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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

AUGUST 1961

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Oak Ridge, Tennessee
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U.S. ATOMIC ENERGY COMMISSION

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

AUGUST 1961

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

SPECIAL NUCLEAR MATERIALS PROGRAM

Dissolver Solution Analyses: Plutonium. — Conditions were established and were evaluated at 1- and 5-mg levels of plutonium content for a relatively simple, three-step, controlled-potential, coulometric procedure for determining Pu(III), (IV), and (VI), either separately or in mixtures of each other, on a single portion of a sample in 1 M $HClO_4$ solution containing a known amount of Fe(III). The first step involves the coulometric titration of Pu(III) to (IV) at a potential of +0.9 v vs the S.C.E. In the second step the Pu(IV) and (VI) are then reduced to Pu(III) by titration at +0.4 v, and the third step involves the reoxidation of Pu(III) to (IV) by titration at a potential of +0.9 v. The difference between the integrated currents of steps two and three is a measure of the Pu(VI) content, while step three yields the total plutonium content. The Pu(IV) content is then calculated from the relationship between these three entities, as follows: Pu(IV) = total Pu − [Pu(VI) + Pu(III)]. The relative standard deviation of each titration is good, about 0.4%, and the errors in determining 5 mg of plutonium in each of the three valence states are less than 1%. Measurement of the exact errors involved in this procedure is hindered by the difficulties involved in preparing stable standard solutions of plutonium in the three valence states.

In the processing and purification of plutonium solutions by anion exchange separation, the disproportionation of Pu(IV) can be serious because neither Pu(III) nor (VI), thus formed, are sorbed onto the resin column. Therefore, further chemical treatment and processing of the plutonium solution is required.

A spectrophotometric study is being undertaken to determine the extent and rate of disproportionation of Pu(IV) in a solution of nitric acid and aluminum nitrate at elevated temperatures. In order to apply the spectrophotometric method of analysis to solutions containing Pu(III), (IV), and (VI), it will be necessary to secure precise absorption data for each of the oxidation states under equilibrium conditions of acidity, temperature, and anionic concentration. The absorption spectra of Pu(III) were obtained for various concentrations of nitric acid at 25, 50, 75, and 95°C.

Dissolver Solution Analyses: Uranium. — Work was initiated on the separation and determination of uranium by extracting it into 0.1 M tri-n-octylphosphine oxide in cyclohexane and then stripping it back into an

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aqueous supporting electrolyte in which the uranium can be titrated coulometrically. The extraction is quantitative from 5 M HCl or 1 M HNO_3, and an interfering amount of organic material is not introduced into the supporting electrolyte. A number of potential stripping reagents have been investigated, the best of which are 0.1 M Na_2CO_3 and 0.5 M Na_3PO_4–0.5 M H_3PO_4. The method offers considerable promise for determining uranium in several types of dissolver solutions, particularly those containing stainless steel components. Some electrochemical studies have been made with boron carbide as an electrode, in which there is an indication that this electrode may prove useful in extending the potential range anodically in controlled-potential coulometric titrimetry. Copper(II) exhibits a well-defined polarographic wave at –0.09 v vs the S.C.E., whereas U(VI) shows an ill-defined wave. A 3/8-in. boron carbide rod used as a working electrode for coulometric titrations of U(VI) did not yield reproducible results. Better results were obtained with an Fe(II) $\rightleftharpoons$ Fe(III) system, but the titration was slow, probably due to inadequate electrode surface area.

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

Pebble-Bed Reactor Physics. – Physics calculations were started on a 5-Mw (thermal) Pebble-Bed Reactor Experiment. The configuration being examined is a cylindrical core, 4 ft high and 2-1/2 ft in diameter, filled with 1-1/2-in.-diam spheres. A graphite reflector 3 ft thick will be used on all sides. An initial estimate of the required composition of the fuel spheres is that each sphere will contain 47.9 g of carbon, 1.9 g of U^{235}, and 2.2 g of thorium. It is anticipated that the reactor will be controlled by B_4C rods in the reflector. It appears that sufficient reactivity control can be obtained by the use of four 3-in.-diam rods placed 5 in. outside the outer edge of the core.

EGCR Valve Tests. – Seat leakage data for the 6-in.-IPS hermetically sealed gate valve supplied by the Darling Valve Company were obtained by pressurizing the valve in the open position and then closing it and venting the downstream side. For upstream-side pressures of 100 to 1000 psig the seat leakage of helium ranged from 4.5×10^{-3} to 7.6×10^{-3} cm^3(STP)/sec; these values are to be compared with the EGCR specification of 4×10^{-3} cm^3(STP)/sec.

GCR-ORR Loop No. 2. – Installation of the main piping and the side-stream cleanup system is approximately 90% complete. The flange buffer-gas system has been installed, and the off-gas piping is approximately 80% complete. The 24-in. shielding plug is being constructed, and the transfer table and the test-plug carrier are being fabricated.

Calculations were made for determining the temperatures and pressures that will exist in the carrier during transfer of a fuel element. The fuel element was considered to have decayed 12 hr before insertion into the carrier. It will reach a relatively high temperature of 2100°F, while the metal baffle surrounding it will be at a maximum of 1370°F. The total pressure rise in the middle chamber of the carrier will be 2.5 psia.

An analysis was made for determining the pressure-relief capacity of the annulus between the 24-in. plug and the test plug. It was concluded that the capacity was sufficient to relieve the nose region in the event of a rupture of the main loop pressure shell.

GCR-ORR Loop No. 1. – All equipment and instrumentation in newly reconstructed loop No. 1 have performed as designed. Pressure-vessel-steel weld-material samples are being irradiated, and the second seven-week test period is nearing completion. The maximum specimen temperature has been held at 555°F, and the maximum temperature difference from top to bottom of the specimen stack has been only 50 to 60°F.

The instrumentation necessary for fuel tests in the rebuilt loop is complete, except for the nitrogen sweep zone and the helium leak-detection system. The secondary container around the compressor piping is completed, but the leakage rate is not yet satisfactory. The rate at present is ~10 cfm at a pressure of 0.85 psig. The target leakage rate is 1.14 cfm at this pressure for a very conservative dose to the thyroid. The shielding around the surge tank and duct for the container is ~40% complete and should be finished by the end of August. Two fuel specimens, one of which will be inserted in the loop for the next reactor cycle, are 75 and 40% complete respectively.

Coated-Particle Fuel Element Studies. – Cylindrical graphite pellets, 1/4 in. in diameter and 1/4 in. long, fueled with coated UC_2 particles of normal enrichment, were carried through the complete preirradiation evaluation procedure. The pellets selected for evaluation were considered to be representative of those fabricated by Minnesota Mining and Manufacturing Company for the ORR-CP-poolside experiment. The pellets were alpha-counted, leached in 8 M HNO_3, alpha-counted a second time, thermally cycled three times to 1450 to 1500°C, leached a second time, and finally alpha-counted a third time. The alpha counts in all cases were essentially those corresponding to background and remained unchanged throughout the evaluation. Based on chemical analyses of the leach solutions, the average amount of uranium removed in the first leach was 3×10^{-3}% of the total uranium in each pellet, and the amount of uranium leached after thermal cycling was 3×10^{-4}%. These values are based on the fuel loading of 0.178 g of U per cm^3 requested in the purchase order. The actual fuel loading is being determined. The leaching results indicate that the samples tested do not contain any cracked particles, since the amount of uranium detected was less than that in a particle. Neutron-activation tests have presented contradictory evidence, however, and therefore the effectiveness of the leaching procedure is questionable. Preliminary results of the neutron activation indicate that a few of the particle coatings were cracked during fabrication. Heating the pellets to 1300 and 1500°C for periods of a day did not cause additional cracking of the coatings, but cracking did occur when the pellets were subsequently held at 2100°C for 6 hr or cycled between room temperature and 2100°C.

Two fueled graphite specimens similar to those scheduled for irradiation in helium sweep capsules at the MTR were subjected to neutron-activation and fission-gas-release tests. The fuel was present as 200-μ UC_2 particles coated with pyrolytic carbon. Previous data indicated that such pyrolytic-carbon-coated UC_2 particles would retain fission gases at temperatures up to about 2100°C. However, these specimens released about 0.5×10^{-4} of the Xe^{133} present when heated 16 hr at 600°C. Additional

heating, including about 20 hr at 1000°C, resulted in a total release of about 7×10^{-4} of the Xe^{133} in the specimens. These data indicate that the fueled graphite specimens contain significant amounts of fuel that is not protected by the pyrolytic carbon coatings.

The first static-capsule irradiation test of a coated-particle fuel element, L-CPlab, was completed on July 25, 1961. The initial specimen central temperatures were 1950 and 2120°F for the "a" and "b" specimens respectively. During the 2902 hr of irradiation, which resulted in a calculated burnup of 9.3 at. %, both thermocouples showed a gradual downward drift, and the final temperatures were 100 to 150°F lower than the initial temperatures.

The ORNL-MTR-48-5 irradiation test of UC_2-graphite elements is in the second of the four scheduled three-week MTR irradiation cycles. The fuel element is a dispersion of 175- to 250-μ spherical UC_2 particles coated with 80 to 100 μ of pyrolytic carbon in a graphite matrix. The element was tested in the fully retracted position for the first five days at a graphite bottle wall temperature of 1050°F, a fission power of ~1.85 kw, and a central temperature of ~1750°F. The element was then fully inserted to obtain a 1500°F graphite bottle wall temperature, ~2.75-kw fission power, and a 2500°F central temperature. Fission-gas escape rate data are being collected.

Liquid-Phase Sintering of UC. — Experiments have indicated that an essentially 100% UC body can be achieved by using approximately 2 wt % U_3Si_2 or USi_2 as a sintering aid. However, bodies containing 5 wt % silicide show discrete particles of silicide. Densities as high as 95% of theoretical have been achieved by sintering specimens containing 2 wt % U_3Si_2 at 1550°C.

Creep Properties of Type 304 Stainless Steel. — A close comparison of the creep curves for type 304 stainless steel in CO_2 and in air has revealed that considerations of minimum creep rate and rupture time can be very deceptive in the selection of the environment that will render the most beneficial strengthening effect. The differences in primary creep must be considered. Hence, a family of isochronal curves for a given percentage of strain as a function of temperature and stress best represents the data. The family of curves for 1% strain from times of 1 to 10,000 hr has been completed for air and CO_2 environments.

Drift of Thermocouples at High Temperatures. — Continued large negative drifts were noted for the type 304 stainless-steel-sheathed Chromel-P—Alumel thermocouples being tested at 870°C in a 7-psig hydrogen atmosphere. After 77 days the average error is −2.21 mv (−55°C). Similar thermocouples exposed to a CO atmosphere for 117 days have drifted slightly positive; the error is +0.25 mv (+6°C).

THORIUM UTILIZATION PROGRAM

Thoria Pellet Development. — The forming techniques described in ORNL-3120 were successfully used to prepare a 1-kg batch of high-density mixed ThO_2—1 wt % UO_2 spheres for use in attrition tests and in studies of uranium leaching by water at 260°C. During sintering, the rough spheres were first fired in air to 600°C to remove the organic binder added as a pressing aid and then to 1350°C in H_2. After being polished by wet tumbling, the

spheres were heated to 1800°C in H_2 to produce pellets with a bulk density of 9.45 g/cm^3. Attrition tests have shown that these mixed-oxide pellets wear no more rapidly than do pellets of pure ThO_2.

Attrition tests of several batches of pure ThO_2 pellets prepared as part of a 50-lb lot of pellets for loop and fluidized-bed studies have shown variations of pellet quality. Attrition rates ranging from 0.35%/hr to 1.4%/hr have been measured for fairly good pellets, while rates as high as 4.8%/hr have been observed for obviously defective pellets. These wide variations reflect difficulties experienced during fabrication and stem from variations in the ThO_2 powder used as a starting material.

Fuel Cycle Development: Chemistry of Solid Fuel Element Materials. — Nine ThO_2-UO_{2+x} samples, prepared for oxygen dissociation pressure studies, were examined to determine the identity and number of phases present. Compositions included the range 10 to 90 mole % of ThO_2 at 10 mole % intervals. The samples were prepared by coprecipitating the mixed oxides from aqueous nitrate solutions with NH_4OH and sintering in air at 1500°C. The results of x-ray diffraction analyses and microscopic examinations of polished sections showed that each sample consisted entirely of a single fluorite cubic phase. The absence of a second phase in these samples indicates that at 1500°C homogeneous ThO_2-UO_2 solid solutions formed in coprecipitation processes are highly resistant to oxidation in air.

Fuel Cycle Development: Fuel Preparation. — The development of the sol-gel process for preparing UO_2-ThO_2 particles of high particle density and compactability was continued. Thorium nitrate was reproducibly and controllably steam-denitrated in an agitated trough calciner to give 5-kg batches of oxide products with residual N/Th atom ratios of 0.05 to 0.066. After >98 wt % dispersion of these oxides to 2 M ThO_2 sols by addition of dilute nitric acid up to N/Th ratios of ~0.11, drying to gels, prefiring to 500°C, and air-sintering at 1200°C, fragments had toluene densities of 9.8 to 9.9 g/cc. Crushing in a pulverizer and sieving gave mixtures which were vibrated to bulk densities of 8.2 g/cc, but material tumbled in a ball mill was compacted to densities >8.5 g/cc. Calcination of the oxide from the trough calciner at 700°C for 16 hr before applying the sol-gel process gave particles with toluene densities of >9.9 g/cc; the particles were vibrated to 8.8 g/cc after ball-milling and sieving. Two-kilogram batches containing 2.5 and 3.9 wt % of fully enriched uranium were prepared for irradiation tests. Sol-gel thoria fragments precalcined to 500°C were thermally fritted from a U.S. standard mesh size of >10 to <200 by quenching in cold water. The product was free-flowing, did not contaminate surroundings by spattering, and did not clinker on sintering at 1200°C.

Thermogravimetric studies of steam denitration of $Th(NO_3)_4 \cdot 5H_2O$ at 25 to 425°C showed three successive reaction regimes. Stability regions were shown at 300 to 320°C and at 425 to 450°C, where N/Th atom ratios were ~0.3 and 0.05 respectively. Between 180 and 320°C the denitration was first order with respect to the mole fraction of residual nitrogen, and the activation energy was ~1.25 kcal per mole of ThO_2.

Eight fuel pins of vibratorily compacted oxide prepared by the sol-gel process have been irradiated for two months in the NRX reactor with satisfactory performance.

Studies on adaptation of the sol-gel process to the preparation of rounded attrition-resistant 1/8- to 1/4-in.-diam ThO_2 particles were continued.

Four samples of sol-gel material were prepared from ThO_2 made by steam denitration of thorium nitrate at 400°C and were tested for attrition resistance by tumbling in water for 20 hr. The particles were initially 58% >4 mesh and 83 to 95% >10 mesh. Tumbling decreased the percentage of particles >4 mesh to 23%, reduced the fraction >10 mesh by 19%, and produced 0.25% fines <200 mesh.

Eight samples of sol-gel material were prepared from ThO_2 made by calcination of thorium oxalate in which the particle size of the oxalate was varied from 1 to 10 μ and the precipitate digestion time was varied from 0 to 4 hr. The ThO_2 for all samples was fired at 900°C for 4 hr prior to sol-gel processing. No differences were observed in brittleness or attrition resistance when the samples were tumbled in water. The fraction of >4 mesh particles was reduced by about 10% from 50%; the fraction >10 mesh, initially 83 to 95%, was reduced by 4 to 10%; 0.75 to 1.83% of fines <200 mesh was produced.

The attrition rates for both types of preparations exceeded the maximum acceptable rate of 0.5%/hr. Comparison of the nitrate-derived and the oxalate-derived sol-gel preparations indicates that for the former there is greater brittleness, greater breakage of large particles, but more attrition resistance and less production of fines.

MOLTEN-SALT REACTOR PROGRAM

MSRE Design. — The package of specifications for reactor system components was released to commercial suppliers for bids. The cost of fabrication of all components is also being estimated by the Y-12 shops.

Reinforcing of all penetrations into the reactor containment cell was found to be necessary, and designs of these reinforcing rings were included in the construction-package addendum.

Details for the fuel-pump support and for the reactor thermal shield were submitted for comment. All structural-support drawings and auxiliary-pump-piping layout drawings were completed.

Initial versions of flowsheets were completed, and revisions are being made as required.

Drawings for the electrical heater, power circuits, and cable tray were submitted for comment. Thermocouple drawings are being revised prior to final approval.

Sampler and enricher drawings were reviewed, and final drafting was completed on some portions.

Design was completed for the positioning jig and tools for the sampler-enricher spool-piece replacement.

Design was completed for the tools for remote brazing of 1-1/2-in. pipe joints. Tools were provided for remotely holding and cutting the pipe, taper-machining the cut end, assembling the joint, and holding the furnace for remote brazing operation.

MSRE Procurement and Construction. — Much of the INOR-8 plate, sheet, and rod has been fabricated by the Haynes-Stellite Company and is being inspected. Delivery is scheduled for early September.

Bids to furnish INOR-8 pipe, tubing, and fittings were reviewed and accepted. Details of contract conditions and the types of inspections to be performed are being worked out with the successful bidders.

One bid was received for furnishing the MSRE moderator graphite, completely machined. This bid is being reviewed.

A prebid conference was held with contractors interested in bidding on the lump-sum modifications to Building 7503. The conference brought about some changes in specifications, and the bid opening date was extended to September 6.

Some prospective fabricators of major components for the MSRE have expressed a desire to obtain samples of INOR-8 in order to become more familiar with its working properties before bidding. It has been agreed that qualified fabricators will be provided with sample quantities of INOR-8 plate, and the bid-opening date has been extended in order to give fabricators time to work the sample material.

Development of Components and Systems. — Construction of the loop piping of the full-scale hydraulic model of the core was completed. The pressure vessel was received from the fabricator and installation was started. The simulated graphite and internal structure are scheduled for delivery on September 15.

The engineering test loop has operated isothermally at 1100°F and 50 gpm for a total of 2500 hr with the MSRE coolant salt. Characteristics of the freeze valve and level indicator and the variations in oxide analysis were determined during 30 fill and drain cycles and 66 sample withdrawals.

Design was completed and fabrication began on an 8-in. INOR-8 container to accommodate full-size core graphite pieces in the circulating loop. A controlled-atmosphere glove box is being fabricated for use in loading and unloading the graphite test pieces.

Performance of a single-seal 1-1/2-in. INOR-8 solder freeze valve for the enricher-sampler failed to meet expectations during 22 thermal cycles. An alternative design using a double-sealing buffered gate valve was prepared. Procurement was initiated for major components of the sampler for evaluation. Testing has indicated that a hydrogen-fired thin-wall 3/4-in.-diam copper capsule is best for sample removal and handling.

A gold-plated Inconel ring-joint gas seal used with Inconel freeze flanges was thermal-cycled between 200 and 1300°F for 21 cycles; the maximum leakage rate, which disappeared after five cycles, was 2×10^{-8} std cc of helium per second.

Construction of equipment for the fuel-drain-tank cooler test was completed, and testing was begun with no salt in the tank. The tube showed no serious distortion after thermal shocking from 1400°F by introducing water.

Combination ring-joint—Flexitallic seal flanges were developed for use where grouping of several connections of similar services within one leak-detectable joint results in space-saving advantages. Satisfactory seals were demonstrated over a series of 250°F steam cycles with both oval and octagonal rings enclosing four 1/2-in. Flexitallic inserts. A method of holding the flanges together during bolt torqueing to give reproducible ring alignment was also demonstrated.

Design of the long-handled tools and in-cell equipment for remotely operating freeze flanges is virtually complete. In the maintenance mockup, the primary loop piping is being installed to study component replacement problems.

MSRE Pump Program. — The pump with the molten-salt-lubricated journal bearing has operated continuously for 11,700 hr with molten salt $LiF-BeF_2-UF_4$ (62-37-1 mole %) and has been started 82 times.

Weld repairs were made satisfactorily to INOR-8 castings of the volute for the prototype fuel pump by the Laboratory and also by the founder. The founder for the coolant-pump impeller and volute castings reported shrinkage and gas defects in the first pair of INOR-8 castings made. A second pair of castings reportedly have fewer shrinkage defects but still contain gas inclusions. A third attempt is being made with different core sand to reduce gas.

Fabrication of the prototype fuel pump has been resumed with the weld-repaired volute casting. Reactor-pump drawings are being subjected to project review prior to start of fabrication of the pump rotary elements.

Thermal analysis of the coolant-pump tank indicated no necessity for forced-convection cooling. Computations are being made for determining the necessity for variable flow control of the coolant for the fuel-pump tank.

Metallurgy. — Tests were conducted to establish a suitable braze material, joint design, and procedure for the remote brazing of joints in MSRE pipe.

The brazing alloy 82% Au—18% Ni was selected for this purpose because of its good mechanical properties and resistance to corrosion by fused fluoride salts. Shear tests of the joint at 1300°F resulted in minimum shear strengths for the braze of 12,500 psi. Microhardness measurements on specimens from 10,000-hr diffusion tests run at 1300°F indicated a diffusion layer of 2 mils.

A tapered tube-and-sleeve joint design was tentatively adopted because of its strength and ease of assembly and alignment, and induction heating was selected because of the rapid and controlled heating aspects and the ease of handling the coil remotely. A ceramic envelope was used to confine a high-purity argon purge, which was demonstrated as adequate for brazing.

Several cylindrical joints were brazed in the laboratory and were found to be satisfactory by metallographic and ultrasonic inspections. Techniques are being developed for remote ultrasonic inspection.

Instrumentation Development. — Development of a float-type level transmitter for measuring molten-salt levels in the fuel- and coolant-salt pump bowls is continuing. Fabrication of a loop for use in testing the prototype transmitter is nearing completion. Design and fabrication of the prototype transmitter are complete except for the differential transformers. Several transformers were designed and tested. An early design, which used ceramic-coated wire, failed repeatedly at temperatures below 1200°F. The present design, which uses bare nickel wire wound in spiral grooves on a lava form, shows promise of success. Except for occasional short shutdown periods for inspection, this transformer has been in operation for two weeks at temperatures above 1200°F. This period includes 24 hr of operation at 1500°F. There has been no evidence of insulation failure to date. Preliminary data indicate that the combined zero and range shift between 900 and 1300°F will not exceed 3%. Since Armco iron is used as the core material, the transformer becomes insensitive above the Curie points, approximately 1400°F.

Development of a single-point conductivity-type level element is continuing. The resistivity of molten $LiF-BeF_2-UF_4$ (62-37-1 mole %) was measured at various frequencies, and a conceptual design was made of a level probe for use in the MSRE storage tanks.

Reactor Analysis. — Checkout of the two-dimensional version of the IBM 7090 gamma-heating program Nightmare is estimated to be 90% complete, and a preliminary report on the new program is in preparation. Computing time for a problem of moderate size is 10 to 20 min.

Dose rates outside the reactor cell from individual sources within the reactor cell were estimated, assuming reactor operation at 10 Mw for one year; various cooling times following such operation were also considered. The primary radiation source during power operation was the thermal-neutron-capture gamma radiation from the iron in the reactor shield. The addition of about 1 ft of barytes concrete block on the inside of the instrument room reduced the dose rate in the room to about 1 mr/hr.

The gamma-ray dose rates above the reactor cell and directly over the reactor core were recalculated, considering the shield to consist of 3.5 ft of barytes concrete plus 3.5 ft of ordinary concrete. The revised dose rate above the solid shield during 10-Mw operation was about 15 mr/hr; with a 1/2-in. crack in the 3.5 ft of ordinary concrete, the dose rate above the shield was about 80 mr/hr.

STUDIES AND EVALUATIONS

Fuel Shipment Studies. — Differences between measured temperature rises and calculated values, based on radiant heat transfer, in electrically heated mock fuel elements in a horizontal simulated shipping carrier were $<15°C$ for fuel bundles containing up to 64 tubes and heat generation rates per tube of up to 0.0514 w/cm.

REACTOR SAFETY

Calculation of Fission Product Concentration. — Concentration of fission products in the fuel and in the coolant stream of a generalized reactor power plant employing unclad fuel material has been calculated by using actual fission product chains and wide ranges of assumed values of both escape rates from the fuel and removal rates from the coolant stream. Complete tables of data calculated with the IBM 7090 computer are now available. As experimental data on escape rates from fuel and deposition rates from the coolant become available, the calculated data may be employed to determine coolant stream concentrations, which would in turn furnish a basis for determining the degree of coolant purification required to maintain the circulating activity below a given level.

Release of Fission Products on In-Pile Melting of Reactor Fuels. — A brief description of the equipment for the in-pile fuel element meltdown experiment was given in a previous report (ORNL-3062, p 11). Also in a previous report (ORNL-3156, p 11) a description is given of the first run in which a stainless-steel-clad UO_2 fuel specimen (3% enriched) was subjected to meltdown conditions in the ORR. This experiment was removed from the reactor and transported to the hot cell for examination and preparation of samples for analysis.

During the meltdown operation the purge gas from the furnace was passed through particulate filters, an iodine trap, and a liquid-nitrogen-cooled charcoal trap where noble-gas fission products were collected. A radiochemical analysis of this gas yielded an amount of Xe^{133} (5.27-day half-life) approximately equal to the total amount of Xe^{133} generated in the

specimen. Radiochemical analysis of the fuel from the experiment will give further information on release of noble gases.

A second stainless-steel-clad UO_2 specimen (25% enriched) to be melted is being installed in the experimental equipment. This unit is about 75% completed and is being assembled in a sequence adaptable to hot-cell assembly of similar units which will contain preirradiated fuel specimens.

Two modifications are being made on the reactor facility: (1) the water piping and control valves which supply the hydraulic cylinder are being altered to permit more rapid insertion and retraction of the reactor furnace; (2) the gas exit line is being altered near the liquid-nitrogen charcoal trap so that off-gas samples can be taken during the meltdown operation.

For future experiments, fuel specimens with varying degrees of burnup will be required. Heat transfer calculations have been made, and a device is being designed for irradiation of the specimens.

FUNDAMENTAL HEAT TRANSFER AND FLUID DYNAMICS

Thermal Transient Studies. — An experimental investigation of turbulence in relation to surface-temperature transients is in progress. These transients, which may originate from instabilities in the hydrodynamic boundary layer and which occur when there is a flux of heat across a fluid-solid interface, are a potential source of thermal fatigue failure and accelerated corrosive attack of reactor heat exchange surfaces. It is estimated that surface amplitudes as large as ±7% of the film temperature difference might occur in aqueous systems; the corresponding maximum amplitudes in typical fused-salt and liquid alkali-metal systems are approximately ±12 and ±25% respectively.

Preliminary operation of a constant-heat water-flow system incorporating an electrically (dc) heated test section consisting of 10 ft of 2-in. stainless steel pipe has demonstrated the feasibility of measuring instantaneous surface temperatures by means of "gun-barrel" type thermocouples. The system has also been used to explore the operation of a constant-temperature hot-film anemometer to be used as a tool in the study of fluid turbulence.

Vortex Heat Transfer Studies. — The heat transfer characteristics of pure ethylene glycol in axial and swirl flow were studied under both heating and cooling conditions. The friction factors for swirl flow are in agreement with a previously developed generalized relation, and the nonboiling heat transfer coefficients for swirl flow could be correlated with the earlier water data. The axial-flow nonboiling heat transfer coefficients are best correlated by the relationship $N_{Nu} \propto N_{Re}^{0.98}$ rather than by the traditional relationship $N_{Nu} \propto N_{Re}^{0.80}$. The burnout data are adequately correlated by a newly developed additive prediction method which depends on the premise that the two modes of heat transfer — boiling and convection — contribute without mutual interaction to give the total heat flux at burnout. This method is being tested with available data. Thus far, the results of 498 tests have been compared with predicted values, and the results are encourgaging.

Glycol and water nucleate-boiling data obtained at ORNL have been correlated by using a Kutateladze type of plot. Pool boiling and axial- and swirl-flow forced-convection data fall about a line that is 51% higher than Kutateladze's suggested average.

Fundamental Hydrodynamics. — The first phase of a fundamental experimental investigation of fluid in turbulence has as its objective the development of a reliable method of Reynolds stress measurements suitable for liquid flows. To this end a relationship has been derived between the mean-square fluctuating current of a hot-wire anemometer and the six Reynolds stresses that does not employ the usual low-turbulence intensity approximation. The relatively simple result is a consequence of assuming proportionality between the wire-current reading and the perpendicular velocity component. The result is valid for instruments equipped with the proper linearizing element.

MARITIME REACTORS PROGRAM

NS "Savannah" Shielding Survey. — The assembly of equipment and instrumentation for surveying the NS "Savannah" shield was completed. The mobile shielding laboratory was modified in order to increase the rate at which dose measurements can be made. Existing instrument racks were rearranged and an extra rack was added to accommodate additional instrumentation which was installed. The survey will be conducted after the "Savannah" achieves full-power operation.

Irradiation Effects on Reactor-Vessel Steel. — Charpy impact specimens of type A212 grade B steel from "Savannah" reactor-vessel material were examined at the Naval Research Laboratory, Washington, D.C., after having been irradiated in the ORR pressurized-water loop. In one ORR cycle the specimens had received fast-neutron dosages in excess of that expected in the "Savannah" in 20 years. The temperature of irradiation was approximately the same as the "Savannah" operating temperature (~500°F). The data obtained from this accelerated test indicate shifts in the nil-ductility transition temperature of the specimens as follows:

Fast-Neutron (>1 Mev) Dose (neutrons/cm^2)	Transition Temperature Shift (°F)
4.5×10^{18}	90
1.1×10^{19}	115
2.6×10^{19}	190

Water Chemistry. — The behavior of corrosion-product activity in the ORR pressurized-water loop is being studied with the general objective of seeking means for reducing or controlling the activity. When a sample of loop water was passed through a micropore filter and a cellulose backing pad, an appreciable portion of the Mn^{56}, Fe^{59}, Co^{58}, and Co^{60} activity which had passed through the filter was retained by the backing pad. From this and further tests it was concluded that the activity was present in the (cooled) loop water as dissolved cations. Conductivity measurements indicated that the observed soluble activity of Fe^{59}, Co^{58}, and Co^{60} was present to the extent of 4, 3, and 2×10^3 dis sec^{-1} liter^{-1} respectively. Comparison of these values with the specific activity of Fe^{59}, Co^{58}, and Co^{60} found in samples of "crud" filtered from the loop water supports the view that the dissolved material is in exchange equilibrium with the waterborne and loosely adherent crud. Since such exchange could be a principal

mechanism of activity transport in pressurized-water systems, it is desirable to develop some sort of inorganic ion exchanger (presumably a metal oxide) for use at the operating temperature of the loop.

Fuel Irradiation Testing. — Experimental assembly 3 was removed from and assemblies 4 and 5 were installed in the pressurized-water loop on July 7, 1960. A technique was developed for providing wire neutron-flux monitors in each leg of the loop for each cycle. These monitors were incorporated in assemblies 5 and 6.

ARMY POWER REACTORS

Surveillance of Radiation Effects on the SM-1 Pressure Vessel. — Four test capsules were placed on the core support structure of the SM-1 pressure vessel prior to plant startup. Each of these capsules contained three subsize Izod impact bars made of type A212 grade B steel taken from material used in the pressure vessel. Six test notches were machined into each bar.

The SM-1 plant was started up April 8, 1957, and was shut down April 29, 1960, for core replacement after operating 16.4 Mwyr. During the core replacement operation, one of the surveillance capsules was removed and transferred to ORNL for testing.

The impact test bars were broken, along with three unirradiated control bars. Increases were found in the subsize Izod transition temperature as a result of irradiation. The temperature shifts were, starting with the test notch farthest from the reactor core, 36, 52, 77, 99, 138, and 118°F.

Examination of Core I Fuel Elements from SM-1. — Postirradiation examinations of fuel element S-79, which was exposed for 16.4 Mwyr in the SM-1 reactor, are nearly completed. A radiochemical burnup analysis indicated a peak fission burnup of approximately 47% of the U^{235} atoms. Metallographic examination of fuel plate specimens revealed numerous cracks in the type 304L stainless steel cladding. No cracks were seen in the fuel matrix. The cladding cracks, both intergranular and transgranular, are concentrated in the high-burnup regions of the element. Although the source of the stress that produced the cracks has not been determined, it appears that the incidence of cracks is a function of burnup.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Power Reactor Fuel Processing: Zirconium Dissolution in Titanium Equipment. — The utility of a Darex titanium dissolver would be greatly extended if it could be used to dissolve reactor fuels containing zirconium as well as stainless steel. Since fluoride ion is used in aqueous dissolution of zirconium-base fuels, inhibition of the attack of fluoride on titanium with strong nitric acid, chromate, and fluoboric acid, singly or in combination, is being studied. All systems studied to date are of doubtful value because of unsatisfactory dissolution or corrosion rates, precipitation, or a too sensitive control requirement on reagent composition.

The most promising reagent found is 16 $\underline{M}$ HNO_3–2.64 $\underline{M}$ F^-–0.025 $\underline{M}$ BF_4^-–1.4 $\underline{M}$ Zr. Zircaloy-2 dissolution rates were 3.3 mg cm^{-2} min^{-1}, and titanium corrosion rates were ~5 mils/month. The zirconium must be maintained at $\geq$1.4 $\underline{M}$ to minimize corrosion; titanium corrosion rates were 40 and 0.0

mils/month at loadings of 1.35 and 1.42 M zirconium respectively. Although dissolution and corrosion rates decreased with loading, the Zircaloy-2 dissolution rate was still 2.75 mg cm^{-2} min^{-1} at 1.47 M Zr. The raw product solution was stable hot or cold, and after dilution and with 0.01 M U added. No feed adjustment other than dilution with water appears necessary.

Power Reactor Fuel Processing: Uranium Carbide and Uranium-Graphite Fuels. — When a specimen of uranium monocarbide containing 20% uranium metal reacted with water at 80°C, the uranium metal impurity yielded 2 moles of hydrogen per mole of metal. About 1.65 millimoles of H_2 (35 vol %), 2.85 millimoles of methane (61 vol %), 0.41 millimole of carbon, and 1.10 millimoles of monatomic hydrogen as saturated C_2 to C_6 aliphatic hydrocarbons were produced per gram of sample. The original specimen contained 3.33 millimoles of carbon and 4.03 millimoles of uranium per gram.

In further tests with the 90% HNO_3 process for uranium-impregnated graphite fuels, the extent to which graphitized fuel specimens were disintegrated by nitric acid depended on the acid concentration, the uranium content of the specimen, and the digestion time. In a 4-hr digestion, the geometric mean particle size of powders produced by reaction of specimens containing 8% uranium decreased from 1000 to 150 μ when the nitric acid concentration was increased from 20 to 23.9 M. After a 1-hr digestion in boiling 21.6 M HNO_3, the mean particle sizes were 1500, 320, 275, and 190 μ for specimens containing 0, 0.7, 2.6, and 13% uranium respectively. Most of the particle size reduction occurred during the first 6 hr of contact; for example, with specimens containing 13% uranium, the mean size was reduced only from 130 to 80 μ by increasing the digestion time from 6 to 48 hr.

Uranium was recovered almost quantitatively by the 90% HNO_3 process, at 25°C, from Turret fuel specimens prepared by impregnating type AUC graphite with uranyl nitrate solution prior to baking. After two 4-hr leaches with 90% HNO_3 at 25°C, the uranium loss to the graphite residue was only 0.09%. More than 96% of the uranium was recovered in the first leach and subsequent water washes.

Power Reactor Fuel Processing: Dissolution of Niobium Carbide. — Niobium carbide (NbC), which may be used as a fuel coating, was virtually unattacked by boiling solutions of hydrofluoric acid, nitric acid, and sodium hydroxide in concentrations up to 10 M. However, dissolution was achieved in 1 M HNO_3–HF solutions, where the initial rate of dissolution increased from 2×10^{-3} to 20.3 mg g^{-1} min^{-1} as the HF concentration was increased from 0.1 to 10 M.

Power Reactor Fuel Processing: Mechanical. — Five SRE core I fuel clusters from the second carrier shipment were mechanically dejacketed. An average dejacketing rate of 4.8 kg of U per hr was attained, ignoring downtime for repairs, and an over-all rate of ~2.4 kg of U per hr was maintained including downtime. One cluster of seven fuel rods contained four rods that required tedious auxiliary dejacketing methods because of waxy, oxidized NaK and swollen slugs. One slug, originally 3/4 in. OD by 6 in. long, had grown 6.25% in length and from 1.47 to 5.2% in diameter.

Micrometer measurements of five jackets showed 7 to 20 mils expansion in diameter when the applied internal hydraulic pressure was 1500 psig. This amount of expansion should have freed the slugs so that they could be easily flushed out by hydraulic pressure. However, because of degradation of the NaK to a solid or waxy form, the slugs could be ejected only by the jackscrew or by cutting and slitting the jackets and then prying them off.

The NaK disposal system operated satisfactorily during reaction of ~3000 ml of NaK bonding agent.

The 250-ton prototype shear, including the feed mechanism and hydraulic (oil) system, was received from the Birdsboro Corporation and installed.

A 600-g batch of crushed UO_2-ThO_2 pellets (Davison Chemical Company) was 73% dissolved in 6.5 hr in 13 M HNO_3–0.04 M F^-–0.10 M Al^{3+} in the four-stage Pyrex model leacher.

Power Reactor Fuel Processing: Corrosion Studies. — In continued exposure of types 304L and 347 stainless steel in modified Zirflex solvent extraction solutions composed of 0.75 to 1.5 M HNO_3–0.6 M Al^{3+}–0.36 to 0.49 M Zr^{4+}–2.44 to 3.33 M F^- at 50°C, the over-all corrosion rates decreased with time. After 320 hr the corrosion rate of type 304L appeared linear at 0.1 to 0.2 mil/month, regardless of the test solution or the specimen position. The rate for type 347 also became linear at about 0.11 mil/month. Type 304L suffered grain boundary attack plus etching in localized areas, whereas type 347 was attacked preferentially in the heat-affected zone near the welds, in addition to a general surface-pickling attack.

Types 304L and 347 stainless steels were corroded at maximum rates of 0.01 mil/month during 1000 hr exposure at 35°C in simulated low-acid waste solution containing 2 M HNO_3, 1.82 M stainless steel, and 100 ppm Cl^-. No sign of localized attack was observed.

Haynes alloy No. 25, Nichrome V, and INOR-8 were exposed at 600°C to N_2, saturated at 25°C with CCl_4, in both the presence and absence of volatile $ZrCl_4$. In 24-hr exposures the maximum rates for the three alloys were 127.8, 174.2, and 96.6 mils/month, respectively, without $ZrCl_4$, as compared with 170.3, 109.9, and 90.2 mils/month with $ZrCl_4$ in the gas stream. Triplicate specimens of each alloy were used; the middle specimen corroded most rapidly in the absence of $ZrCl_4$, while the upstream specimens corroded most rapidly when $ZrCl_4$ was present.

Power Reactor Fuel Processing: Continuous Modified Zirflex Process. — Studies were continued to adapt the modified Zirflex process to the continuous dissolution of uranium-zirconium alloy fuels to obtain increased throughputs by continuous operation. In a 2-in.-diam dissolver the Zircaloy-2 dissolution rate increased nonlinearly with increasing F/S (feed rate/surface area) ratio, so that zirconium loadings were lower as the reaction rates increased. Solution loadings were $\geq$80 g/liter, with F/S $\leq$0.06 at reaction rates $\leq$5 mg cm^{-2} min^{-1}. In one run in a 6-in.-diam dissolver with 6.5 M NH_4F–0.6 M NH_4NO_3–0.1 M H_2O_2 as the dissolvent, steady-state operation for 1 hr resulted in an average reaction rate of 5.6 mg cm^{-2} min^{-1} at an F/S of 0.08. The effluent contained 70 g of Zircaloy per liter.

Power Reactor Fuel Processing: Solvent Extraction Studies. — In the TBP-25 first-cycle solvent extraction flowsheet, decontamination from both ruthenium and zirconium-niobium was higher when the feed was 0.15 N acid deficient rather than 1 N acid; decontamination from ruthenium was greater with 4 M HNO_3 scrub than with the normal 0.75 M $Al(NO_3)_3$ scrub. With both an acid-deficient feed and an acid scrub, fission product removal was below the level of analytical determination, indicating greater than threefold improvement in decontamination.

Power Reactor Fuel Processing: Dissolution of Beryllium and Beryllium Oxide Fuels. — In further studies on the dissolution rates of BeO-base fuel specimens in boiling fluoride solutions, the initial dissolution rates of sintered BeO–5% UO_2 in boiling 1 to 5 M HBF_4 were 0.02 to 0.21 mg cm^{-2} min^{-1}. In solutions containing 1 to 5 M HBF_4 and 5, 10, and 15 M HF, the initial dissolution rates were approximately 0.7, 1.0, and 1.5 mg cm^{-2} min^{-1} respectively. The rates were not markedly affected by HBF_4 concentration.

Radioactive Waste Treatment and Disposal: High-Activity Waste Calcination. — A cost study on pot calcination of high-activity waste from a 6-metric ton/day fuel processing plant indicated a range from 0.87×10^{-2} mill/kwhr (electrical) for acidic Purex and Thorex wastes in 24-in.-diam vessels to 5.0×10^{-2} mill/kwhr (electrical) for reacidified Purex and Thorex waste in 6-in.-diam vessels. About seven years of interim liquid storage would be required before acidic Purex waste could be processed in 24-in.-diam vessels.

Radioactive Waste Treatment and Disposal: Low-Activity Wastes. — In a third run in the 60-liter/hr scavenging—ion exchange pilot plant for ORNL low-activity waste, decontamination factors were still satisfactory after 1500 resin bed volumes of waste had been treated (e.g., Sr, >1000; total rare earths, 100; Cs, >100), and the effluent activity was <10% of the maximum permissible concentration.

Radioactive Waste Treatment and Disposal: Disposal in Deep Wells. — Test drilling at the site of the second fracturing experiment is nearly completed. The upper grout sheet, composed of four separate layers formed one above the other, extends out from the injection well some 500 ft to the east and northeast, but only short distances out in other directions. The lower grout sheet extends largely to the north and northwest, but its outer limits are still undefined.

A detailed analysis of the geologic structure of the area, which appears to have determined the directions in which the grout sheet moved, will have to wait until the deviation from vertical of the test holes has been measured; with holes 800 to 1000 ft deep, even a slight bend is significant.

The core hole at the proposed site of the Waste Disposal Fracturing Plant is now at about 2200 ft; the scheduled final depth of the hole is 3000 ft. Rock well suited to disposal operations has been encountered from 1500 ft to the present bottom of the hole.

Radioactive Waste Treatment and Disposal: Disposal in Natural Salt Formations. — Stress measurements in the pillars of the Hutchinson mine have shown the absence of horizontal stress and the presence of vertical stress varying from 1300 to 1600 psi. The absence of horizontal stress was expected; however, the theoretical stress in the pillars based on mine depth and per cent salt extraction is about 1950 psi.

Comparison of the data from the field cavities with theoretical calculations of temperature rises around spheres has indicated that the thermal conductivity of the salt surrounding the experimental cavities is 2.6 Btu hr^{-1} ft^{-1} $(°F)^{-1}$, and the diffusivity is 0.099 ft^2/hr. These values agree quite well with the Birch values for single pure-salt crystals at 80 and 65°C respectively.

A computer code has been designed for the solution of temperature rises in a heat-generating layer of infinite horizontal dimensions (flat

slab) and in the salt surrounding the layer, as a function of time for wastes of variable age. Use of this code has shown that the storage of waste cooled less than one year is wasteful of mine space and that the thickness of the slab (from 1 to 10 ft) has little effect on the temperature rise in the slab.

Studies on radiolytic gas production of wastes in salt have shown that gas production can be eliminated by pressurizing the cavity or by maintaining an atmosphere of hydrogen over the waste. The pressures involved are not extremely high (3 to 10 atm), but do represent a problem in cavity design. Disposal of liquids in salt as a mixed layer of salt and waste appears to eliminate the problems of radiolytic off-gas, as well as cavity alteration.

Radioactive Waste Treatment and Disposal: Clinch River Study. — A continuous water-sampling system was installed at Centers Ferry on the Clinch River. The instruments were so designed that the rate of sampling is proportional to velocity of flow in the river.

Results of sorption tests on membrane filters indicate that essentially no strontium, cobalt, and ruthenium are sorbed as river water samples are filtered. A small percentage of cesium (4%) is sorbed by the filters.

Various types of bottom-sediment core samplers have been tested and evaluated for compaction and retention characteristics. The most effective was a 3-1/2-in.-diam split-tube sampler with a basket shoe.

Results of a recent TVA silt range survey of Clinch River in the study reach have shown that additional cross-section sampling sections, intermediate to the standard silt ranges, do not significantly change estimates of sediment volume.

Distribution coefficients (K_d) were determined for several clay minerals, using cesium and strontium in tap water. Tap water was used, since the chemical composition simulates that of Clinch River water. The strontium K_d for illite was reduced from about 500 in demineralized water to 120 in tap water, and the cesium K_d from 80,000 to 50,000. The cesium K_d is indicative of the high selectivity illite exhibits for this element.

Radioactive Waste Treatment and Disposal: Fundamental Studies of Minerals. — Sodium vermiculite shows three distinct sorption-of-strontium responses in the pH range 2 to 11. Up to pH 4.5 the response depends on the displacement of aluminum from octahedral layers of the mineral. Thus, less than 10% sorption was observed at pH 2 and only 59% at pH 4.0; in both cases maximum sorption was observed after contact for less than 96 hr. Sorption increases uniformly (to approximately 70%) between pH 4.5 and 9.0 up to 96 hr of contact. At pH 11, sorption sharply increases to 91% at 96 hr.

Comparison of strontium removals by sodium vermiculite, clinoptilolite, Duolite C-3, and Dowex 50 showed that sodium vermiculite is ineffective at low pH but competitive at basic pH. Exchange capacity and stability of vermiculite at low pH are important considerations in strontium sorption reactions.

Particle diffusion processes have a pronounced influence on cesium breakthrough curves obtained from small columns operated for rather short periods of time. Extrapolation from bench-scale experiments to the field may require correction for this phenomenon, as the cesium loading increases with increased time of column operation. For prolonged times of column operation, the effects of particle diffusion are not so pronounced. The choice of particle size for an operating column may be determined, in part at least, by the kinetics of the exchange reaction.

PHYSICAL RESEARCH PROGRAM

PHYSICS AND MATHEMATICS RESEARCH

High-Energy Physics. — A necessary consequence of parity nonconservation in Λ^0 decay is a longitudinal polarization of the decay proton in the rest frame of the Λ^0. A sufficient number of K^- interactions in helium which yielded Λ^0 hyperons, whose decay protons scattered, have been measured to determine the sign of the helicity. Thirty events were found and analyzed at ORNL. These, together with 120 events analyzed at other laboratories, yield a negative asymmetry parameter.

High-Energy Electronuclear Machines. — Cyclotron Analogue II was first operated on August 4, 1961. The Analogue, an eight-sector AVF cyclotron, models approximately the beam dynamics of an 850-Mev proton cyclotron under study at ORNL. A series of energy measurements at various radii, based on pulse-height spectra of x rays produced by the electrons striking an aluminum target, are in approximate agreement with design calculations. The maximum electron energy so determined appears to be greater than 500 kev; the design energy for $\nu_r = 2$ is 440 kev, although isochronous fields are provided for acceleration to about an electron rest mass. The beam profile, visually observed on a phosphor-coated probe, shows evidence of the resonance at $\nu_r = 4/3$ and at the coupling resonance $\nu_r + \nu_z = 2$. In the initial test, the threshold voltage for acceleration to full energy was about 350 ev (per turn). It is expected that optimization of the average field coil currents and an improved electron source will reduce this threshold.

Basic Studies of Cryogenic Electromagnets. — The High-Field Solenoid was completed and was successfully operated at a power input up to 3.2 Mw. The field was calibrated at values to 55 kilogauss; extrapolation gave a field of 90 kilogauss at the 3.2-Mw input, the effective limit of the power supply. This copper solenoid has a usable central cavity approximately 1-7/8 by 10 in. and is water-cooled for continuous operation.

In the initial experiment with the High-Field Solenoid the superconductivity of Nb-Zr was examined at 4.2°K. The 0.020-in. Nb-Zr wire (25% Zr) carries in excess of 100 amp (5×10^4 amp/cm^2) for transverse fields below 45 kilogauss; the current drops rather rapidly to 1 amp at 72 kilogauss. These tests were made with a pulsed (0.001-sec) current in the wire.

Theoretical Physics. — Four problems have been coded for the IBM 7090 computer as part of a program of numerical studies of nuclear reaction theories. This is a cooperative effort with members of the Electronuclear Research Division, the Neutron Physics Division, and the Mathematics Panel.

First, a highly flexible code to calculate the distorted-wave theory of direct nuclear reactions in zero-range approximation, neglecting spin-orbit coupling, which was written for the IBM 704, has been running successfully on the IBM 7090 for some months. It is being used to interpret (p,d) and (He^3,n) reaction data obtained at ORNL and reaction data from other laboratories, as well as for purely theoretical studies of distorted-wave effects.

An extension of the above program, to calculate the distorted-wave theory with spin-orbit coupling effects included, has been largely rewritten to incorporate improvements in computation and to utilize the capacity of the IBM 7090. This code is now running.

The pilot program for the third problem, the strong-coupling direct-interaction theory of inelastic scattering, is in operation and considers coupling between two nuclear levels. Preliminary results indicate that the physical coupling is at least intermediate in strength for many excitations in even-even nuclei. Extensions to coupling between more than two channels are being prepared, in particular to investigate "double excitation."

The fourth problem, the optical model for elastic scattering with nonlocal (velocity-dependent) potentials, has been coded for a separable potential. Preliminary results indicate that such a nonlocal potential, with fixed parameters, is able to reproduce the energy dependence of the observed scattering, which otherwise requires a local potential with parameters that vary with energy. Spin-orbit coupling is included. A search program has also been written that automatically obtains parameters which give a best theoretical fit to given "experimental" points.

Also being coded is a program to calculate the distorted-wave theory without the zero-range approximation.

High-Voltage Experimental Program: Total Neutron Cross Section of Pb^{208}. – Measurements of the total neutron cross section of Pb^{208} (99.75%) with ~3 kev energy resolution were reported in the 1.0- to 1.9-Mev range [Bull. Am. Phys. Soc. 6, 251 (1961)]. These measurements have been extended to lower energies (~0.72 Mev) in order to overlap the results from Duke University (Bilpuch, private communication, April 12, 1961). For the ORNL measurements a stilbene crystal, in which pulses due to gamma rays were depressed by Brooks-Owen pulse-shape discrimination, was used as a neutron detector. The resonance at 0.724 Mev shows a peak cross section of ~13 barns, in agreement with Duke University data. Between 0.72 and 1.9 Mev, at least 60 resonances were observed with J values ranging from 1/2 to 7/2. In order to decide on the parity of the levels, angular distributions of the scattered neutrons are necessary. The apparatus for detailed differential cross-section measurements is being assembled.

High-Voltage Experimental Program: Decay of the Compound Nucleus U^{235*} by Fission and Neutron Emission After Formation by 1050-kev Neutrons on U^{234}. – The dip in the fission cross section of U^{234} at 1050 kev is believed to be due to competition from neutron emission, which leaves the residual nucleus in a band of excited levels lying between 788 and 984 kev. The drastic change in the angular distribution of fission fragments which occurs coincidentally with the dip in cross section can be explained if it can be shown that neutron emission from the higher J states of the compound nucleus competes more favorably with fission than does emission from the lower J states.

A modified Hauser-Feshbach calculation was carried out for neutrons incident on U^{234} with 1050 kev energy. The results show that neutron emission takes place more readily in general from the states of higher J than from those of lower J, thus lending credence to the above hypothesis. The

following tabulation, which shows the results of these considerations, is a summary of relative decay probabilities of the (J,π) states of the compound nucleus U^{235*} by neutron emission for an incoming neutron energy of 1050 kev. Note that P' represents the relative probability of decay. To find the absolute probability of decay, P' would need to be divided by the sum of itself plus the probability of decay via fission.

(J,π)	$1/2^-$	$3/2^-$	$5/2^-$	$7/2^-$	$1/2^+$	$3/2^+$	$5/2^+$
P'	1.14	1.58	1.06	1.04	1.26	1.65	1.54

In this table the excess of the relative neutron emission probability, P', from the compound state of (J,π) equal to $3/2^-$ over the emission probability from the $1/2^-$ is of most significance.

Further calculations are in progress, and these together with the details of the above considerations will be reported later.

Multiparticle Medium-Energy Nuclear Physics: The 86-Inch Cyclotron. – Radiations from the electron-capture decay of Eu^{150} indicate two isomeric states with half-lives of 14 hr and > 5 yr. The transition data were obtained from internal-conversion-electron and gamma-ray spectra. The energies and intensities of the transitions belonging to 14-hr Eu^{150} indicate excited states at 334.1 (2^+), 740.7 (0^+), 1153.1, and 1256.1 kev. Levels at 334.1 (2^+) and 773.3 (4^+) kev, as well as states of higher excitation, are populated by the decay of > 5-yr Eu^{150}.

Multiparticle Medium-Energy Nuclear Physics: The 63-Inch Cyclotron. – Alpha-particle spectra and angular distributions from sodium targets bombarded with 28-Mev nitrogen ions were measured. The cross section for the radiative capture of nitrogen on phosphorus ($N^{14} + P^{31} \rightarrow Ti^{45}$) was found to be 1.7 μb by radiochemical methods and absolute beta counting. Proton-transfer reactions ($N^{14} \rightarrow O^{15}$) were investigated for Al^{27}, Cl^{35}, and K^{39} targets. Thin platinum films were bombarded with 25-Mev nitrogen ions; faint tracks in the platinum were observed with an electron microscope. The aim of this experiment was to investigate formation of tracks in thin films by ionizing particles. Thoria was bombarded with 24-Mev nitrogen ions to simulate radiation damage by fission fragments.

Electronuclear Machines: The Isochronous Cyclotron. – The initial phase of testing the ORIC 76-in. magnet has just been completed. After the pole tips were aligned, the Hall probe-positioning mechanism was placed in the magnet and the field was measured at 7200 points in an automatic sequence requiring 12 to 15 hr. In this first phase of measurements, field surveys at nine main-coil current levels were made for determining the basic field patterns produced by the main field coils with only the three-sector steel pole tips in place. An extended series of measurements will be required to evaluate the range of contributions of the valley coils, harmonic coils, and circular trimming coils. Early analysis of results from the basic measurements indicates that the field configuration is consistent with predictions from model measurements.

The valley coils and harmonic coils were completed, welded in stainless steel "cans," and vacuum-tested. The cans were then filled with glass beads and impregnated with epoxy resin. One of the two disks of

circular coils is also ready to be canned; special welding techniques were developed to prevent distortion.

The detailed design of the dee and liner components was completed, fabrication techniques were developed and tested, and the fabrication is proceeding satisfactorily. The structures are formed from sheet copper; flattened copper tubing is being attached by Heliarc braze welding (BT silver alloy) in a pattern designed to minimize distortion.

Models of the power-amplifier grid circuit and the power-amplifier resonator coupling line were fabricated. Design of the rf trimmer and its hydraulic control was completed, and the system is being fabricated. The plate supply for the rf power amplifier was completed and tested satisfactorily, except for further efforts on the part of the vendor to reduce the voltage ripple. A "breadboard" intermediate amplifier was constructed and is being tested; a driver amplifier is under construction.

The basic design of the beam deflection system and the ion optics system for focusing the external beam and directing it to the various target positions are essentially complete. Beam stops and shield plugs are being designed. An elaborate safety interlock system was designed and is being installed to permit safe maintenance work in one experiment room while the beam is being used in another room.

The 32-in. diffusion-pump assembly was installed except for the Freon baffle, and the mechanical forepumps and main vacuum lines were vacuum-tested. The 20-in. diffusion pump was assembled and checked out, and is ready to be attached to the dee-stem house.

Stable Isotope Development. – Monitoring the calutron receiver with an oscillograph indicated the possibility of extreme defocusing of the beam when the ionic sound frequency of the source plasma is in resonance with the cyclotron frequency of the ion beam. At this point the beam may be swept back and forth across the pocket with an energy span of several kev. The ionic sound frequency is a function of electron temperature, atomic weight of the ions, and the dimensions of the arc chamber. With the proper choice of ionization chamber size and support gases, it is expected that this defocusing effect can be minimized, with a resultant improvement in the purity of the separated isotopes.

Striking pads of Mg, Cu, and Ni have been prepared for calutron receivers by compressing the metallic powder in a mold under several tons of pressure. Although pads formed in this manner are less dense, it is hoped that their porous structure will provide a more retentive striking surface for the ion beam.

Special Separations. – A total of 18 heavy-element shipments were made during July and August.

CHEMISTRY RESEARCH

Thorium Oxide Studies. – Photomicrographs of unirradiated pellets (code P-82) and those irradiated three months in D_2O in the LITR ($\sim 2 \times 10^{13}$ neutrons cm^{-2} sec^{-1}) showed essentially no differences attributable to irradiation. Both the original and irradiated pellets were composed of heterogeneous structures with regions of varying densities, 1- to 2-μ

pores, occasional pores 25 to 50 μ long and 10 μ wide (probably resulting from the manufacturing process), and some impurities. The interiors of the irradiated pellets showed a uniform, mottled gray-black coloration similar to that of the exterior surfaces of the pellets.

Photomicrographs of Davison thoria pellets that had been treated with dibasic aluminum nitrate, which experienced considerable damage under irradiation (20 out of 50 pellets disintegrated during a four-month irradiation), showed a much higher porosity and many more large pores than the P-82 pellets. The epoxy resin used in mounting the pellets for polishing penetrated into the interior of one of the two pellets used in the metallographic examination, indicating that some of the interior pores were surface-connected. A wear test on some of the surviving irradiated pellets showed only slightly more wear than that shown by the controls. Hence it appears that the rather excessive damage shown under irradiation probably resulted from the high porosity of the pellets and poor quality control in their manufacture.

Recalculation of rate data from a series of radiolytic hydrogen and oxygen recombination experiments carried out at 250, 265, and 280°C with a slurry of 1600°C-fired ThO_2 containing 100 g of Th per kg of D_2O and 300 ppm Pd (based on thorium) showed an apparent activation energy for the palladium-catalyzed H_2-O_2 reaction of 31 kcal ($k = 1.67 \times 10^{14} e^{-31,000/RT}$). The value previously reported was 24 kcal.

Equipment Decontamination. – Corrosion acceleration in oxalate-peroxide mixtures at 0.05 to 0.10 M H_2O_2 was eliminated by use of acetate instead of citrate as the buffer and peroxide stabilizer, and by raising the pH from 4.0 to 4.5. Glycolate accelerated corrosion even more than the citrate. Oxalate-peroxide was effective on ruthenium which had been baked onto steels in helium at 110°C (decontamination factor = 47 in 20 min at 95°C). Alkali hydroxide solutions, acidic or basic reducing agents, and oxalate-peroxide were useful in removing carrier-free I^{131} from metals after the iodine had been deposited on the metals from helium at 400°C (decontamination factor up to 1000). Type 347 stainless steel contaminated with nonsmearable americium was decontaminated by factors of 23 to 635 by various acidic reagents in 20 min at 95°C. Fluoride aided the decontamination. The decontamination factor in low-corrosive oxalate-peroxide was 80. Cerium and europium, simulating transuranics, were removable from various metals by factors of 22 to 6000 in six different reagents. Decontamination from transuranics therefore appears feasible.

Protactinium Chemistry. – The solubility of protactinium in sulfuric acid solutions is being investigated in an attempt to find a solvent, not containing fluorides, which will form stable solutions containing several milligrams of protactinium per milliliter. Stable solutions have been reported containing 17 mg/ml in 7.7 N H_2SO_4 and 36 mg/ml in 3 N D_2SO_4. In initial solubility experiments with 3.5 N H_2SO_4 a gel containing 30 mg of protactinium per milliliter formed after several hours. The protactinium concentration decreased gradually to 6 mg/ml after two weeks as the protactinium precipitate formed a layer on the container surface, but the gel was not broken. In 9 N H_2SO_4 an apparently stable solution was obtained with about 8 mg of protactinium per milliliter. As the acid concentration increased above 9 N the protactinium solubility decreased.

Radiation Chemistry of Organic Moderators. – Exposure of purified biphenyl samples in evacuated Pyrex tubes to temperatures of 300 and 400°C has been continued for 718 and 1212 hr, respectively, without visible signs of decomposition in either case.

Various diffusion-pump oils have been exposed to high temperatures in order to determine their relative thermal stabilities. Small samples of the oils were placed in Pyrex tubes, evacuated, and then heated gently under vacuum before being sealed preparatory to pyrolysis. Sample pairs of purified m-bis(m-phenoxy-phenoxy)-benzene were placed in furnaces at regulated temperatures of 400, 450, and 500°C. A faint yellow discoloration of the material became apparent after 34, 2, and 1.5 hr at the respective temperatures. The sample at 500°C was dark brown and extremely viscous after 20 hr. However, the sample at 400°C appeared to continue decomposing until it had been exposed for 175 hr, after which there was no further increase in the intensity of its yellow color, although the sample was heated for 300 additional hours. This suggests that only an impurity in the ether is being pyrolyzed at 400°C rather than the ether itself.

Samples of Consolidated Vacuum Corporation's Octoil-S (2-ethylhexyl sebacate) and Octoil (2-ethylhexyl phthalate) were also prepared for pyrolysis in the manner described above. Yellow discoloration of the Octoil-S began after 2.5 hr at 400°C and after only 1 hr at 450°C, in each case crystallizing to a white solid upon being cooled to room temperature. Octoil produced evidence of having even lower thermal stability, for it became markedly discolored after 45 min at 450°C, depositing a white solid upon reaching room temperature. The sample at 400°C was not discolored after 3.5 hr, but it did precipitate a white solid upon being cooled to room temperature, indicating that chemical change had occurred. There was no visible evidence of the onset of this change, however.

Surface Studies: Chemical Development. – Observations on the conductance of dilute electrolytes in a cell containing a porous plug of ThO_2 have indicated a marked reduction of the cell constant when a peptizing electrolyte (Na_2CO_3) was used. The effect was reversible with a flocculating electrolyte (Na_2SO_4) and was indifferent to 0.1 N KCl. A similar shift has now been observed with $Th(NO_3)_4$ as the peptizing electrolyte. However, in contrast to the observations with Na_2CO_3, the $Th(NO_3)_4$-peptized plug is flocculated with 0.1 N KCl.

The study of particle-particle repulsion in ThO_2 slurries is of interest to the development of breeder reactors; preliminary tests were made in a study of the adsorption of ions on the surface of ThO_2 particles as a function of solution concentration, temperature, and pH. Ideal particles for study, which would permit best comparison of experimental results with theoretical estimates, would be spherical, of small diameter, and would have no internal surface available for adsorption. The actual particles used were made from the oxide formed by the calcination of 1.0-μ particles of thorium oxalate; this oxide, consisting of many flat crystallites with much internal surface area, was reduced in size by digestion in hot 0.01 N nitric acid and classified by sedimentation. The fraction between 0.2 and 0.3 μ in effective diameter was retained for test. Its surface area by nitrogen adsorption was still five times that of ideal particles. The acidity of the equilibrium suspending solution in the adsorption tests

was followed through the pH range 6 to 3; the calculated fraction of surface sites covered by adsorbed acid increased from 0.04 to 0.15. The smallness of the coverage at the low pH was surprising, suggesting that much of the surface available for nitrogen adsorption was not effective for ion adsorption. The results encourage the belief that efforts to approach more closely the ideal spherical particles will give useful information concerning the theoretical and practical aspects of particle-particle interaction.

Analytical Chemistry Research. – The separation of selenium from tellurium and other elements by solvent extraction was investigated. Selenium, as hexabromoselenic acid, H_2SeBr_6, is extractable into benzene from mixed solutions of HBr, H_2SO_4, and $HClO_4$. The degree of extraction at different concentrations of these acids was determined, and optimum conditions were established by the use of radioactive Se^{75} (112-day) and stable selenium in solution. Microgram or milligram quantities of selenium (as much as 33 mg) were extracted, with benzene, to the extent of 97% or more from an aqueous system composed of 1 M HBr, 7 M H_2SO_4, and 7 M $HClO_4$. This method is suitable for the separation of Se from Te, Sb, Ga, and Sn, elements which are not extractable under these conditions.

Work was continued on the determination of antimony by controlled-potential coulometric titrimetry. On the basis of precision studies, a relative standard deviation of 0.4% was found for the reduction and subsequent oxidation of 2 to 5 mg of antimony. Efforts are now being directed to the application of this method to the determination of Sb(V), which must be chemically reduced to Sb(III) prior to coulometric titration.

Aqueous Solution Chemistry. – The bisulfate acid constant was computed from 25 to 225°C from data on the solubility of Ag_2SO_4 in H_2SO_4 solutions. The values at any temperature within the indicated range are expressed by the equation

$$\ln K_2 = -\frac{1283.108}{T} + 12.31995 - 0.04223215T ,$$

where T is the absolute temperature. In addition, the thermodynamic constants for the reaction $HSO_4^- = H^+ + SO_4^{2-}$ were calculated.

Radiation Chemistry. – In the Co^{60} gamma radiolysis of aqueous nitric acid solutions, the absorbed energy causes, in addition to the decomposition of nitrate ions to nitrite and oxygen, a decomposition of acid molecules to OH and NO_2 radicals. Evidence for these radicals is based on a comparison of the effect of added $NaNO_3$ and HNO_3 in the radiolysis of dilute aqueous solutions of ceric sulfate with and without thallous sulfate present. Addition of thallous ion to a ceric sulfate solution increases the yield for ceric reduction. According to the mechanisms proposed for these two systems, the increase in cerous yield is equivalent to $2G_{OH}$, where G_{OH} is the 100-ev yield of hydroxyl radicals produced by the decomposition of water. The addition of $NaNO_3$ to these two ceric systems increases the ceric reduction rate by the same amount, indicating that the $NaNO_3$ has no effect upon G_{OH}. Adding HNO_3 to these two ceric systems, however, produces a greater increase in the ceric reduction rate when

thallous ion is present. The difference in the two yields increases linearly with HNO_3 concentration, suggesting an additional source of OH radicals, postulated to be the decomposition of HNO_3 to OH and NO_2.

High-Temperature and Structural Chemistry. – The specific conductivities of solutions of cerium and praseodymium metals in their triiodides were found to rise rapidly to values, at saturation, more than 50 times the conductivities of the triiodides. On the other hand, the conductivity (essentially ionic) in solutions of neodymium in molten NdI_3 goes through a maximum (only 2.5 times the conductivity of NdI_3) near 50 mole % NdI_2. The latter compound is known as a nonconducting solid, while the corresponding solid iodides of cerium and praseodymium are known as metallic conductors. Further experiments are in progress to determine whether the maximum near 50 mole % NdI_2 is due to a mechanism involving electron exchange between Nd^{2+} and Nd^{3+}.

The curve of conductivity vs concentration for solutions of praseodymium metal in $PrCl_3$ was found to lie between the almost horizontal curve for neodymium in $NdCl_3$ (little or no electronic conductance; solutions are mixtures of $NdCl_3$ and $NdCl_2$, both known as insulating solids) and those of lanthanum and cerium metals in their trichlorides. In the latter, the conductivities rise rapidly to values, at saturation (~9 mole %), approximately five times those of the trichlorides.

Transuranium Elements. – A prototype of the transuranium facility intercell conveyor was fabricated and was installed in the cell mockup for testing. A glove box for solvent handling is being tested to determine solvent fire hazards and conditions resulting in loss of containment. A pressure differential of 6 in. H_2O will displace the plate-glass viewing window. In order to avoid flammable concentrations of solvent vapor, the solvent temperature must be low enough to give vapor pressures below the flammable partial pressure. At the design ventilation rates, the solvent vapor concentrations throughout the box approached the equilibrium concentrations.

Following removal of plutonium from a solution of highly irradiated Pu-Al alloy, the americium, the curium, and rare-earth fission products can be recovered by sorption from neutralized nitrate solution on anion exchange resin. In tracer experiments at 85°C, increasing the resin (Dowex 1) size from 50–100 mesh to 50–60 mesh to 10–50 mesh increased the americium loss from 0.9 to 2.3 to 31%. Decreasing the cross linkage from 10× to 4× for 50–100 mesh resin increased the americium loss from 0.9 to 9%.

Results of solvent extraction scouting tests indicated that einsteinium and californium are as difficult to separate as trivalent americium and curium, the maximum Es/Cf separation factor being 1.65 for the system di(tetramethylbutyl)phenylphosphonate vs 1 M HCl. Distribution coefficients (organic/aqueous) between 0.6 M Alamine 336 in diethylbenzene and 10 M LiCl containing 0.02 M HCl are as follows: californium, 11.8; fermium, 9.6; einsteinium, 8.9; and berkelium, 3.0; giving Cf/Fm, Cf/Es, and Cf/Bk separation factors of 1.2, 1.3, and 4.0 respectively. Distribution coefficients (o/a) between 1.05 M mono-2-ethylhexylphenylphosphonate in diethylbenzene and 2.25 M HCl are Cf, 0.66; Es, 0.83; and Fm, 2.08. Unless a more favorable extraction system is found, californium, einsteinium, and fermium will be separated from each other by chromatographic elution with ammonium α-hydroxyisobutyrate from a cation exchange column.

Uranium-232 Preparation. – As a joint effort with the British and the Canadians, ORNL will prepare two samples of U^{232} of high isotopic purity for nuclear cross-section measurements. One sample, approximately 20 mg, will contain 100 ppm of U^{233}, and the other, approximately 1 g, will contain 1% U^{233}. The chemical flowsheet for the process was developed and was tested with tracers. The design work on the necessary hot-cell equipment is almost complete, and the special equipment is being constructed.

A charge of 50 g of Pa^{231}, as the oxide, will be irradiated in the ORR at a flux of 10^{14} neutrons cm^{-2} sec^{-1}. The flowsheet provides for dissolving this in hydrochloric acid containing a small amount of hydrofluoric acid, isolating the uranium by anion exchange, and purifying and concentrating the uranium by solvent extraction. The effluent from the anion exchange step, containing the protactinium, will be treated with aluminum chloride to complex the fluoride, and the protactinium will be loaded onto a second anion exchange column. After elution from this column, the protactinium will be precipitated with ammonium hydroxide, filtered, and ignited to the oxide. The entire procedure will then be repeated to prepare the second sample of U^{232}.

Target Preparation. – Electron bombardment melting equipment has been installed in the 24-in. evaporator, and normal iron has been successfully evaporated. The techniques are being developed prior to evaporating $Fe^{56,57}$. An additional 18-in. evaporator has been delivered and is in use.

Thickness measurements of thin (15 to 150 $\mu g/cm^2$) carbon films have been made by use of a densitometer. The measurements have been confirmed by multiple-beam interferometry to be accurate within ±7%.

Studies have continued with the decelerating ion receiver. The addition of a mechanical device to rotate the target has resulted in a more uniform deposition of the collected material. Targets of B^{10} and B^{11} on tantalum and Ni^{58} on copper have been prepared.

METALLURGY AND MATERIALS RESEARCH

Research and Development on Pure Materials. – Experiments to establish the conditions necessary to avoid LiF evaporation during the growth of single crystals were completed. Total evaporation occurred at pressures as high as 10^{-1} mm Hg in a one- to two-day period at temperatures as low as 815°C. No significant evaporation occurred under the above conditions of pressure and time at 735°C. No significant evaporation occurred at 905°C during one day at 250 mm pressure. Less than 1 wt % of LiF was evaporated over an 11-day period at 900°C with helium pressures ranging from 760 to 1010 mm; the LiF that deposited from the vapor under these conditions formed needles which were single crystals approximately 4 mm long and 100 μ square.

The furnace for producing single crystals of LiF was reassembled in the new laboratory in Building 4500. A baffle, consisting of a ceramic insulator and a silver foil, was added to increase the axial thermal gradient at the freezing-point isotherm.

CONTROLLED THERMONUCLEAR RESEARCH

Arc Research. – Studies of the lithium arc in the long solenoid have yielded the following results and information.

A new type of cathode construction has been adopted. This construction consists in depositing 0.040 in. of tungsten with a plasma jet over 0.015-in.-wall tantalum tubing of the desired diameter. Cathodes made this way give a more uniform erosion of the cathode and increased life.

Lithium arcs have been run under magnetic-field conditions approximating those expected in the DCX-2. The anode was placed in a field of 7.2 kilogauss, and the cathode in a field of 22 kilogauss. Under these conditions it was necessary to use cathodes with diameters of 5/16 in. or less when the anode opening was 1 in. in diameter. Larger cathodes burned into the anode and also caused the lithium to leave the anode at a much higher rate.

Spectroscopic measurements of the backstream, by use of lines from excited Li^+ and the 3-m spectrometer, yield values for the drift velocity of from $(0.9 \text{ to } 1.5) \times 10^5$ cm/sec.

Probe measurements with a Langmuir type of probe give an average electron density of 1.5×10^{11} electrons/cm^3, with an average energy of 10 to 12 v and with some measurements as high as 25 v. Direct measurements of the probe potential in the backstream show voltages up to full voltage of the cathode.

Measurement of the amount of lithium deposited on a cooled copper plate yields a value of 2×10^{19} particles/sec for a 100-amp arc. Combining this particle flux with probe-determined densities yields an average drift velocity of approximately 10^7 cm/sec.

DCX-1 Facility. – The modifications for reducing the dynamic pressure in the plasma region were completed, and a steam bakeout of the apparatus was begun. After 20 hr, when a fault to ground developed in the west extension coil, the apparatus had to be returned to atmospheric pressure for replacement of the coil.

The apparatus was pumped down again, and a second bakeout was continued for 43 hr. During 19 of these hours the liner section was heated to 200°C with superheated steam. When the apparatus had cooled to room temperature, the sections that had been baked were cooled to liquid-nitrogen temperature, and the titanium evaporators were energized to deposit pumping film onto the cold surfaces.

After 4 hr of evaporation the pressure (ion gage) in the liner region was 1.2×10^{-8} mm Hg with the evaporators still energized (a heat load of 16 kw) and with an 8.5-ma injected H_2^+ beam. No reductions in base pressure were obtained by cutting off the evaporators or by filling the two cold traps in the liner region with liquid helium.

The system would maintain base pressure for about 2 hr, and then the pressure would gradually increase. Warming up the liquid-nitrogen-cooled sections about 50°C and then recooling them returned the system to nearly the original base pressure within a few minutes. Two explanations for this feature have been suggested: that the effective surface area for titanium pumping is gradually reduced by condensation of gases or that at these low temperatures the sorption of gases in the titanium film is limited to a monolayer depth.

The response of the vacuum system to a 10-ma injected H_2^+ beam was encouraging. With a fresh titanium surface the pressure rise associated with this beam was less than 1×10^{-9} mm Hg (ion-gage pressure) in both the liner and intermediate regions. Near the end of the runs the less effective surface pumping resulted in pressure rises of about 3×10^{-9} mm in the liner and 4×10^{-8} mm in the intermediate region.

Near base pressure, proton mean containment times in excess of 5 sec were obtained. Oscilloscope traces of the neutral-particle detector signals measured near base pressure during plasma buildup periods were not the simple exponentials expected but were much more complicated. The traces gradually became exponential as the pressure was increased. The character of the traces near base pressure is not yet understood.

DCX-2 Facility. – Installation and assembly of the DCX-2 facility continued. The mirror and outboard booster coils were tested to 54 kilogauss axial field and were approved for use. The inboard booster and dip coils were assembled and were installed on the track and are undergoing approval tests.

The vacuum system was assembled and preliminary leak checking was completed on all tank sections. The main tank port surfaces required remachining. The tank has since been reinstalled on the track and is ready for complete tests.

The injection duct design was completed and the duct should be available for installation during October.

Vacuum. – Pumping speeds and sticking factors for several gases were measured under six standard conditions in the large-scale getter pump test facility. Indications are that at liquid-nitrogen temperature only a surface layer is formed for any of the gases measured. Room-temperature sorption of hydrogen is not limited to a single surface layer, indicating that the hydrogen probably can diffuse throughout the titanium film. Nitrogen, oxygen, carbon dioxide, and carbon monoxide appear to be limited to surface-layer sorption even at room temperature.

BIOLOGY AND MEDICINE PROGRAM

ENVIRONMENT RADIATION STUDIES

Terrestrial Ecology: Evaluation of Fission Product Distribution and Movement in White Oak Drainage System. – The vertical and lateral distributions of radionuclides in the former bed of White Oak Lake are being determined. Preliminary results from core samples taken along transects at the lower and upper ends of the lake indicate that (1) there is more gamma activity per gram at the upper transect than at the lower one, (2) most of the gamma activity is contained in the first 12 in. of soil, and (3) activity in the upper few inches of each core is rather uniformly distributed.

Terrestrial Ecology: Clinch River Studies. – A portion of the radionuclides released to a stream are sorbed on the suspended free-flowing particulate matter. This matter consists of both inorganic and organic

phases, which must be separated for the evaluation of their respective roles in the movement of radionuclides in a river. The particulate matter from relatively large quantities of Clinch River water (6.5 gal) was concentrated and removed by constant-flow centrifuging. In order to separate the organic from the inorganic fraction, the isolated particulate matter was centrifuged through a bed of sucrose whose density increased with depth. This method takes advantage of the relatively low specific gravity of organic matter (1.1 to 1.2) and the relatively high specific gravity of the inorganic matter (2.6) to effect the separation of the two fractions. The organic matter did not pass through the 80% sucrose layer, but the inorganic fraction did. Results to date indicate that 99.7% of the organic matter, containing only 1.6% of the total inorganic matter, can be separated.

Terrestrial Ecology: Forest Ecology. – Studies of the natural cycling of Cs^{134} inoculated into white oak trees in the spring of 1960 continue. Movement of this radioisotope from woody tissue, in which it overwintered, into leaves has been found to be as great (32.7% of original inoculation) as that from the initial inoculation. Exports of Cs^{134} from leaves during the growing season were 12.7% leached out by rain, 49.9% returned to woody tissue, and 37.4% remaining in the leaves to reach the soil as litter. Mobility of radiocesium in the natural soil profile is significantly greater in wet soils. While the 0 to 1-in. soil layer contained over 60% of the Cs^{134} rained out of trees and litter, this element penetrated to a depth of over 10 in. in wet illitic soil. Uptake from Cs^{134}-tagged litter applied to soil around white oak saplings was found to be low (0.2% of the amount applied) but was as great as that from Cs^{134} solution applied to soil around saplings.

Terrestrial Ecology: Soil Microbiology. – Experiments on uptake accumulation and release of Co^{60} and Cs^{134} by the fungus *Trichoderma viride* were continued. Previous work established that leaching with water for 24 hr released 11% of the Co^{60} and 18% of the Cs^{134} present in fungus mycelium. Freezing of the mycelium resulted in loss of 89% of Co^{60} and 98% of Cs^{134}. These findings were utilized in leaching experiments testing the release of Co^{60} and Cs^{134} from sterile and fungal-infected leaf litter. Less Co^{60} (26%) than Cs^{134} (59%) could be leached from the sterile litter. The fungus reduced the quantity of Co^{60} and Cs^{134} found in the leachates after 3, 6, 10, and 15 days by 6, 24, 25, and 30% and by 36, 39, 28, and 27%, respectively, thus immobilizing in the mycelium 2, 9, 9, and 12% of the Co^{60} and 23, 24, 16, and 15% of the Cs^{134} that was initially present in the litter. These figures indicate that Co^{60} leaches less rapidly from litter than Cs^{134}. After three weeks the concentration of Cs^{134} in the mycelium was 16 times higher than that in the litter. Freezing and subsequent leaching of frozen litter and mycelium resulted in release of 31% of the Co^{60} and 37% of the Cs^{134} originally immobilized by the fungus. Comparison of these figures with the 89 and 98% release of Co^{60} and Cs^{134}, respectively, from frozen mycelium without litter suggests that this reduction in isotope release is due to adsorption of nuclides from the mycelium onto the litter.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND INSTRUMENTATION

Radiation Physics and Dosimetry. – An electron beam interacting with a dispersive (atomic or molecular) medium produces two intense sources of instability that are represented by a growing longitudinal wave and a growing transverse wave. The longitudinal wave has frequencies that are equal to the atomic binding frequencies of the surrounding medium and is designated as the "Bohr wave." The transverse wave has frequencies determined by the Vavilov-Cerenkov criterion and is similar to the Vavilov-Cerenkov wave produced by a single particle interacting with the medium. These sources of instability are "continued" into lower frequency ranges in which they produce growing waves of a "hybrid" type that are characterized by an electric vector having both longitudinal and transverse components. The longitudinal and transverse waves represent the "fundamental modes" that exist in the medium in the absence of the beam. The perturbation produced by the beam is responsible for the instability of the fundamental modes and for the occurrence of the coupling between these modes. The coupling produces electromagnetic waves in which the electric field has a longitudinal component. The conditions for coupling and the character of the instabilities have been investigated.

ISOTOPE DEVELOPMENT PROGRAM

Neutron Activation Analysis Research. – Activation analysis techniques were applied to the determination of trace elements in ecological specimens of soil, vegetation, and water. In samples of these materials, microgram amounts or less of I, Br, Mn, As, Se, La, Th, Co, Sc, Al, Zn, Cr, Ta, Ce, U, Cu, and Sb were determined either by a nondestructive assay method, with gamma-scintillation spectrometry, or by radiochemical methods of analysis. The high sensitivity of activation methods is emphasized as a result of this work, since more conventional methods are usually less sensitive for the elements cited.

Activation analysis techniques, by use of the irradiations from neutron sources having a thermal neutron flux of 6.5×10^{11} neutrons cm^{-2} sec^{-1}, followed by nondestructive assay by means of gamma-scintillation spectrometry, made possible an estimation of the amount of Cu, Zn, Na, Br, Cl, Hg, and Mn in drugs and pharmaceuticals with a greater degree of sensitivity than has been possible heretofore by the more conventional methods. Samples of hormones, vitamins, analgesics, and antacids have been assayed.

Recovery of Fission Products. – In the processing of 17% tributyl phosphate in Amsco against 1 M $NaNO_3$ in the MK II stacked hydroclone contactor, which consists of 1.5-in.-diam, 2-in.-high hydroclones with 0.5-in. underflow and overflow ports, the capacity limits were found to be determined by the gradually increasing entrainment of organic phase in the aqueous phase rather than by a sudden onset of flooding. The percentage of the organic phase entrained in the aqueous phase followed the empirical relation $E = 0.21\ a/o\ (a + o)^{1.53}$, where a and o are feed rates of aqueous and organic phases in liters per minute. For example, the organic entrainment was 3% at a total flow of 1.5 liters/min and an a/o of 10.

Radioisotope Research and Development. – The 41-kilocurie Sr^{90} SNAP 7-C heat source was completed. The source consists of 28 pellets of $Sr^{90}TiO_3$, weighing approximately 50 g each and encapsulated in Hastelloy C, and generates approximately 250 w (thermal).

A product of 2200 mc of I^{125} resulted from the irradiation of 75 ml of normal xenon at approximately 85 psig pressure for three weeks at approximately 2×10^{14} neutrons cm^{-2} sec^{-1}. A sufficiently low concentration of I^{126} for product-grade material was achieved, although the irradiation period was approximately ten times longer than previous irradiations. The three-week irradiation period allows insertion of a much larger sample in the lattice of the ORR than can be accommodated in the hydraulic tube, with a consequent production cost decrease of greater than a factor of 10.

Radioisotope and Stable Isotope Production. – Ion currents equivalent to 2510 g of separated isotopes were monitored during the past two months. Isotopes of Ca, Sn, Ni, W, Sm, Gd, and Lu were separated and collected.

Chemical recovery and refinement were completed on 34 new inventory lots of isotopes of Ca, Cl, Dy, Ga, K, Ni, Sm, Tl, and Zr, and on returned samples of Ca, Dy, Er, Mg, and Pb.

Samples of Sm^{148} and Sm^{150} were recovered, refined, and analyzed in preparation for a "second-pass" separation. The Sm^{150} was enriched to 95.41% from its normal abundance of 7.47% in the first pass and to 99.975% in the second pass. The Sm^{148} was enriched from 11.27 to 96.61% on the first pass and to 99.91% in the second pass.

Samples of Ni^{61} and Ni^{64}, products of enriched feed, were recovered, refined, and analyzed. Isotopic analyses of the first samples indicate the highest enrichment ever attained for these isotopes: 99.97% Ni^{61} and 99.92% Ni^{64}.

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