of U(IV) by controlled-potential coulometric titrimetry. The studies indicate that U(IV) can be oxidized to U(VI) at +0.1 v vs the S.C.E. at a mercury electrode in a medium of pH 8 which is 6 wt % sodium tripolyphosphate in water. This method has been applied to the determination of the U(IV) and of the total uranium content in samples of $U_3O_8$. The relative standard deviation appears to be about 2% for samples containing 1 mg of U(IV).

[redacted]

# REACTOR DEVELOPMENT PROGRAM

## GAS-COOLED REACTOR PROGRAM

EGCR Temperature Coefficient. – The free-gas model for neutron thermalization has been used to calculate the EGCR moderator temperature coefficient of reactivity over a range of temperatures and exposures. For the core at operating temperature the moderator coefficient at zero exposure equals $-2.4 \times 10^{-5}$ $(\delta k/k)/°C$, and the coefficient becomes positive at an exposure of 5000 Mwd/metric ton. For the initial core at its calculated terminal exposure of 7100 Mwd/metric ton, the moderator coefficient is positive for average graphite temperatures above 900°F and reaches a peak value of $+1.7 \times 10^{-5}/°C$ at 1350°F. Over the range from room temperature to fuel temperature of 1700°F, the fuel (or Doppler) coefficient varies from $-3.5 \times 10^{-5}/°C$ to $-2.0 \times 10^{-5}/°C$, so that the isothermal temperature coefficient is always negative.

EGCR Top and Bottom Dummy Test. – The temperature of the EGCR graphite moderator columns is controlled by the annulus coolant flow that is diverted from the main stream through fixed orifices located in the bottom dummy assembly. The flow that diverts to this annulus is a function of the pressure drop across the fixed orifices in the bottom dummy, the pressure drop in the annulus, and the pressure drop across the controlled gap in the top dummy.

The fixed orifice is located upstream of the adjustable orifice in the bottom dummy assembly and downstream of the bottom fuel-assembly spider. Since the orifice is close-coupled to regions of flow turbulence, the effects of adjustable orifice position, main channel flow, and fuel spider geometry were incorporated in the test to determine the effect on the fixed orifice flow vs pressure drop.

A test rig has been constructed to determine the design parameters for the top and bottom dummy as related to this annulus flow. With the orifice hole plugged, the leakage from the main channel to the annulus was determined prior to recording annulus test data. This leakage was determined as a function of the pressure drop from the main channel to the annulus and, therefore, must be applied as a correction factor to the data for pressure drop vs flow across the fixed orifice.

With an empty graphite fuel sleeve above the bottom dummy, the flow vs pressure drop across the fixed orifice was recorded for pressure drops from 1 to 9 psi for an adjustable orifice position of 0 and 5 in. and main channel flow of 600 to approximately 2700 lb/hr of air.

GCR-ORR Loop No. 1. – Loop No. 1 continued to operate in the irradiation of prototype instrumented EGCR fuel elements. On December 17, the second of a pair of elements having a heat generation of 25,000 Btu $hr^{-1}$ $ft^{-1}$ was removed from the loop; it is ready for hot-cell examination. The third element, one generating 35,000 Btu $hr^{-1}$ $ft^{-1}$, has been inserted and will be irradiated during the next reactor cycle.

GCR-ORR Loop No. 2. – Electrical and instrument cables and thermocouples for loop No. 2 have been pulled from the penetration junction box to the vicinity of the equipment in the cell. The heater has received

its final helium leak check. Temporary instrumentation has been installed on the South Facility cooling-water lines in order to establish pressure drops through the various cooling passages. The test-plug carrier has been received from Paducah and installed on its dolly outside the ORR building.

Drawings for the test plug for the low-power fission-gas source are complete except for final checking. Detailing of the gas sampling system is 80% complete. Layouts and 35% of the detailing have been completed for the cold-finger assembly, which is designed to be inserted in the beam hole when the reactor is at power and a test is not being conducted.

High-Temperature Measurements for Gas-Cooled Reactor. — Chromel-P—Alumel thermocouples sheathed with type 304 stainless steel were tested at 870°C in 7-psig hydrogen and in 7-psig carbon monoxide. Examination of the hydrogen-exposed couples by slow withdrawal from an isothermal tube furnace revealed inhomogeneity which was most severe in the region 10 to 11 in. from the hot junction. This corresponds to an exposure temperature of 780 to 660°C and is in agreement with previous experience.

The tests of thermocouples in 150-psig hydrogen at 870°C and in a graphite-helium environment at 700°C are continuing. The couples exposed to the high-pressure hydrogen for 78 days show essentially no drift. The emf of the low-temperature graphite-helium couples is down −0.08 mv (−2°C) after 112 days.

A W vs W—26% Re thermocouple was tested in a graphite-helix furnace for 120 hr at 1700°C in helium. During the first 24 hr of operation no drift data were obtained. During the last 96 hr the emf of the couple decreased by 1.2 mv (60°C) without apparent change in furnace temperature as measured by an optical pyrometer and furnace power input.

BeO Irradiation Studies. — The current BeO irradiation experiment, ORNL-41-7, is being tested in the J-12 facility at the ETR. The irradiation temperatures range from 550 to 1150°C at an estimated fast flux of the order of $2 \times 10^{14}$ nv.

The instrumentation and the hardware for irradiation tests 41-8 and 41-9 have been reviewed in detail with the Phillips Petroleum Company personnel.

Evaluation of Finned Beryllium Tubing. — Low-voltage radiography has been completed on the helical-finned beryllium tubing from Nuclear Metals, Inc. Because of the removal of numerous tubing lengths for other tests, only 22 of the original 50 pieces were available for continued evaluation. Of the 22 sections, 13 contained varying amounts of high-density material, and 18 had a few small shallow pits. Six discrete indications which had been detected by eddy-current examination were relocated and marked for correlation with other examinations and metallography.

A special jig has been constructed to measure the wall thickness of the tubing between the fins. The newly developed through-transmission eddy-current method will be used.

## THORIUM UTILIZATION PROGRAM

Thoria Pellet Development. — Studies of the effect of heating time on the sintered density of $ThO_2$ pellets have shown that, for a given green

density, increasing the sintering time from 41 min to 60 min at 1750°C in hydrogen increases the sintered density by about 2%. For small cylindrical pellets pressed mechanically to green densities of 4.10, 4.50, and 5.19 g/cm$^3$, sintering at 1750°C for 1 hr in hydrogen produced final densities of 8.50, 9.07, and 9.54 g/cm$^3$. The surface areas of the pellets were, respectively, 0.13, 0.081, and 0.012 m$^2$/g. When wet-milled with 1/4-in. spherical alumina balls, the pellets lost 4.1, 1.2, and 1.0 wt % per hour. In accelerated attrition tests by the spouted-bed technique, the pellets lost 2.0, 0.61, and 0.85 wt % per hour. Microscopic examination of the pellets after ball milling showed that large particles of material had been removed from the pellets of lowest density, indicating poor bonding due to low forming pressures. Attack on the highest-density pellets was accentuated at lamination cracks, resulting from too high forming pressures. Experiments in which green pieces were formed mechanically at low green density (4.1 g/cm$^3$) and then isostatically pressed at pressures up to 35,000 psi have shown that lamination-free pieces can be obtained with a sintered density of 9.6 g/cm$^3$.

Fuel Cycle Development: Chemistry of Solid Fuel Element Materials. — A study of the oxidation in air of $ThO_2$-$UO_2$ solid solutions was extended to include measurements at lower temperatures. Powdered samples were prepared from sintered and hydrogen-reduced pellets containing $ThO_2$ and $UO_2$, varying in composition by 10 mole % intervals. During equilibration in air at 183 to 193°C, weight gains were not found after three weeks for specimens containing 0 and 80–100 mole % $ThO_2$, nor after seven weeks for specimens containing 10–20 and 60–70 mole % $ThO_2$. Specimens containing 30 to 40 mole % $ThO_2$ continued to gain very small amounts of weight even after nine weeks.

These results are compatible with the results of equilibrium dissociation-pressure measurements.

Fuel Cycle Development: Fuel Preparation. — Development studies on the sol-gel process for preparing highly compactible 3 wt % uranium–thorium oxides were continued. Oxide fragments satisfactory for vibratory compaction (> 8.7 g/cc, > 87% of theoretical density) were obtained by the addition of uranyl nitrate (50 g of U per liter) solution to thoria prepared by steam denitration at 425°C without subsequent air calcination. Mixed sols adjusted to pH 3.2 to 3.5 with ammonia gave gels of uniform composition on evaporation. Sizing in the evaporated-gel step gave mixtures which were vibrated to bulk densities of 8.68 and 8.63 g/cc. Mixed oxides of > 8.5-g/cc packed density were prepared from the dried gels in two steps: (1) calcination in air by increasing the temperature at a rate of 300°C/hr to a maximum of 1150°C and holding for 4 hr and (2) reduction at 1150°C for 4 hr in argon–4% hydrogen.

In engineering development, the 15-kg $ThO_2$-batch rotary steam denitrator was operated in eight runs without mechanical difficulty. Control of dispersibility and of N/Th ratio of the $ThO_2$ product was demonstrated in the last four runs. Sol-gel product prepared as a 10-kg batch was compacted to 8.9 to 9.0 g/cc by vibratory compaction in tubes 0.5 in. in diameter and 4 ft long.

Fuel Cycle Development: Fuel Fabrication Studies. — Previous work with fused $ThO_2$ has indicated that for a distribution containing two size

fractions, the vibrated bulk density tends to increase with an increase in the amount of the fine fraction smaller than 10 μ. A maximum limit on the amount of material smaller than 10 μ has been established at ~35% of the fine fraction. A decrease in density results if more than this amount is smaller than 10 μ.

Vibrated bulk densities of sol-gel $ThO_2$-$UO_2$ obtained with distributions containing three size fractions were at least 2% greater than those obtained with distributions containing two size fractions.

A Braun disk pulverizer was found to be unsatisfactory for generating the medium-size fraction, because iron contamination was excessive and the plate spacing changed during operation. The medium fraction can be obtained by ball milling. A Denver fireclay-jaw crusher was found to be satisfactory for producing the coarse fraction from the crude sol-gel $ThO_2$-$UO_2$.

Conceptual design of the remote-fabrication facility (planned for construction in cell 4 of Building 3019) for fabricating $U^{233}O_2$-$ThO_2$ fuel rods for Brookhaven National Laboratory is almost completed. This facility will serve as a pilot plant to the proposed Thorium Fuel Cycle Development Facility. Detailed design of the fuel-rod fabrication equipment is ~10% complete. In addition, the preparation of a preliminary proposal for AEC approval will be completed by January 1, 1962.

## MOLTEN-SALT REACTOR PROGRAM

MSRE Design. — Drawings for the radiator enclosure and the reactor vessel were issued for construction. The heat-exchanger and pump-mount drawings were completed. Drain-tank drawings were modified to increase the diameter from 48 to 50 in. to provide more gas space. Drain-tank-cell layout drawings are now 60% complete.

The manpower applied to electrical drawings was increased in order to ensure completion of the electrical design on schedule. All phases of electrical design are now in progress.

Design of the control rod, drive, and position-indication system was completed, and a mockup is being built for testing.

The component coding system for freeze flanges, pump top, and freeze valves is 75% complete. Final drawings for the thermal shield and reactor support are in preparation.

Construction drawings were revised to provide for the graphite sampling plug and sleeve relocations in the cell shielding blocks.

MSRE Procurement and Construction. — Final shipments of INOR-8 from Haynes Stellite Company were delayed because of lack of equipment to perform some of the ultrasonic inspections required. All INOR-8 materials from Haynes Stellite, except replacements for rejected material, are now expected to be shipped to Oak Ridge in January.

Excavation for the drain-tank cell was completed by the contractor, and welders are being qualified for welding of the structural steel in the cell. The contractor is also installing reinforcing steel; he estimated 6% completion of the major building modification work as of December 1.

Development of Components and Systems. — The attempt to dissolve deposited $ZrO_2$ in the engineering test loop by additions of $ZrF_4$ to the flush salt was unsuccessful (see ORNL-3223, p 7); so a mixture of $H_2$ and HF was bubbled through the salt for a total of 68 hr in a 500-hr period. Judging from the amount of by-product water collected, this hydrofluorination treatment was successful in converting the $ZrO_2$ to $ZrF_4$. The chromium content of the salt mixture ($LiF$-$BeF_2$-$ZrF_4$, 62-34-4 mole %) increased ~25 ppm during the 500 hr, indicating only a small amount of corrosive attack.

The loop was shut down for major alterations, which include the installation of a container in which graphite will be tested for compatibility with the salt. Fabrication problems have delayed the scheduled delivery of the graphite by two months.

A 2-in. Stellite-faced, buffered gate valve planned for use in the sampler-enricher system was cycled open and shut 100 times. With 40-psig helium buffer pressure between the two seals, the leakage through both seals was only 0.3 cc of He per minute with an acceptable closing torque of 75 lb-ft.

The test of the MSRE core-tank heater has continued for 2306 hr; the surface temperature of the heater is 1425°F and produces a 1325°F tank-wall temperature. The typical heater for 5-in. pipe runs has been operated continuously for 1150 hr with a power input of 440 w per linear foot of 5-in. pipe, producing a 1275°F average pipe-wall temperature.

The development of a joint for remote brazing was continued. An INOR-8 female joint member was "tinned" with approximately 0.015 in. of gold-nickel braze and brazed in a bench setup by heating it to 1850°F for 30 sec under an argon atmosphere and with continuous axial pressure. The joint was tested hydrostatically to 4500 psi, helium leak-checked, inspected ultrasonically, and sectioned for metallography; it appears to have no disqualifying flaws. A test braze made with 0.005-in.-thick preformed braze-metal sheet stock instead of tinning has passed the hydrostatic and helium leak check and is undergoing metallography.

The brazed-joint test has been cycled 83 times with a total exposure (to 1230°F salt) of 56 hr.

In the MSRE maintenance mockup, freeze-flange clamps were operated both directly with manual torque wrenches and remotely with the "freeze-flange torqueing tool." These operations indicated the necessity for making design modifications in the clamps to avoid imposing bending moments on the piping during flange makeup. Equipment for jacking the circulating pump and for pipe alignment was installed. The equipment for covering opened flanges to reduce the spread of contamination was fabricated and was received for testing.

Testing of the 3-1/2-in. Inconel freeze flange was discontinued after gasket-groove distortion was noted for the second time. The old design provided insufficient metal in the land outside the groove to resist the clamping forces without distortion. The 5-in. Inconel flange of MSRE design has been thermal-cycled 33 times between room temperature and 1200 to 1400°F with no measurable permanent distortion.

MSRE Pump Program. — Inspection of the experimental pump with the molten-salt-lubricated bearing showed slight pitting and some signs of rubbing on the journal and bearing. All pin joints in the gimbals mount

were operable, and the other parts appeared to be in a satisfactory condition.

The Inconel centrifugal molten-salt pump (PKP type) was inspected and then reassembled for operation after replacement of a slightly worn lower shaft seal.

Castings for the volutes and impellers for the fuel and coolant pumps were received from foundries and are undergoing inspection. Some shrinkage defects were found, as well as some deviations in chemical analysis. Radiographs of the impeller castings are being reviewed.

Inspection and assembly of the rotary element for the MSRE prototype pump were begun, and completion is expected within four weeks. The flexible pump mount to be utilized in the MSRE is being installed in the pump test stand.

Hydraulic performance data, including startup and coastdown characteristics, were taken in the water test of the coolant pump with an 11.6-in.-diam impeller. The data compared favorably with the vendor's prediction.

Final design drawings of the reactor fuel-pump tank, the lubrication units, and the containment vessel for the fuel- and coolant-pump drive motors were completed and are being reviewed.

Metallurgy. — A series of tests were initiated to evaluate the effects of $CF_4$ on INOR-8 and other materials. Three such experiments were completed during the past month after operation at 600, 700, and 800°C, respectively, for 500 hr at a $CF_4$ pressure of 6 psig. None of the materials showed significant reaction with $CF_4$ at 600 or 700°C. At 800°C, INOR-8 and nickel appeared relatively unaffected; however, Inconel, molybdenum, and type 304 stainless steel specimens exhibited relatively heavy reaction films. Deep pits were evident on one of three molybdenum specimens in this test. Some seal leakage occurred during the 800°C test and may have contributed to the attack noted.

Metallographic examinations of test specimens and analyses of gas samples removed from the test are in progress.

A second series of tests is being planned. Specimens of INOR-8 in the first series showed a light "tarnish," which may have resulted from reaction with oxygen contaminants in the gas. (Preliminary analyses of gas samples have shown as much as 2.5 mole % CO in the gas appropriated for these experiments.) The degree of passivation which this film may have provided to the INOR-8 specimens will be checked in the second series of tests, which will contain fused fluorides as well as $CF_4$. The $CF_4$, after passing through a purifier, will be continuously bubbled through a molten salt containing INOR-8 metal coupons. Test temperatures will again be in the range 600 to 800°C.

Instrumentation Development. — Further progress was made in the development of a differential scanner-alarm system for use in monitoring MSRE temperatures during heatup. The use of "make-before-break" contacts on the mercury-jet commutating switch resulted in a larger reduction in the random noise generated in the switch. A very good differential-temperature profile was obtained when the modified switch was operated in a mockup of the scanning system. Development of an alarm switch for use with the scanning system was initiated.

A prototype single-point level probe operated satisfactorily in MSRE salt for two months. After the probe was removed, an even deposit of green material was found adhering to the portion of the probe which had been above salt level, but otherwise there was no deterioration. A full-size model of the probe which will be used in the MSRE is being constructed and will be tested.

Work is continuing on the development of a float-type continuous-level transmitter for measurement of molten-salt level in the MSRE fuel- and coolant-salt systems. Additional tests were performed on a transformer consisting of nickel windings on a Lava core. Except for some initial difficulties with a Fiberfrax insulation used in the transformer assembly, this transformer has operated satisfactorily for extended periods at various temperatures up to the Curie point of the iron core (1400°F). Span and zero shift were negligible over the range 700 to 1400°F. Below 700°F the span is strongly affected by temperature. Tests were also performed on a transformer wound with Inconel wire. Although this transformer has not suffered mechanical damage from heating, it has exhibited an excessive zero and phase shift. Proper electrical balancing and phasing may eliminate this shift. A third transformer, constructed of Hastelloy C wire, wound on a quartz form, failed due to embrittlement of the Hastelloy wire when heated to 1200°F. A transformer of the first type (nickel wire on Lava form) was installed on the molten-salt-level test stand, and preparations are being made for testing of the complete level transmitter.

Investigations of methods of attaching Inconel-sheathed, mineral-insulated thermocouples to INOR-8 pipes and vessels are continuing. A side lug attachment, with an INOR-8 tab Heliarc-welded to the Inconel sheath and to the pipe, appears to be satisfactory. Future thermocouple tests will use this method of attachment. Preparations are being made for installation of thermocouples on MSRE engineering test loops, in order to test the proposed methods of installation, to determine the accuracy with which externally attached thermocouples will measure the salt temperature within the pipe, and to obtain data on long-term drift of thermocouple calibration.

Reactor Analysis. — Preliminary calculations were made to determine the temperature-coefficient effect of adding a non-1/v poison to the MSRE. Since the major contribution to the temperature coefficient is from increased leakage with increased temperature, little change in temperature coefficient results from poison addition. For example, rhodium has an average thermal absorption cross section that varies approximately as $(T_N)^{-0.32}$, where $T_N$ is the neutron temperature; the addition of 3% poison as uniformly distributed rhodium changes the graphite temperature coefficient of reactivity from $-5.8 \times 10^{-5}/°F$ to $-6.1 \times 10^{-5}/°F$.

Some minor modifications were made to the IBM 7090 program EQUIPOISE-3 (a two-group, two-dimensional diffusion theory calculation) in order to compute the neutron lifetime and the reactivity resulting from unit changes in certain group constants, by use of first-order perturbation theory. Also, checkout of the two-dimensional gamma-ray-heating program 2DGH for the IBM 7090 was completed.

REACTOR SAFETY

Release of Fission Products on Out-of-Pile Melting of Reactor Fuels. The release behavior of fission products resulting from the oxidation of trace-irradiated $UO_2$ has been discussed in a summary report (ORNL CF-60-12-14). Included in that report is a plot of fission product release as a function of temperature which shows a definite discontinuity in most of the release curves with a minimum at 900°C, where there is also a minimum in the curves for oxidation rate vs temperature.

Data from recent experiments with irradiated $UO_2$ (PWR type irradiated to a burnup of 1000 Mwd/ton) which was oxidized in air at temperatures ranging from 500 to 900°C are contained in the table below:

| Time (min) | Temp (°C) | Percent Released | | | | | |
|---|---|---|---|---|---|---|---|
| | | Xe-Kr | I | Te | Cs | Ru | Sr |
| 9 | 500 | 4.4 | 4.6 | 0.14 | 0.02 | 0.13 | < 0.001 |
| 11 | 600 | 6.6 | 3.4 | 0.03 | 0.004 | 0.33 | 0.003 |
| 12 | 700 | 8.5 | 9.4 | 0.1 | 0.01 | 0.63 | 0.001 |
| 15 | 800 | 10.5 | 9.1 | 0.05 | 0.016 | 5.2 | 0.0006 |
| 15 | 900 | 11.2 | 15.2 | 0.4 | 0.005 | 18.9 | 0.001 |

These irradiated specimens were exposed to air for only 15 min, as compared with 90 min for the trace-irradiated $UO_2$, because of the much greater oxidation rates observed with the higher-burnup material. The above-mentioned type of discontinuity in the data for release vs temperature is noticeable only in the iodine and tellurium results and occurs at 800°C rather than at 900°C. The iodine and ruthenium release values obtained in these experiments at 900°C were approximately twice those obtained with trace-irradiated $UO_2$ at the same temperature; at lower temperatures comparable results were obtained with both types of material under the conditions prevailing in these experiments.

Release of Fission Products on In-Pile Melting of Reactor Fuels. — The second experiment (25%-enriched $UO_2$ specimen) on the release of fission products on in-pile melting of reactor fuels has been performed. Experiments 3 and 4 (6.5%-enriched $UO_2$ specimens) are ready to be installed and operated. Radiochemical analysis of the first experiment (3%-enriched $UO_2$ specimen) is 90% complete (ORNL-3223, p 10).

In experiment 2 the sample cladding was melted off during the insertion to the irradiation position. The automatic liquid-nitrogen filling system made it possible to collect the continuous flow of fission gases released during the 24 hr in this position. Temperatures recorded during the insertion to the melting position indicated that the maximum temperature obtained in the furnace was approximately 3500°C.

Additional modifications are being made to the ORR in-pile facility. A tube furnace is being installed on the primary helium inlet line to allow

further purification of the helium sweep gas. Malfunction of the cooling-water shutoff valve at the bottom of the facility tube has made it necessary to replace the bottom unit of the facility tube.

The design of the F-3 facility, in which fuel specimens for future meltdown experiments will be preirradiated to obtain high concentrations of fission products, is 95% complete.

Deposition and Removal of Fission Products from Gases. – Investigation of methods for characterization of radioactive aerosols was continued. Most of the experimental work was concerned with measuring diffusion coefficients for particles transporting $I^{131}$. Diffusion cells of both rectangular and cylindrical configurations have been used. Activity profiles resulting from aerosol deposition on the surfaces of the diffusion cells are determined with a gamma spectrometer, and the data, which may represent a range of particle sizes, are analyzed by means of an IBM nonlinear regression code. The deposition profile for an aerosol of given diameter is governed by the corresponding diffusion coefficient, among other factors. Results thus far obtained appear promising; according to preliminary calculations, particles with diameters of ~0.3 μ and also ~40 A, along with molecular iodine, showed the expected behavior in the diffusion cells.

## MARITIME REACTORS PROGRAM

Physics Studies. – Additional lifetime calculations were made with the CANDLE code to investigate the effects of various fuel management schemes on the burnup of an initial two-zone core I (4.2 wt % $U^{235}$ inner zone, 4.6 wt % $U^{235}$ outer zone) in the NS "Savannah" reactor. The schemes examined were: (1) switch the inner and outer fuel zones at the end of core I burnup; (2) remove the burned inner zone, replacing it by the burned outer zone and inserting a fresh outer zone; and (3) leave burned core I fuel in place, but replace the stainless steel fuel container with a Zircaloy fuel container. Of the three fuel management schemes studied, the Zircaloy substitution (scheme 3) was found to be the most attractive for increasing reactivity lifetime.

Bioassay Program. – The ORNL Health Physics Division assisted the NS "Savannah" Medical Department in the development of a shipboard bioassay program. Experimental studies were carried out to determine the lowest detectable limits of a representative corrosion product ($Co^{58}$) in raw urine specimens. External body counting experiments using a man-simulating phantom indicated that about 0.1 μc of either $Fe^{59}$ or $Co^{60}$ or a combination of both can be detected in either spleen or lung. Tests in counting samples of serum from neutron-irradiated blood showed that a 2- by 2-in. NaI(Tl) crystal is much less satisfactory for detection of $Na^{24}$ than a 3- by 3-in. crystal. NS "Savannah" counting equipment was used in these experiments.

Power Reactor Fuel Processing: Solvent Extraction. – In laboratory tracer-level tests with synthetic Thorex solutions, at least 90% of the protactinium was extracted with the uranium and thorium, but only when the solution acidity was > 1 N. Decontamination from fission products having high neutron cross sections was to a factor of about 100.

An unirradiated sample of Pebble-Bed Reactor graphite-base fuel was leached with 90% $HNO_3$ containing fission products to synthesize an irradiated fuel solution. Less than 1% of the activity was adsorbed by the disintegrated graphite. During evaporation of the leach solution to remove excess acid, ruthenium volatilization increased as the concentration of the uranium in the pot increased, but totaled only about 1% of that in the solution. After evaporation the feed was diluted with water to 3 M $HNO_3$, and the uranium was extracted with 10% tributyl phosphate in Amsco. Decontamination factors were 5000, 1000, 600, and $10^5$, respectively, from gross gamma, ruthenium, zirconium-niobium, and rare-earth elements.

The addition of 0.03 M fluoride to an $NH_4NO_3$ scrub solution increased the removal of thorium from a synthetic $U^{233}$ product solution to the limits of analytical detection. With an $Al(NO_3)_3$ scrub, fluoride addition was less effective, but precise thorium values cannot be determined at these low levels.

Power Reactor Fuel Processing: Zircex Process. — In the Zircex process, in which uranium-zirconium fuel is hydrochlorinated at ~500°C, absorption of volatilized $ZrCl_4$ in boiling aqueous solutions is being considered as an alternative to desublimation because of the difficulties in operating a solids condenser remotely and in preventing plugging of the reactor vessel by sublimation of $ZrCl_4$ at points cooler than 300°C. Investigation of the melting and boiling points of constant-boiling hydrochloric acid—zirconium solutions indicates that zirconium concentrations > 4 M can be achieved. At 4 M zirconium, the solution boiling point is about 113°C and the melting point about 105°C. Zirconium tetrachloride hydrolyzes in the vapor phase, without condensation, and does not cause plugging during transition from the off-gas temperature (about 400°C) to the boiling-solution temperature. In the boiling solution, zirconium forms the soluble compounds $ZrOCl_2 \cdot HCl \cdot nH_2O$, which solidify at about 105°C. The absorption vessel must be equipped with a condenser-rectifier designed to permit withdrawal of cold water-saturated hydrochloric acid, but hydrogen off-gas would pass through. Zirconium waste may be transferred to waste tanks as solution in heated lines. Preliminary calculations, on a 3-ft-diam sphere of solid waste, indicate a 10°C temperature rise in the center of the sphere over ambient temperature due to fission product heat.

Power Reactor Fuel Processing: Graphite-Base Fuels. — Fuel specimens of $UO_2$-graphite, containing from 0.7 to 12% uranium, and up to 0.45% iron as impurity, were burned in oxygen at 750 to 850°C, and the oxide residue was digested 4 hr with boiling 10 M $HNO_3$. The uranium loss to the nitric acid—insoluble residue decreased from 2 to 0.002% as the U/Fe weight ratio in a specimen increased from 5 to 45.

In the reaction of type GBF graphite with concentrated $HNO_3$, the mixture of water-soluble organic acids produced after 211 hr of digestion contained 50% mellitic acid (benzenehexacarboxylic acid). Infrared analysis of the mixture confirmed the presence of the carboxyl groups and showed only a small amount of C-H bonding, as would be expected if the mixture consisted primarily of benzene polycarboxylic acids. No evidence for the existence of nitro groups in the acids was obtained.

An irradiated (nvt = $10^{15}$) graphite compact containing about 4% uranium as pyrolytic-carbon-coated uranium carbide particles was leached three times, each time for 6 hr, with boiling 90% $HNO_3$, to determine the

integrity of the coatings. Although the matrix was completely disintegrated, a total of only about 1.5% of the uranium was solubilized, indicating that nearly all the fuel particles remained intact during irradiation. The cylindrical specimen was 1 in. in diameter and 1.5 in. high, and 3.5 ml of acid per gram of specimen was used.

Power Reactor Fuel Processing: Chop-Leach Process. — Scouting tests indicated that shearing into the flat side of a fuel assembly with a stepped blade rather than into a corner is unsatisfactory because of tube spreading and drag-back. A straight movable blade was unsatisfactory because chunks of tubes rather than discrete pieces were produced.

In a life test, a 4-in.-diam stainless steel bellows for a 4-in. flapper valve to be used as a seal for the shear-conveyor-leacher equipment failed at 35,400 cycles, equivalent to a life of about 4 yr. Test-stand evaluation of a duplicate bellows gave an acceptable life of 50,000 cycles.

To ensure that a batch of fuel is covered by the acid leachant in each flight of the leacher, the leacher must be reversed an amount equal to the angle of repose each time a batch of fuel is moved forward one flight. An angle of repose of 28° was measured for a 36-kg batch of chopped porcelain-filled ORNL mark I fuel assemblies and a 9.7-kg batch of solid 1/2-in.-diam stainless steel rods 1 in. long; an angle of 32° was measured for 2.5 kg of empty ORNL mark I sheared tubes while they were being transported through the inclined rotary leacher.

Power Reactor Fuel Processing: Beryllium- and BeO-Containing Fuels. — A simulated GCRE fuel element about 8 in. long, weighing 36.58 g, was digested with boiling 3 M $HNO_3$–4 M HCl for 9 hr. The Hastelloy X cladding was penetrated at the end of 3 hr, after which the $UO_2$ was rapidly leached from the $UO_2$-BeO pellets. Eighty percent of the Hastelloy X and 20% of the BeO were dissolved. About 0.2% of the uranium originally present in the BeO pellets remained undissolved and was not removed by washing with hot water.

Power Reactor Fuel Processing: SRE Fuel Dejacketing. — All SRE mechanical dejacketing equipment was removed from cell A of Building 3026 for decontamination and storage for possible future use. Decontamination of the cell by remotely spraying its interior surfaces with steam, water, detergent, and oxalic acid solutions and by using a vacuum cleaner for removal of dried particulates decreased contamination from a general cell reading of > 10 r/hr to ~80 mr/hr.

Metallurgical examination of the embrittled type 304 stainless steel fuel jackets showed evidence of hardening by cold working but no carbide precipitation or nitrogen absorption. A uranium–stainless steel eutectic was found which randomly covered very small areas of the internal surfaces of the jackets. The formation of this alloy accounts for some of the difficulties encountered in dislodging slugs from the jackets.

Power Reactor Fuel Processing: Corrosion. — Rolled and welded Haynes alloy No. 21, heat-treated after being welded at 2100°F and quenched, was corroded in boiling Darex dissolver solution at maximum rates of 2.35 and 2.03 mils/month for water- and air-quenched specimens respectively. These rates are about one-third those obtained on non-heat-treated specimens but are higher by a factor of ~2.5 than those obtained on the (apparently)

cold-rolled, unwelded material obtained from Battelle Memorial Institute. Some intergranular attack was observed in the heat-affected zone near the welds. Similar heat treatment of welded Haynes alloy No. 25 increased the corrosion rate from about 0.31 mil/month to 16 mils/month in initial Darex dissolver solution.

Titanium 45A, Corronel 230, and type 1100 aluminum were corroded at maximum rates of 0.18, 1.79, and 127 mils/month, respectively, in 24 hr exposure to boiling 20 M $HNO_3$. The titanium rate decreased to 0.07 mil/month in 168 hr exposure. The vapor-phase specimen of Corronel suffered intergranular attack after longer exposure.

Nichrome V exposed to $CCl_4$-$N_2$ at various temperatures between 550 and 725°C was corroded at rates which increased from about 2 mils/month at 550°C to about 1100 mils/month at 725°C.

Power Reactor Fuel Processing: Zirflex Process. — Four prototype PWR blanket rods, Zircaloy-2-clad $UO_2$, irradiated from 191 to 356 Mwd/ton and decayed 2 yr, were declad with boiling 6 M $NH_4F$-1 M $NH_4NO_3$. The final F/Zr mole ratio in the solution was 7, and the average decladding time was 1.5 hr. The end-cap residues were ~5 g per pin. Maximum losses of uranium and plutonium to the decladding solution were 0.04 and 0.22% respectively. The core pellets in the rods were largely shattered, with < 1% smaller than 10 mesh.

Core dissolution was complete in 5 M $HNO_3$ in ~40 min, yielding a solvent-extraction feed solution containing < 4 M $HNO_3$ and 100 g of U per liter. The solid residue from decladding and core dissolution was < 0.001% of the initial core weight and consisted of traces of Ca, Fe, Cr, and Sn; U and Pu were not detected.

Radioactive Waste Treatment and Disposal: Disposal in Deep Wells. — Test drilling at the site of the second fracturing experiment is now nearly completed, and the lateral extent of the grout sheets appears to be sufficiently defined. However, the well presently being drilled is encountering the several key horizons and the upper grout sheet at depths so much shallower than did the two immediately adjacent wells that at least one and perhaps all of these holes must depart considerably from the vertical. Until these and the other test holes are surveyed, the conclusion that the injection well was, by chance, drilled into a down-folded or down-faulted block is unwarranted. It now appears that both the structure of the rock and the shape of the two grout sheets are much more regular than at first believed.

A study by the MIT Practice School suggests that the safe limit for disposal by hydraulic fracturing, if the waste mixture in place is not to overheat, is of the order of 2000 curies per gallon of waste six months old. The thermal problem, then, is not likely to place much restriction on the type of waste handled, although somewhat longer aging before disposal might be desirable.

A Laboratory subcontract to develop waste-cement-clay mixtures for disposal by hydrofracturing has been signed with Westco Research, a subsidiary of the Western Company of Fort Worth.

Radioactive Waste Treatment and Disposal: Disposal in Natural Salt Formations. — Samples of shale and recrystallized salt from the field-scale experiments were submitted to the Geochemistry and Petrology Branch,

U.S. Geological Survey, for identification. In the acid cavity, soda niter (sodium nitrate) constituted the wall deposits and halite (sodium chloride) the floor cover. Only quartz was identified in the shale partings. The neutralized-cavity walls were covered with halite crystals along with an unidentified material. A shale sample was also examined, with only quartz and a second unknown found. Clay minerals were not found in any of the shale samples.

A computer program has been written to determine gamma radiation dose in the floor, walls, and ceiling of rooms in which packaged solid wastes are buried in the floor. Results obtained for the case of 6-in.-diam cans containing 2-yr-cooled, pot-calcined, acid Purex waste spaced at 5-ft centers indicate that the total cumulative dose in salt at the center line between cans will be about $3 \times 10^6$ rads. The program is being modified to determine the total dose in salt immediately surrounding the cans. Salt near the cans will be irradiated to doses greater than $1 \times 10^8$ r, and some decrease in structural strength is anticipated; however, the major portion of the salt in the floor will not be affected.

Radioactive Waste Treatment and Disposal: Engineering, Economic, and Hazards Evaluation. — As a first step in estimating the cost of ultimate disposal of high-activity wastes in salt deposits, the salt-mine area requirements are being calculated. The minimum area required to dissipate the heat from a mixed fission product source depends on (1) the concentration and age of the fission products, (2) the maximum permissible temperatures in the waste and salt, (3) the heat dissipation characteristics of the waste and the salt and of the air (if any) between the waste and salt, and (4) the manner in which the waste is disposed of in the mine. The amounts and concentrations of fission products were taken as those corresponding to Purex and Thorex liquid or calcined solid wastes that might be produced by processing of 1500 metric tons/yr of 10,000-Mwd/metric ton uranium and 270 metric tons/yr of 20,000-Mwd/metric ton thorium. The maximum permissible temperatures were taken as 200°F for liquid disposal, and 1650°F in the waste and 400°F in the salt for calcined-solid disposal.

For liquid disposal on the floor of a salt mine the net area requirements dropped from 35 acres/yr to 10.5–5.2 acres/yr as the waste age in-increased from 0.3 yr to 3–30 yr, respectively, after reactor discharge. To obtain the gross area requirements, these numbers should probably be doubled, to allow for only 50% excavation of the mine area. The mine area requirement for calcined-solids disposal in racks above the floor was found to be a factor of 2.3 less than that for liquid disposal, the factor being not quite equal to a ratio of 2.6 between maximum permissible temperature rises for the two cases because of poorer heat dissipation by salt at higher temperatures. For this case the minimum age from reactor discharge to burial varied from 0.3 to 8 yr.

Radioactive Waste Treatment and Disposal: Clinch River Study. — Core samples of bottom sediments taken at ten equally spaced intervals along five cross sections, Clinch River mile (CRM) 4.7, 7.6, 11.9, 15.3, and 19.2, were submitted to the U.S. Geological Survey for mineralogic and exchange-capacity analyses. Results indicate that the vermiculite, kaolinite, and interstratified vermiculite-mica contents are about the same in the clay-size fraction of bottom sediments in the study reach. Quartz is the most abundant mineral in these sediments. The micaceous clay

content is greater downstream from CRM 15.3 than at this or the next upstream section. The exchangeable magnesium content and the determined exchange capacity of the bottom sediments are nearly constant through the study reach. The exchangeable calcium content is greatest near the mid-sections of the study reach; the total cations in excess of the determined capacity are probably caused by dissolution of calcium carbonate.

From studies of records of flow in the Clinch River and in White Oak Creek the median daily dilution factor for the period 1951–60 was found to be 570. The minimum daily and monthly dilution factors for recurrence intervals of 10 yr were found to be 5.6 and 68 respectively. The mean monthly dilution factors are generally less than the median factor from January through May.

Radioactive Waste Treatment and Disposal: Fundamental Studies of Minerals. – Four sulfide minerals, pyrite ($FeS_2$), chalcopyrite ($CuFeS_2$), chalcocite ($Cu_2S$), and stibnite ($Sb_2S_3$), were investigated for sorption of ruthenium. Sorption was quite high for all minerals when synthetic waste solution tagged with $Ru^{106}Cl_3$ was used, but sorption of ruthenium from the untreated seep solution of operating waste pits was low. Sorption was increased when $Na_2S_2O_4$ was used to reduce the ruthenium in an alkaline medium. Optimum removal of ruthenium from solution occurred in the pH range 6 to 11, where polymeric ruthenium dioxide is at a maximum.

# PHYSICAL RESEARCH PROGRAM

## PHYSICS AND MATHEMATICS RESEARCH

High-Energy Electronuclear Machines: Cyclotron Analogue II. – Reassembly of the Analogue in its permanent location was completed and experimental studies were resumed. This eight-sector, spiral-pole electron cyclotron models the beam dynamics of a large cyclotron (the $Mc^2$ Isochronous Cyclotron) which will accelerate protons to an energy nearly equivalent to their rest mass, 938 Mev. The installation of a single coil to approximately cancel the horizontal component of the earth's magnetic field and the use of a newer electron source greatly improved the operation. The maximum dee voltage for accelerating electrons to near maximum energy (445 kev) was reduced to 60 v rms. This indicates a maximum energy gain of 170 ev per turn; at least 2600 turns are achieved during acceleration. By changing the axial focusing frequency in discrete steps without changing the isochronism and radial focusing conditions, it was shown that the resonance previously identified as $\nu_r = 4/3$ is really the difference coupling resonance $\nu_r - \nu_z = 1$. Beam loss at this resonance is essentially eliminated when the horizontal component of the earth's field is canceled.

High-Energy Electronuclear Machines: The $Mc^2$ Isochronous Cyclotron. – As a result of the successful operation of Cyclotron Analogue II, the earlier design concepts for the large proton cyclotron are being reviewed. Designs for the facility, in particular the beam rooms, have undergone substantial modification. Present plans are for separate large areas with fixed shielding for protons, pions, and muons, and a small area for medical and biological studies. Preliminary estimates for the shielding thicknesses are 22 ft of ordinary concrete or the equivalent for the proton and medical areas, and 11 ft for the pion and muon facilities. The possible economic advantage of the use of earth shielding is being investigated. A 0.15-scale model of three of the eight magnet sectors is being designed. A review of the radio-frequency system is in progress; any reduction in cavity size would, of course, permit more efficient magnet design.

Basic Studies of Cryogenic Electromagnets. – Several long, 50- to 100-ft, samples of 10-mil Nb–25% Zr wire were tested in the High-Field Solenoid; they were wound in the form of small solenoids 1 in. long. A loose-wound sample carried ~20 amp at 0 and 50 kilogauss, with a broad maximum of 53 amp at 15 to 30 kilogauss. An epoxy-cast sample with interwound copper carried 70 amp at zero field; in the 20- to 50-kilogauss range the critical current decreased to values similar to those obtained with the close-wound epoxy sample. All samples carry ~10 amp at 60 kilogauss and 0 amp at 69 kilogauss. A short 33% Zr sample (10 ft, 20 mils) carried 4 amp at 80 kilogauss, 50 amp at 70 kilogauss, and about four times as much as the 10-mil sample below 70 kilogauss.

The 1800-ft Nb–25% Zr coil, previously reported, finally failed after repeated cycling – probably due to a series of high-voltage discharges from the midpoint of the inner coil to one of the coil frame members. For the 1800 ft of wire, the transition time, superconducting-to-"normal," was ~10 msec; occasionally, microsecond transitions occurred, and high-voltage (several kv) sparks were generated.

Scintillation Spectrometry and Instrument Development. — The "resolution" of the Brooks pulse-shape discrimination circuit [Nuclear Instr. & Methods 4, 151 (1959)] was measured for two phototubes which were essentially identical in all respects except photocathode response. One tube was a standard RCA 7264 with S-11 response; the other was a developmental-type RCA C-7273 with a multialkali photocathode having S-20 response. Appreciably better pulse-shape discrimination at low energies was obtained with the tube having S-20 response. Measurements were made of the emission spectra of stilbene and anthracene by use of a grating monochromator and the C-7273 as a detector. Relative spectra, uncorrected for monochromator and detector responses, were obtained for 2-mm-thick crystals under bombardment by alpha particles and gamma rays. The emission spectra of both stilbene and anthracene show differences for alpha relative to gamma irradiation. The effect, which is an increase in the emission in both the ultraviolet and the red region compared with that at the peak of the emission spectrum, is more pronounced with stilbene than with anthracene.

High-Voltage Experimental Program: Large Liquid Scintillator Pulse-Shape Calculations. — Monte Carlo codes for calculating pulse-height distributions in gamma-ray scintillation counters have been described in a recent publication by C. D. Zerby and H. S. Moran (see ORNL-3169). These codes served as a basis for a mockup of the 1.2-m liquid scintillator used in pulsed Van de Graaff neutron capture studies [Phys. Rev. 122, 182 (1961)]. By use of a new Monte Carlo code based on a constant-nuclear-temperature model, the single gamma energy results have been combined into capture cascades. Total escape ranges up to about 12%, depending chiefly on average multiplicity. The case of favored transitions to discrete excited states or to the ground state, as found in studies of gamma-ray distribution following thermal-neutron capture, has also been investigated.

The calculated pulse-height distribution for a range of binding energies and multiplicities shows very good agreement with the spectrum fractions found empirically [Phys. Rev. 122, 182 (1961)]. The results of detailed analysis of the experimental data indicate systematic changes in modes of capture cascades as a function of atomic weight.

High-Voltage Experimental Program: The $Si^{28}(\alpha,\alpha)Si^{28}$ Reaction. — Elastic scattering of alpha particles by $Si^{28}$ has been measured at bombarding energies from 3 to 5.3 Mev at center-of-mass scattering angles of 90, 125, 141, and 162°. Targets of $Si^{28}O_2$ about 12 kev thick for 3-Mev alphas were prepared by evaporation on 20-$\mu g/cm^2$ carbon foils. Scattered particles detected by surface-barrier counters underwent resonances at 3.90, 4.32, 4.45, and 4.72 Mev, corresponding to excitation energies in $S^{32}$ at 10.33, 10.70, 10.81, and 11.05 Mev respectively. The region covered in $S^{32}$ (9.5 to 11.6 Mev) contains some 33 known levels, of which about 19 can be formed by the $P^{31}(p,\alpha)Si^{28}$ reaction [Nuclear Phys. 14, 472 (1960)]. The absence of most of these states in the observation of elastic scattering is attributed to their small alpha widths. The resonance at 4.72 Mev does not correspond to any previously known level of $S^{32}$.

Multiparticle Medium-Energy Nuclear Physics: The 86-Inch Cyclotron. — The levels of $W^{183}$ were studied by conversion-electron spectroscopy with sources of electron-capturing $Re^{183}$ ($T_{1/2}$ = 68 days). The data obtained

are sufficiently improved to allow further comparison with the theoretical analysis carried out by Kerman [Kgl. Danske Videnskab. Selskab, Mat.-fys. Medd. 30, No. 15 (1955)] employing the data of Murray et al. [Phys. Rev. 97, 1007 (1955)] on the $\beta^-$ decay of $Ta^{183}$. Kerman proposed three intrinsic states with associated rotations and a strong rotational-particle-coupling between the ground ($1/2^-$ [510]) and the 209-kev ($3/2^-$ [512]) states. There is now some evidence for a rotational ($9/2^-$) state at 562.6 kev which belongs to the K = 3/2 band, and a similar state at 595.2 kev based on the $7/2^-$ [503] intrinsic state at 453.1 kev. The energies of both postulated ($9/2^-$) states are reasonable, since the predicted energy for the former state is 562.9 kev, and the value of the inertial parameter ($3\hbar^2/I$) for the latter is 94.7 kev, to be compared with 100 kev for $W^{182}$. The data allow determination of a number of M1/E2 mixing ratios which are in quite close agreement with theoretical ratios calculated by Kerman. A 5-sec isomeric state at 309.4 kev ($9/2^+$ or $11/2^+$) is observed in $Re^{183}$ decay which proceeds to spin states 7/2 and 5/2 of the K = 1/2 band by 102.4-kev (M2) and 210.3-kev (E3) radiations respectively. Recently, Gallagher and Nielsen [Nuclear Phys. 24, 422 (1961)] studied the isomer of $W^{183}$ which had been chemically separated from its parent, 5.2-day $Ta^{183}$.

Multiparticle Medium-Energy Nuclear Physics: Heavy Particles. – The 63-Inch Cyclotron was shut down on October 31. The Heavy-Ion Group moved to X-10, where it will use the tandem accelerator until the ORIC is finished. At present, apparatus is being designed and built for use on these two accelerators.

Cyclotron Operations. – Although operation of the 63-Inch Cyclotron was terminated as of October 31, tentative plans are to maintain the machine in operational standby for approximately a year. Maintenance personnel at Y-12 will maintain the utilities serving the machine, and periodically the cyclotron will be started to ensure that it is operational.

Responsibility for operation of the 86-Inch Cyclotron was transferred to the Isotopes Division on December 1. The Nuclear Physics Research Group will continue to use the machine part time until the ORIC is available.

The cyclotron operations personnel are now test-operating components of the Isochronous Cyclotron.

Electronuclear Machines: The Isochronous Cyclotron. – The cyclotron magnet, complete with the three valley coils, nine harmonic coils, and ten trimming coils per pole tip, is now fully installed. The 19 power supplies and regulators were installed and tested, and most of the magnetic-field measurements have been made. About 50 runs have been completed to determine the effect of various currents in each trimming coil at various levels of the main field.

The ion source with its complex machinery for remote control was completed, assembled, and pretested. Performance testing awaits availability of the magnet field. The dee was completed, installed on the dee stem, and mounted in the resonant structure; the dee and the liner were pretested and are ready for installation.

A prototype of the five automatic servomechanisms for tuning the rf system was designed, built on an rf amplifier, and tested. The operating parameters of the power amplifier–dee coupling network were measured over a range from 9 to 16 Mc/sec; the physical circuit was modified and is

being reinstalled. Power loss and tuning parameters were measured for the pi network to the grid of the power amplifier, with operation from 7.5 to 22.5 Mc at full power.

Stable Isotopes Development. – Closure of the collimating slot associated with the second filament of a double-filament M-16 source unit was effective in preventing preliminary activation of the second filament and plugging of the collimating slot. Innage time per run on hafnium and molybdenum has been increased from 60% to 100% by utilization of this technique.

Thermal diffusion columns for the enrichment of $C^{13}$ have been installed. The plant will use methane and will enrich the $C^{13}$ to > 85%.

Special Separations. – A second-pass separation using 24 g of gadolinium enriched to 85.8% $Gd^{156}$ yielded, through two recycles, a 2.435-g purified sample containing 99.82% $Gd^{156}$. Previously separated samples contained too much high-cross-section $Gd^{155}$ and $Gd^{157}$ to permit satisfactory measurement of the cross section of $Gd^{156}$.

Thirty-five grams of silver which had been used as a plating on uranium was processed in the calutron to enrich the $Ag^{108}$ formed during irradiation. A 21-mg sample was recovered as metallic silver in which the $Ag^{108}$ content was estimated to have been enriched from ~1 ppm to 100 ppm.

The recently adopted practice of chlorinating metallic oxides with carbon tetrachloride to produce charges within the calutron has improved process efficiency and increased the life of the calutron ion source. The practice is currently in use with lutetium and hafnium separations. It is not expected to be applicable to all elements, and in the case of molybdenum, chlorination of the sulfide is planned to achieve the same effect.

## CHEMISTRY RESEARCH

Thorium Oxide Studies. – Several different thoria powder and pellet preparations (50 g total) are being irradiated in $D_2O$ in the LITR at 260°C and a flux of $\sim 3 \times 10^{13}$ neutrons $cm^{-2}$ $sec^{-1}$; an autoclave especially designed for multiple-sample irradiations is being used. The preparations are separately contained in thin-walled stainless steel tubes but share a common gas phase, and in some cases a common liquid phase, through the sintered stainless steel filter plugs used for the tube closures. An oxygen overpressure of 290 psi at room temperature was provided.

In the first 185 days of a projected six to seven months of irradiation, 14% of the initial oxygen has been consumed, and 50 psi of radiolytic gas has been produced. The estimated total heat to be removed has risen from an original value of 800 w to 920 w owing to buildup of $U^{233}$.

Equipment Decontamination. – Carbon steel in helium at EGCR temperatures (about 1050°F) is very sensitive to oxygen contamination in the helium, quickly acquiring a blue oxide scale or color when exposed to small traces. Oxygen residues in the EGCR during startup, or subsequent air inleakage, may therefore impede decontamination because of the necessity of dissolving the oxide film. Laboratory-produced thin, blue oxide films were removed in 1 hr, with acceptable corrosion rates, by a boiling 10% solution of a proprietary inhibited phosphoric acid. Thicker films were

more resistant. Helium containing no oxygen was slightly reducing to the film at 1050°F, probably because of a trace of hydrogen.

Oxalate-acetate-peroxide preparations continued to give good results in the low-corrosion decontamination of several fission products baked onto carbon steel in helium. When the pH was lowered from 4.5 to 2.5, corrosion was accelerated by $H_2O_2$ up to 0.6 M and inhibited above 0.6 M. Decontamination by the lower pH solution at 60°C instead of 95°C continued to prove feasible, provided the $H_2O_2$ was increased to prevent corrosion.

Protactinium Chemistry. – Absorption spectra of 15 and 34 N $H_2SO_4$ solutions of protactinium were essentially identical, with a single, rather broad peak at 2250 A. In 5 N $H_2SO_4$ the spectrum was significantly different, with no peak but with increasing absorption down to the limit of measurement at about 1970 A.

Distribution coefficients (organic to aqueous) for amine extractions of protactinium from sulfuric acid showed a decrease in extraction as the acid concentration was increased from 5 N to 15 N. The distribution coefficients decreased from $5 \times 10^3$ to $\sim 10^2$ for Primene JM-T, from 300 to 4 for N-benzylheptadecylamine, and from 20 to 0.1 for Alamine 336. All amines were 0.1 M in diethylbenzene, and the protactinium concentration was 0.08 mg/ml in the sulfuric acid solutions before extraction.

Spectrophotometric Studies of Solutions at Elevated Temperatures and Pressures. – Design and drawings of the spectrophotometer and the associated auxiliary and control systems for study of solutions at high temperatures and pressures were completed, as well as specifications for the fabrication, testing, and installation of the system at ORNL. It is anticipated that with this system it will be possible to make spectral measurements up to the water critical point (~372°C) in both water and deuterium oxide systems.

Construction and installation of the miniature loop system and its related control instrumentation were completed. The loop was designed for use with an unmodified Cary model 14PM spectrophotometer to operate at pressures up to 200 psi and temperatures to 150°C.

In further work on mathematical resolution of spectral fine structure and complex overlapping absorption spectra, programs for the IBM 7090 computer are being prepared, and codes for the Oracle and the IBM 704 are being converted for operation on the IBM 7090 machine. An automatic digital data-output system was constructed and tested and is now operating in conjunction with the spectrophotometer. A preliminary discussion of the system has been written, which includes an IBM punched-card output of the spectrophotometric data with the wavelengths and absorbancy information punched in pairs. Data may be obtained at wavelength intervals as close as 1 A. The card decks of the decimal spectral data are obtained simultaneously with the strip-chart record.

Radiation Chemistry of Organic Moderators. – Purified biphenyl samples in evacuated Pyrex tubes were pyrolyzed for 48 hr at a controlled temperature of 490°C. The pyrolyzed material was separated on a vacuum line into three different boiling fractions: permanent gases, intermediate boilers, and high boilers, with the boiling point of biphenyl (256°C) as the dividing line between intermediate and high boilers.

A 6-ft column of Apiezon L (30% by weight) with 2% Carbowax 20 M was used for the analysis of the intermediate-boiling fraction. Only one

pyrolytic product was found with temperature programming between 75 and 200°C. The product had a retention time of 4.1 min, and by comparison with known compounds was identified as benzene.

In a similar manner a 12-ft column of lithium chloride (20% by weight) was used for the separation and identification of the components of the high-boiling fraction. With temperature programming between 200 and 350°C, o-terphenyl had a retention time of 10.8 min; m-terphenyl, 14.2 min; and p-terphenyl, 15.2 min. These three compounds comprise more than one-half the quantity of high boilers formed in this experiment. The five additional compounds present have not been definitely identified. They are all higher boiling than the terphenyls and are likely to be isomeric forms of quaterphenyl and quinquephenyl.

The results thus far indicate that the reaction mechanism for the pyrolysis of biphenyl must involve the rupture of the C-C bond between phenyl groups, resulting in the formation of benzene and polymeric materials.

Surface Studies: Chemical Development. — The first measurements of ionic adsorption on $ThO_2$ slurries (needed for the estimation of particle-particle repulsion) displayed unsatisfactory reproducibility. Very substantial improvements in the reproducibility have been achieved by using a commercially available paint shaker to provide efficient mixing in the equilibration steps in the preparation of the slurries and in the course of the adsorption measurements. By use of material whose preparation was described in ORNL-3190, a reproducibility of 5% was achieved between two runs over the pH range from 3 to 5, whereas previous runs made without the better shaking had shown differences of up to 50% in the measured fraction of the sites covered at a particular pH value.

Instrumentation for Analytical Chemistry Research. — The design and the fabrication of a new, high-sensitivity, high-resolution, scanning, recording, single-beam flame spectrophotometer have been completed. Wherever advantageous, unaltered commercial units were utilized. The spectral range from 2700 to 8700 A is covered in the first order so that the instrument may be used in the analysis of radioactive solutions of cesium. A chopped-light-beam system with a synchronous detector is utilized to provide a stable zero level with infrared-sensitive multiplier phototubes and to allow the use of the instrument for either emission or atomic absorption photometry. While the magnitude of the signal as a function of time is being recorded, so that abnormal excitation events will be recognized, the time integral of the signal over a fixed time interval may be accumulated by an analog computer and recorded; the latter readout option is particularly useful at very high sensitivity. The sensitivity of the instrument is such that a water solution containing less than 1 part/billion of lithium gives a detectable signal.

A direct-readout system has been designed for installation on the model Q-2005, automatic, electronic, controlled-potential coulometric titrators. The readout in volts is a power-of-10 multiple of the quantity titrated in grams. The system has been installed on several coulometric titrators, for which it is preferred over the readout circuitry of the previous design.

Radiation Chemistry. – Hydrogen atoms produced by radiolysis of aqueous solutions were found to oxidize ferrous ions in aerated sulfuric acid solutions in two different ways. The ratio of oxygen concentration to acid concentration appears to determine the proportion of hydrogen atoms oxidizing each way. This proportion is not affected by the ferrous ion concentration between $10^{-3}$ and $2 \times 10^{-4}$ M. These facts indicate that there is a competition between oxygen and acid for hydrogen atoms, with the formation of two different complexes, each of which oxidizes ferrous ions. It is postulated that the two oxidation paths are the following:

$$\text{I.}\quad H + O_2 \rightarrow HO_2$$
$$HO_2 + Fe(II) \rightarrow Fe(III) + HO_2^-$$

$$\text{II.}\quad H + H_3O^+ \rightarrow H_4O^+$$
$$H_4O^+ + Fe(II) \rightarrow Fe(III) + H_2 + H_2O$$

The ratio of the rate constants, $k_{H+O_2}/k_{H+H_3O^+}$, equal to 700,000 ± 6%, was constant between 1 and 17 M sulfuric acid.

Transuranium Elements. – Variables were investigated in the system 2-ethylhexylphenylphosphonic acid–2 M HCl, which is proposed for separation of californium and curium. Addition of 10% 2-ethylhexanol, a degradation product, decreased the distribution coefficients by a factor of 10 but did not change the californium-curium separation factor. Aliphatic diluents gave higher distribution coefficients than did aromatic, and extraction increased with alkyl branching on the benzene ring. Hydrochloric acid distribution coefficients over the range 1 to 10 M HCl were < 0.001. Californium and curium distribution coefficients were inversely proportional to the cube of the hydrogen ion concentration over the range 1 to 4 M HCl and were directly proportional to the sixth power of the hydrogen ion concentration over the range 6 to 10 M HCl. The californium-curium separation factor was about 100 over the range 1 to 4 M HCl and about 10 over the range 6 to 10 M HCl.

The extractability of various corrosion and fission products was determined for the 2-ethylhexylphenylphosphonic acid–2 M HCl system; $Fe^{3+}$, $Zr^{4+}$, and $Sn^{2+}$ were extracted with californium, while $Sr^{2+}$, $Cu^{2+}$, $Ru^{3+}$, $Ni^{2+}$, $Cr^{3+}$, $Co^{2+}$, $Mn^{2+}$, $Cd^{2+}$, and $Zn^{2+}$ remained in the aqueous phase. Stripping the californium with 6 M HCl removed only the $Sn^{2+}$, leaving $Fe^{3+}$ and $Zr^{4+}$ in the organic phase.

A corrosion study indicated that alpha activity at 2 w/liter had no direct effect on corrosion rates of Hastelloy B, Hastelloy C, titanium 45A, tantalum, Zircaloy-2, and glass in chloride solutions. However, secondary effects, from the $H_2O_2$ produced by radiolysis of water, were accelerated corrosion rates for Hastelloy B and C and much greater corrosion resistance of titanium 45A in 6 M HCl. Zircaloy-2 and tantalum showed no effect due to radiolysis.

Uranium-232 Preparation. – An ion exchange method for separating uranium and protactinium was tested on a small scale with 208 mg of $Pa^{231}$ irradiated as the oxide to $2.5 \times 10^{18}$ nvt and decayed 30 days. The oxide was dissolved in 8 M HCl–0.4 M HF to a protactinium concentration of

10 g/liter and passed through a 2-ml column of Dowex 1-X4 (50–100 mesh) resin. The loaded column was washed with nine displacement volumes of 8 M HCl–0.4 M HF. The combined feed and wash effluents contained most of the protactinium and less than 7% of the $U^{232}$. The $U^{232}$ product eluted from the resin with 0.5 M HCl contained 73 μg of $U^{232}$ and < 300 μg of protactinium. The $U^{232}$ in the protactinium product and the $Pa^{231}$ in the uranium product were below the limits of detection of the analytical method. The irradiated protactinium oxide had apparently sintered, so that dissolution required heating for several hours at 80°C. Instead of the 150 μg of $U^{232}$ calculated for the sample, only about 80 μg was found in the oxide solution. Additional irradiations are being made in a monitored flux to determine the reason for this discrepancy.

Solvent extraction of uranium from mixed HCl-HF solutions by either tributyl phosphate or trilaurylamine appears satisfactory for final uranium purification. Uranium and protactinium distribution coefficients between 40% TBP in Amsco and 8 M HCl–0.5 M HF were found to be ~50 and 0.003, and between 0.1 M trilaurylamine in diethylbenzene and 7.5 M HCl–0.9 M HF, 140 and 0.002 respectively.

Target Preparation. – Four-inch-diameter uranium targets prepared by a cathode sputtering technique were subjected to x-ray absorption analysis at ORGDP; less than 5% variation in thickness over the entire target area was revealed.

Very adherent deposits of uranium up to 300 μg/cm$^2$ have been obtained by electroplating uranium onto platinum and nickel backings. A thin overplate of nickel on the uranium deposit renders the source smearproof while reducing the total alpha count by only 3%.

A small high-vacuum system has been constructed for experimental use in developing methods for producing metallic rare-earth targets. A quartz vacuum oven is provided along with an electron-impact evaporation furnace. The equipment is being tested.

The electron-beam equipment in the 24-in. evaporator has now been used successfully in producing targets of $Fe^{54}$, $Fe^{57}$, $B^{10}$, and $B^{11}$ with target thicknesses varying from 5 to 200 μg/cm$^2$.

Improvements in rolling techniques have resulted in thin foils of Fe (1 mg/cm$^2$), Pt (3 mg/cm$^2$), Sn (2 mg/cm$^2$), and Ta (30 μin.).

Thin films of $Tc^{99}$ metal were evaporated onto both glass and platinum backings.

## METALLURGY AND MATERIALS RESEARCH

Research and Development on Pure Materials. – The previously described LiF crystals grown from the vapor phase have been found to contain less than 0.01 wt % magnesium (by emission spectroscopy). All faces of these crystals were shown to be of the 100 type by x-ray diffraction. Examination of the crystals with polarized light failed to disclose any strains.

An experiment in the growth of large crystals of $Li^7F$ from the melt is in progress in the modified Stockbarger furnace. Approximately 455 g of high-purity $Li^7F$ was dehydrated with anhydrous HF. From the cooled melt, 300 g of colorless, inclusion-free material was hand-picked under a

dry-helium atmosphere and loaded into the furnace capsule. The mechanical lowering of the melt through a temperature gradient at the melting point is now in progress.

The oxides $ThO_2$, $ZrO_2$, and $TiO_2$ form simple eutectic systems with the solvent $Na_2B_4O_7$ and have extensive primary phase fields within these systems. Consequently, tests have been made of these systems as sources for pure oxide crystals. Optically perfect crystals of $ThO_2$, $1 \times 1 \times 1$ mm, and platelets up to $1 \times 3 \times 3$ mm containing inclusions, have been grown. Monoclinic crystals of $ZrO_2$ were found in sizes up to $0.1 \times 1.5$ m, but dendritic growth of $ZrO_2$ was pronounced. Rutile ($TiO_2$) was produced in the colorless form, which is typical of the stoichiometric compound.

The oxides $UO_2$, $ThO_2$, $ZrO_2$ (monoclinic), $TiO_2$ (rutile), and MgO also form simple eutectic systems with the solvent LiF. Rutile grown from this solvent was blue (typical of an oxygen-deficient structure); MgO formed truncated octahedra showing 100 and 111 faces. The possibility of growing these oxides as larger crystals from a lithium fluoride solvent is attractive, since this material is available in a highly purified form which will not act as an impurity source and since the crystal growth does not seem to be complicated by the formation of intermediate compounds.

Deformation in Crystalline Solids. – Work has continued on the development of means for measuring that part of the deformation energy which remains stored in metals and alloys. The method depends upon measuring the work used for deformation and the heat liberated within the sample, the stored energy being the difference. The method for measuring the heat by utilizing it to vaporize a suitable fluid has been known for some time, but only recently have the necessary equipment and techniques been fully developed. This phase would now appear to be essentially complete, and progress is being made on measurements on a series of copper-zinc and nickel-aluminum alloys. The measurements have been made mostly at −30°C but are to be extended to other temperatures.

One interesting observation is that aluminum in nickel increases the stored energy approximately the same amount as the strength is increased. That is, the increased work required to deform an alloy over that required for pure nickel is retained as stored energy, and hence 40% of the work can be stored by an alloy as compared with about 10% for pure nickel. A possible interpretation is that the increased resistance experienced by the dislocations is due entirely to the small ordered regions which they must cut through.

Fundamental Investigation of Radiation Effects in Solids. – Electrical-property measurements on semiconducting materials can be utilized to determine the nature of radiation-induced defects. A model of interstitial atoms and lattice vacancies (Frenkel-type defects) can be applied to experimental results that were obtained by using (1) $Co^{60}$ photons, (2) thermal-neutron $(n,\gamma)$ recoil damage, and (3) cadmium-gamma-ray-induced defects in samples that were shielded against thermal neutrons with cadmium metal. A reasonable agreement between experiment and theory is obtained. It is of interest that the relative effects of (2) and (3) are comparable in semiconducting materials.

CONTROLLED THERMONUCLEAR RESEARCH

Plasma Physics and Arc Research. — A broader and basic experimental approach that may give a qualitative, quick indication of the relative stabilities of various magnetic arrangements appears to be possible with techniques recently developed. For example, a low-energy plasma (~50-ev ions) generated at the juncture of an electron beam and a directed gas jet (in a vacuum environment) can be swept along magnetic field lines to a collector, where an indication of quiescence or turbulence is given by the reproducibility or the lack of reproducibility of the time-analyzed pattern of the collection current. In this arrangement, with argon gas being used, turbulence is seen to set in at magnetic fields of 300 gauss or more in a mirror arrangement, 500 gauss in a uniform field, and not at all in the quadrupole cusp at such field strengths as have been applied.

DCX-1 Facility. — More measurements have been made comparing the buildup and decay rates of trapped protons. At the highest $I_{H_2} + \tau_{decay}$ (16 ma-sec) the decay signals of the neutral-particle detector were nearly exponential, and the rise signals were linear for a substantial portion of the buildup. By the present model the latter characteristic is predicted when the $I\tau$ product is near the critical value required for an exponentiating buildup of the trapped-proton density by dissociation of the $H_2^+$ on protons already trapped. However, the suspiciously low value of the $I\tau$ product at which these observations were made and the failure to observe other predictions of the model at lower $I\tau$ values suggest strongly that the model is inadequate.

The vacuum tank was disassembled and thoroughly cleaned, and a new high-vacuum liner is being installed.

DCX-2 Facility. — Measurements of the magnetic-field uniformity in the DCX-2 have been carried out and confirm the results of the calculations. In particular, with 12 kilogauss in the central region, a mirror ratio of 3.3, and the proper injection dip, the deviation from uniformity over a distance of 24 in. from the midplane was less than 2 gauss. The accelerator tube was operated at 600 kv and 50 ma beam current, and there was no evidence of significant beam deflection or PIG discharges as a result of the magnetic field being turned on.

Assembly of the magnetically shielded injector was delayed by difficulties in manufacture, but the injector should soon be available. The delay permitted necessary maintenance and various mechanical modifications to ensure simple operation.

Vacuum. — A pump specially designed to utilize the polyphenyl ether pump oils was constructed and assembled. Accelerated decomposition tests on the penta- and hexaphenyl ethers were completed; these oils have very much higher thermal stabilities than do Octoil-S, Octoil, and Convoil-20. An oil diffusion pump (Consolidated Vacuum Corporation type PMC 720) charged with the pentaphenyl ether has been under test for 35 days with no changes in operating conditions from those used with Octoil. Observations which can be reported are that (1) no significant difference exists in pumping speeds for air and (2) the conditioning time for the ether is somewhat less than for the Octoil and in the one system yielded a slightly lower base pressure.

Studies on Superconducting Electromagnets. — A superconducting solenoid was built by using 10,000 turns of fused-nylon-insulated Nb–25% Zr wire 11 mils in diameter and 3400 ft long. The coil has an inside diameter of 0.5 in., an outside diameter of 2.2 in., and a length of 1.8 in. The critical current was 18.5 amp at 4.2°K, producing a calculated and measured field of 42 kilogauss. The ratio of the actual critical field strength of the solenoid to that expected from the short-sample characteristic is relatively high, namely about 80% as compared with 70% in somewhat smaller solenoids. No essential improvement of field strength was observed when the temperature was lowered from 4.2°K to 1.5°K. A kind of conditioning effect occurs when the current is repeatedly raised from zero to quenching value and seems to depend on the performance of the joints. Experiments are being performed to analyze the voltage distribution in the coil for normal operation.

## BIOLOGY AND MEDICINE PROGRAM

### ENVIRONMENT RADIATION STUDIES

Terrestrial Ecology: Evaluation of Fission Product Distribution and Movement in White Oak Drainage System. — The amount of strontium detected at temporary water-sampling stations along White Oak Creek at miles 2.6 and 3.9 and at the tributary stream draining the northwest portion of the area, during the period April 20 to August 4, 1961, appears to vary with the discharge. This suggests that most of the strontium enters the stream as a result of ground- and surface-water leaching and not of sporadic releases or spills, etc. The percentage of sorbed strontium in the creek water varied from 0.5 to 2.9% at the northwest tributary station and from 1.1 to 17% at the station at White Oak Creek mile 2.6. There is a relation between the percentage sorbed and the concentration of solids in the water.

Approximately 560 μc of strontium was detected at the station at White Oak Creek mile 3.9 during the period June 2 to July 28, 1961. Thus an average of 1.1 μc per acre of watershed was contributed to the creek during this period. (The drainage area above this station does not contain any Laboratory facilities or waste disposal areas. Thus the activity detected at this station is the result of fallout.) Assuming this rate to be representative of the entire drainage system, the amount of strontium associated with fallout at the station at White Oak Creek mile 2.6 for the same period was 1435 μc, or 2.5% of the total detected. For the northwest tributary station, 697 μc or 4.4% of the total strontium passing that station was due to fallout.

Terrestrial Ecology: Mass of Arthropods in Forest Soils. — Investigations on the role of soil arthropods in the breakdown of leaf litter and release of radioisotopes require information on the normal densities, masses, and rates of activity of these animals. The biomass (weight per unit area) of the larger arthropods (exclusive of mites and collembola) in the forest floor was measured in pine and oak stands of the AEC-controlled reservation. Values (milligrams of dry weight per square meter)

were 229 ± 22.3 and 240 ± 57.9 for pine and oak stands respectively. These estimates are strikingly similar to biomass estimates of 200 to 300 $mg/m^2$ for insects feeding on above-ground vegetation on White Oak Lake bed. The above-ground community, however, contained about 100 arthropods per square meter, while the forest floor supported about 2000 arthropods per square meter (exclusive of mites and collembola), the difference being due to the smaller size of the forest-floor arthropods. Weights of the forest-floor litter (L, F, and H layers combined) were greater in the pine than in the oak forests (1700 $g/m^2$ vs 950 $g/m^2$, dry weight), owing to the more rapid incorporation of organic matter into soil in the oak forests.

Terrestrial Ecology: Soil Microbiology. – Effects of leaf species, forest type, and altitude on microbial floras at four seasons were highly significant and showed high correlations with differences in rates of breakdown between 14 stands in the southern Appalachian mountains. Such differences help explain varying rates of biological release of chemical elements and radioisotopes from organic matter in biogeochemical cycles. Leaves of five species placed in 2- by 2-dm nylon net bags in deciduous and evergreen forests on north and south slopes at three altitudes and collected at two or four seasons were used for plating serial dilutions of standardized washings of 5-mm leaf strips. In summer and autumn, bacteria dominated on rapidly decomposing mulberry ($17 \times 10^5$ to $12,600 \times 10^5$ vs $2 \times 10^5$ to $38 \times 10^5$ for white oak), but fungi dominated for oak ($1.4 \times 10^5$ to $79 \times 10^5$ vs $1.6 \times 10^5$ to $21 \times 10^5$ for mulberry), per gram of dry litter. Average counts decreased along with leaf decay rate from mulberry to sugar maple, white oak, shumard oak, and beech, and decreased along with temperature with increasing altitude (1040 and 1600 m in the Great Smoky Mountains), to 60% of values at Oak Ridge (260 m) for bacteria and to 16% for fungi, in spite of a moister climate in the Smokies.

Terrestrial Ecology: Clinch River Studies. – The specific activity of $Sr^{90}$ in clamshells collected from the Clinch and Tennessee Rivers has been used to describe the behavior of $Sr^{90}$ released from the Laboratory to the Tennessee River System. When $Sr^{90}$ is released from the Laboratory a specific activity is established with the stable strontium in river water. The specific activity of the water mass may be changed only by dilution of the contaminated water by uncontaminated water, since the concentration of stable strontium is virtually constant throughout the Tennessee River. Clams, which concentrate strontium up to 8500 times the concentration in the water, take up stable and radioactive atoms in the same proportion as they occur in the water.

The behavior of $Sr^{90}$ was determined by comparing the specific activity of $Sr^{90}$ in clamshells collected from locations 58, 164, and 489 river miles downstream from the release site with that of clams collected immediately downstream from the mouth of White Oak Creek. The specific activity of $Sr^{90}$ in the clams from the distant locations was equal to that expected on the basis of dilution of the contaminated water by uncontaminated water. Thus there was no significant loss of $Sr^{90}$ from the Tennessee River water, indicating that Sr and $Sr^{90}$ are in equilibrium with other components of the system such as organisms and bottom sediments.

Mollusks that deposit $CaCO_3$ may be used as indicators of the behavior of $Sr^{90}$ in aquatic environments. This study also suggests that low-level radioactive waste releases to the environment may be used as tracers to determine the fate of radionuclides released when the specific activity is determined in organisms which concentrate the isotopes released.

## ISOTOPE DEVELOPMENT PROGRAM

Neutron Activation Analysis Research. – An investigation into the use of (n,n′) reactions originating in a reactor flux of neutrons (in this instance, $6 \times 10^{13}$ neutrons $cm^{-2}$ $sec^{-1}$) for activation analysis has shown that yttrium in quantities as small as 1 μg can be determined by the reaction $Y^{89}(n,n')Y^{89m}$. This isomer decays with a half-life of 16.1 sec and emits gamma radiations having an energy of 0.915 Mev. This particular method has been used to determine the concentration of stable yttrium in fission product mixtures originating from the fission of thorium.

A similar method has been used to study the feasibility of applying the $Pb^{204}(n,n')Pb^{204m}$ reaction to the determination of stable lead by an activation analysis method. Lead-204m decays with a 68-min half-life and emits 0.374- and 0.91-Mev gamma radiations. At least 10 μg of lead can be determined by this method if a flux of $6 \times 10^{13}$ thermal neutrons $cm^{-2}$ $sec^{-1}$ is used.

A number of other stable elements can be identified by the use of (n,n′) reactions, and the investigation is now being extended to include the determination of stable Br, Rh, In, Ir, and Au. Technetium-99 can also be determined by use of this reaction and will be investigated.

Recovery of Fission Products. – The solvent-extraction flowsheet (ORNL-2993) for recovery of fission product strontium and mixed rare earths with di(2-ethylhexyl)phosphoric acid was successfully demonstrated in continuous, miniature mixer-settler equipment with Purex 1WW waste solution from Hanford. In the first cycle, > 99% of the strontium was recovered with decontamination factors of 20 from Cs, 20 from Zr-Nb, 180 from Ru, and $> 2 \times 10^3$ from Fe, a performance as good as or better than predicted on the basis of tracer runs. Approximately 90% of the total rare earths was extracted and ~90% of that extracted was stripped, for an overall recovery of ~80%. In the second cycle, ~99% of the total rare earths was extracted and separated from strontium. Strontium was recovered from the second-cycle raffinate in a third extraction cycle, yielding a product solution containing ~1 g of Sr, 1 g of Ni, and 20 g of Na per liter. Overall Sr recovery was ~98%, with overall decontamination factors (based on first-cycle feed) of $\geq 1.7 \times 10^3$ from rare earths, $\sim 10^5$ from Zr-Nb, $\sim 3 \times 10^3$ from Cs, and $10^4$ from Ru.

Extraction of nitric acid and iron sulfate from Purex 1WW solution with amines is being studied as a means of producing a feed solution highly amenable to strontium and cesium recovery by solvent extraction. In preliminary tests with a primary amine, ~90% of the nitrate and iron and most of the sulfate were removed from synthetic 1WW solution in a single contact.

In preliminary tests, sodium dinonyl naphthalene sulfonate in hexone and p-dodecylphenol in xylene selectively extracted cesium from simulated low-acid (formaldehyde-treated) waste, complexed with tartrate and adjusted to high pH's. p-Dodecylphenol also had separation factors > 15

from Na and other alkali metals. Both reagents had separation factors > 100 for Cs from Zr-Nb, Ru, and rare earths and > 20 for Cs from Fe and Al and were readily stripped with dilute nitric acid.

Radioisotope Research and Development. – Approximately 4 μc of the $Th^{229}$ daughter was separated from 9 g of $U^{233}$ by passing an 8 M HCl solution through Permutit-SK resin. Uranium was adsorbed onto the resin, while the thorium remained in the solution. This separation established a feasible method of thorium-daughter removal from uranium.

The procedure for preparing Decalso radioisotope standards has been modified to allow fusing of the activity on ordinary 1-in.-square ceramic tile pieces. A procedure for labeling these Decalso standard sources has been developed by using a combination of decalcomanias and die-casting of portions of the label in the original tiles.

A $Cs^{137}$ calorimeter was fabricated from depleted uranium. Preliminary results show an energy of 4.59 ± 0.05 w/kilocurie.

A container evaluation program has been started, and a number of casks have been tested for leaks (hydrostatic test at 200 psig), integrity of welds, water content inside the lead cavity, and general condition of the outside container.

Equipment for the $I^{131}$ cell has been repaired. The enclosure for glassware equipment was installed to ensure safety against effects of possible breakage of glass and resultant release of vaporized $I^{131}$.

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