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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

OCTOBER 1961

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

OCTOBER 1961

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

SPECIAL NUCLEAR MATERIALS PROGRAM

Dissolver Solution Analyses. – Polymerized plutonium can be determined by controlled-potential coulometric titrimetry if the polymer is first converted to the monomer by treating the test portion with a trace of HF and then fuming off the HF with H_2SO_4. Since the E^0_{Pu} in 1 N H_2SO_4 is 0.50 v vs the S.C.E., the reduction is made at 0.32 v, and the subsequent oxidation is carried out at 0.68 v vs the S.C.E. Oxygen must be excluded. The total plutonium, monomer plus polymer, is obtained by this procedure with a relative standard deviation of 0.5%. Controlled-potential coulometric titrimetry can also be used to determine monomeric plutonium in the presence of polymeric plutonium; when the standard procedure is used, polymerized plutonium does not interfere because it is electrolytically inactive.

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

GCR-ORR Loop No. 1. – Operation of GCR-ORR loop No. 1 has been continuous since September 3. A prototype EGCR instrumented fuel element having a heat generation rate of 25,000 Btu hr^{-1} ft^{-1} is being irradiated. The equipment has performed well.

GCR-ORR Loop No. 2. – The 24-in. shield plug for use with GCR-ORR loop No. 2 has been fabricated and will be installed soon. The transfer table and the special tools that will be used in the remote insertion of the plug have been delivered. Installation of the main process piping is essentially complete. The operations manual is being prepared.

Structural Integrity of EGCR Through-Tubes. – The fourth in a series of five 8-in.-OD, 0.250-in.-wall tubes of type 304 stainless steel is being subjected to an external pressure loading of 460 psi at 1200°F. A comparison of the measurements made after 2168 hr with the initial measurements and with the 1056-hr measurements has revealed that the initial

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elliptical shape of the tube has continued to be magnified. However, the deformation which occurred during the last 1112 hr was considerably less than that which occurred during the first 1056 hr, although it should have been greater based on the constant stress and creep strains being linear with time. The fact that the change in deformation was less indicated that the tube material exhibited a large primary creep stage.

EGCR Instrumented Fuel Assemblies. – Detail design and advance procurement are in progress for the fabrication and assembly of four instrumented fuel assemblies for the EGCR. Each assembly consists of three instrumented fuel-rod bundles, one standard fuel-rod bundle, a flow-measuring section, and the associated rods and tubing necessary to support the columnar unit from the shield plug and Bridgman-type reactor nozzle closure. The instrumentation includes 18 thermocouples and a Venturi flow-measuring nozzle with associated gas-pressure transducers. One fuel element will be equipped with thermocouples designed for measurement of temperature in the fuel-pellet center, fuel-rod cladding, gas stream, and graphite sleeve.

Pebble-Bed Reactor Experiment. – An analog computer study of the transient behavior of the Pebble-Bed Reactor Experiment is being made. Data obtained thus far indicate a mean fuel temperature of 1096°F (550°F in and 1250°F out), a mean ball surface temperature of 1049°F, a core heat capacity of 531 Btu/°F, and a coolant circulation time of 16 sec.

Coated-Particle Fuel Element Studies. – Capsule L-CPlab, which contained pyrolytic-carbon-coated fuel particles in tantalum containers and was irradiated in the LITR to a calculated burnup of 9.3 at. % at central temperatures of approximately 2000°F, is being examined at the G-E plant, Vallecitos. Both tantalum cylinders have been removed from the Inconel container, and no significant changes are evident in their appearance or dimensions. The fuel particles have been removed from the b section, and no damaged particles have been detected.

In compatibility studies of pyrolytic-carbon-coated carbide fuel particles and uncoated fuel bodies with water vapor, two lots of coated particles, 3M-D (400- to 500-μ diameter, 80- to 100-μ coating) and 3M-F (250- to 300-μ diameter, 40- to 50-μ coating), were leached in 8 M HNO_3 for 8 hr at 90°C. The uranium removed, expressed in terms of UC_2, was 0.45 and 0.09% respectively.

A sample (~2 g) of each of the leached materials was exposed to flowing helium (~100 cm^3/min) containing 2.5 vol % water vapor for 100 hr at 900°C. The wet helium was passed downward through a bed of the coated fuel particles. The losses in weight were 3.9 and 1.0%, respectively, for lots 3M-D and 3M-F. The effluent gas was analyzed, but because of the low concentrations no accurate materials balance was obtained. The loss of carbon, as calculated from the hydrogen content of the effluent gas, was ~5% for 3M-D particles and ~3% for 3M-F particles.

Leaching of the coated fuel particles with HNO_3 after heating at 900°C gave 0.08 and 1.8% losses of UC_2 for 3M-D and 3M-F particles respectively. Microscopic examination after the exposure at 900°C showed tnat the coating had been pitted and had a much duller appearance.

Thermal Conductivity Studies of UO_2. – Measurements of the thermal conductivity of 93.4%-dense UO_2 were extended to 529°C in the radial-heat-flow apparatus. The results obtained are summarized below:

Temperature (°C)	Thermal Conductivity [w cm^{-1} $(°C)^{-1}$]	
	k_m	k_t
424	0.04487	0.04806
443	0.04416	0.04728
500	0.04204	0.04502
529	0.04097	0.04387

k_m is the measured conductivity and k_t is the value corrected to theoretical density.

Graphite-Metal Diffusion Studies. — Metallographic examination of type 304 stainless steel specimens from diffusion tests of graphite-metal couples at 1200°F revealed considerable carburization of all specimens whose surfaces were not protected by preoxidizing or copper plating. The extent of carburization was not significantly different from that obtained in specimens tested at 1300°F.

Another test was concluded after 1000 hr operation at 1000°F under a vacuum of approximately 5×10^{-6} mm Hg. The calculated contact loads were 500, 1000, and 2000 psi. Visual examination of the specimens revealed no bonding of the type 304L stainless steel to graphite, in contrast with the bonding that occurred in previous tests at higher temperatures. Also, bend tests of the 0.005-in. specimens indicated no significant drop in room-temperature ductility. It was noted that some of the specimens in the 1000°F test had developed thin oxide films.

Beryllium Capsule Irradiations. — Two beryllium capsules containing UO_2 pellets, which were fabricated by the French CEA, have been irradiated and disassembled. Designs of these capsules, designated 04-4 and 05-4, were identical with those of previously examined capsules 03-3 and 04-3. The irradiations were terminated ahead of schedule because of indications of fission-gas leakage during operation. Upon removal of the NaK from the outside capsule surfaces, the central region of capsule 05-4 appeared somewhat darker and rougher than other areas, but no other unusual features were seen. Dimensional measurements showed no significant changes in capsule 04-4, but capsule 05-4 was somewhat smaller in diameter and had bowed a maximum of about 18 mils. Both capsules were leak-tested by immersion in hot silicone oil. Some bubbles were observed coming from capsule 04-4, but evidence of a leak was inconclusive. Analysis of samples of the NaK from around the capsules by gamma-ray spectrometry showed only Ag^{110}. The silver, which was used as an extrusion lubricant during forming of the beryllium tube, was then removed from the NaK by chemical methods. Examination of the silver-free solution revealed no gamma-ray activity, indicating no fission products. Analysis of gas samples for Kr^{85} and Xe^{133} indicated that somewhat less fission gas was present than was found in previous capsules. This indicates lower gas release rates or loss of part of the gas prior to sampling. Scans of gross gamma-ray

activity versus length along the capsules showed no unusual flux distributions or fuel shifting.

The pellets in capsule 04-4 were extensively cracked but remained in position during opening. No pellets were removed intact; so no dimensional measurements were possible. After extensive visual examination, a small force was applied and the capsule broke in half, showing the beryllium tube to be extremely weak and brittle. The UO_2 pellets were severely cracked throughout the capsule. This shattering of the capsule indicates that it was under a high tensile stress brought about by the collapse of the tube during high-temperature irradiation and then cooling from the operating temperature, since beryllium has a higher coefficient of thermal expansion than that of UO_2. Further, this capsule was irradiated under a higher pressure, that is, a maximum of 850 psi compared with 300 psi for capsule 04-4. If leaks developed in the capsules, they were not of sufficient size to admit the NaK which surrounded the capsule during irradiation.

THORIUM UTILIZATION PROGRAM

Thoria Pellet Development. — Correlations between certain physical properties of thoria pellets and the performance of those pellets in accelerated attrition tests were established for several of the batches of ThO_2 pellets mentioned previously (ORNL-3190, p 5). Pellets of lower bulk density (8.5 to 9.0 g/cm^3) abraded less rapidly than did those of higher density (9.0 to 9.6 g/cm^3). The better pellets had a larger volume of surface-connected porosity, a smaller average pore size, and a narrow pore-size distribution. Whereas 80% of the porosity of the better pellets was contained in pores ranging from 0.03 to 0.3 μ in diameter, 80% of the open pores of the poorer pellets had diameters between 0.4 and 100 μ. The BET (nitrogen) surface area of the better pellets was also appreciably larger than that of the poorer pellets.

A program was initiated to correlate measurable physical properties with performance of cold-pressed-and-sintered thoria bodies under simulated reactor conditions. The initial studies are being carried out with small cylindrical pellets prepared from a single batch of carefully homogenized ThO_2 powder. It was shown that greater densification occurred during sintering for 90 min at 1650°C in hydrogen or argon than in propane-air combustion gases.

Fuel Cycle Development: Chemistry of Solid Fuel Element Materials. — A necessary part of the study of the chemistry of solid fuel element materials is the determination of the chemical composition and crystal structure of the phases present at equilibrium. Both temperature and oxygen pressure are known to have an important effect on these properties of the system UO_2-ThO_2. Experiments have recently been completed in which UO_2 and seven ThO_2-UO_{2+x} solid solutions were equilibrated with air at 290 ± 3°C for 98 days, after having previously been treated with hydrogen at 800°C to reduce the O/U ratio to 2.00. Chemical analyses of the equilibrated samples were obtained; final compositions ranged from $UO_{2.80}$ to $Th_{0.8}U_{0.2}O_{2.095}$. The solid solutions remained as single-phase fluorite crystals during the oxidation at 290°C. The total oxygen contents of these

samples were higher than those previously equilibrated at temperatures of 650 to 1500°C. The oxidized sample of uranium dioxide contained two phases previously observed by Hoekstra and Siegel [J. Inorg. & Nuclear Chem. 18, 154 (1961)] as the products of oxidation at 400°C – U_3O_8 and a UO_3 phase of the analytical composition $UO_{2.82}$. The value of the overall O/U ratio obtained with the UO_2 sample, 2.80, is not inconsistent with the result of a long extrapolation of the recent definitive results for the U-O system at temperatures of 1075 to 1800°C (C. A. Alexander and T. A. Shevlin, "Uranium Oxide Pressure-Temperature-Composition Relationships from 1000 to 2000°K," paper No. 14, Second Conference on Nuclear Reactor Chemistry, Gatlinburg, Tenn., Oct. 10–12, 1961).

Fuel Cycle Development: Fuel Preparation. – The sol-gel procedure for preparation of dense, compactible UO_2-ThO_2 was simplified. Addition of 3 to 7 wt % of uranium to thoria sols as $UO_2(NO_3)_2 \cdot 6H_2O$ rather than as ammonium diuranate produced a homogeneous mixture of uranium and thorium in the sol and the final fired product. During precalcination of dried gels, rates of temperature rise of 100 and 300°C/hr, maximum temperatures up to 1050°C, and times at maximum temperature of 1 to 17 hr had no significant effect on the final fired particle density when the powder was air-fired at 1200°C for 4 hr. Uranium in valence states other than U^{4+} can be reduced in the final powder by heating in atmospheres of argon, argon–4% hydrogen, nitrogen, and pure hydrogen; all appeared to be equally effective in decreasing the O/U ratio. Thus it may be possible to remove the hydrogen firing from the flowsheet.

In nine batch runs (5-kg scale) using the sol-gel flowsheet, products were test-vibrated to packed densities of 8.4 to 8.8 g/cm^3 (with eight samples > 8.5 g/cm^3) in 5/16-in.- and 1/2-in.-diam stainless steel tubes. Variations in the N/Th ratio of 0.09 to 0.13, evaporation of sol beds of 3/4- to 2-in. depth, and air-calcination temperatures of 500 to 700°C for thoria prior to sol preparation appeared not to be critical with respect to obtaining packed densities of 8.5 g/cm^3 or greater.

Eight fuel element specimens for irradiation under GCR conditions in the ORR poolside facility were prepared by pneumatic vibratory packing of sol-gel-prepared UO_2-ThO_2 (2.5 and 3.9 wt % enriched uranium) to bulk densities of 8.58 ± 0.03 g/cm^3 in 5/8-in.-diam stainless steel tubes containing a 1/8-in.-diam molybdenum axial thermowell and to 8.75 ± 0.08 g/cm^3 in 1/2-in.-diam stainless steel tubes.

Fuel Cycle Development: Fuel Fabrication Studies. – Evaluation of the ThO_2-UO_2 produced by variation in the sol-gel process has continued. Vibrated bulk densities obtained in 5/16-in.-OD, 11-in.-long, 0.025-in.-wall type 304 stainless steel tubes ranged from 8.38 to 8.73 g/cm^3. A vibrated bulk density of 8.54 g/cm^3, as specified for the BNL development program, was obtained in a type 304 stainless steel tube of nominal dimensions 1/2-in. outside diameter, 4-ft length, and 0.035-in. wall.

Vibratory-compaction experiments are being conducted to obtain high bulk densities for fused ThO_2 particles by use of two-size-fraction mixtures rather than three-size-fraction mixtures. The use of two-size-fraction mixtures would simplify milling and blending procedures and make for more efficient utilization of material. Preliminary results indicate that densities corresponding to 87% of theoretical can be readily achieved by

this procedure. Since previous studies have shown no critical density dependence on the size distribution of the coarse fraction, a −6 +20 size fraction, fairly uniform within itself, was used as a standard for comparing densities obtainable with different fine-fraction distributions.

MOLTEN-SALT REACTOR PROGRAM

MSRE Design. — Some minor changes were made to drawings. These changes included redesign of some of the graphite core stringers to remove the grooves for molybdenum bands, which have been eliminated from the design. Shop drawings for the fuel-to-coolant-salt heat exchanger were started in preparation for fabricating the major components in UCNC shops.

Additional piping drawings were prepared for the electrical, instrumentation, and auxiliary piping in the cell penetrations. Piping for the pump lubrication system and overpressure-gas lines was detailed.

The drawings for the 10-in.-diam access plug at the top of the reactor vessel have been checked. Slight modifications to the control-rod design were made. These include enlarging the inside diameter to give better cooling and substituting a rare-earth oxide poison for B_4C in order to eliminate the pressure problem from helium evolved from B_4C under irradiation.

Additional support lugs were added to the inside of the primary containment cell to support the primary-system components. Detailing of support members continued for the reactor, the primary piping, and the auxiliary piping.

The instrument-application drawings were reviewed and considerably simplified by the elimination of unnecessary valves and thermocouples.

A complete dynamic study of the operation of the MSRE system was made on the analog computer. A basic control system was designed on the basis of the simulator results. Specific control-rod-actuator design was started.

A basic heating element for the fuel piping was designed. The design was checked in a test unit and found to be satisfactory. The radiator electrical heater and wiring layout was completed.

Ten minor changes to the major building modifications package were made. Typical of the changes was the elimination of the liners and paint on the fuel-transfer and fuel-storage cells and the substitution of A-300 steel for stainless steel for the diaphragm seal on the primary containment and fuel drain cells.

Prints of the filter pit stack, the inlet air filter, the control-room partition changes, and the vent house were issued for approval.

Prints showing electrical services for the exhaust fans, compressors and air dryer, maintenance shop, change room, and control room were issued for approval. Drawings for the cooling tower are nearing completion.

Specifications are being prepared for the exterior site-modifications contract.

Design of the stereotelevision vehicle was finished. This completes the design and specification of the remote-maintenance complex consisting of the mechanical manipulator, the stereotelevision viewing system, the remotely operable cranes, the remotely operable tongs for handling shielding, and the maintenance control room.

Design of the graphite-sample replacement system and of small-component handling tools continued.

MSRE Procurement and Construction. — Contracts for furnishing INOR-8 pipe and tubing were awarded to Wall Tube Company, Michigan Seamless Tube Company, and International Nickel Company.

All difficulties in fabricating INOR-8 plate, sheet, and rod have been cleared up with Haynes-Stellite. Delivery of this material is expected to be completed in November.

The contract for producing the completely machined graphite stringers for the MSRE core was awarded to the National Carbon Company.

Following the prebid conference with prospective component fabricators, the fabricators indicated that they would not be interested in lump-sum contracts for producing the components and would only consider cost-plus-fixed-fee contracts. As a result the request for bids was withdrawn and preparations are being made to fabricate the components in UCNC shops, where considerable experience exists in fabricating INOR-8.

The low bidder for the major building modifications package was Kaminer Construction Company, Atlanta, Georgia. A contract totaling $1,242,000 has been awarded to this company, and work has begun.

Development of Components and Systems. — Installation of the reactor vessel for the full-scale hydraulic model of the MSRE core was completed. Velocity profiles measured in the entrance volute were substantially the same as predicted with the one-fifth-scale model. Fabrication tolerance problems will delay delivery of the simulated graphite and internal structure until December.

The engineering test loop has operated with MSRE coolant salt isothermally at 1100 to 1200°F and 50 gpm for a total of 4000 hr. Sediment in the pump bowl was identified as ZrO_2 precipitate produced by the addition of BeO. The ZrF_4 content was increased from < 0.5 to 4 mole % in an unsuccessful attempt to redissolve the sediment.

The reactor-vessel heater test has operated 1130 hr at a vessel wall temperature of 1300 to 1350°F. A prototype box heater for 5-in. pipe was operated at 1300°F. The circumferential temperature was constant within 10°F in the empty pipe, but a part of the heater insulation cracked from excessive thermal stress. Modifications are being made to reduce the stress. The drain-tank cooler test was successfully thermal-shock-tested 20 times at initial temperatures up to 1400°F. The measured heat removal rates verified the design calculations of the test.

A pipe joint, brazed in the laboratory, was cycled 30 times without obvious damage in a 1260°F salt loop arranged to simulate drain-line conditions.

A contract was let for the construction of the tools for the remote brazing of 1-1/2-in. pipe joints. An INOR-8 female joint member was "tinned" and is being prepared for a braze test.

The primary-pump auxiliary lines were added to the reactor cell model.

Major components, loop piping, and some in-cell equipment were installed in the maintenance mockup. Preliminary operation of the freeze-flange clamps and the replacement of the circulating-pump motor and rotary element indicated the need for some changes; these are being designed. Long-handled tools were designed for gasket handling, the flange cover system, and the pump jack system.

MSRE Pump Program. – The pump with the salt-lubricated journal bearing has been operated for 12,400 hr with molten salt $LiF-BeF_2-UF_4$ (62-37-1 mole %) and has been started and stopped 91 times. Following recent stops, the pump shaft could not be rotated by hand; however, the drive motor was capable of starting and operating the pump without evidence of difficulty. The test was interrupted for inspection of the interior rotating components.

The third INOR-8 volute casting for the coolant pump was received and was found to be a very good casting. Dye-penetrant inspection revealed only three very slight indications on the external surface. Radiographic examination revealed only one area of class-1 shrinkage and one area of class-2 shrinkage. The shrinkage areas and two surface defects are being weld-repaired. The founder was asked to pour another INOR-8 volute casting for a spare coolant pump.

Fabrication of the prototype fuel pump is progressing. The INOR-8 volute casting was weld-repaired and was joined to the pump-tank support structure satisfactorily.

Modification of the volute for the coolant-pump water test was completed.

Drawings for the rotary elements for the reactor fuel and coolant pumps are being approved by the project personnel. Pump-tank drawings, calculations, and thermal analyses are being reviewed for compliance with ORNL safety standards. The thermal analyses indicate that the pump tanks for the reactor fuel and coolant pumps can sustain the required 100 cold startup and shutdown cycles and 500 zero-to-full-power cycles.

The mechanical design layout of the drive motors for the fuel and coolant pumps were reviewed and approved. Approval of detailed design awaits submittal of detailed drawings by the manufacturer.

Metallurgy. – Specimens of CGB graphite, submitted as typical of core material for the MSRE, passed the salt-permeation screening test. An average of 0.02% of the bulk volume of these specimens was filled with molten salts during a 100-hr exposure at 1300°F under a pressure of 150 psig. The maximum acceptable permeation is 0.5%.

This material also met the mercury-permeation requirements in the MSRE graphite specification. CGB graphite gained 0.44% by weight; 3.5% is allowed.

Impregnation tests on eight relatively low-permeability grades of graphite with mercury at room temperature and under a pressure of 452 psig indicated that the mercury permeated the graphite to approximately the same extent as would molten salt in the standard permeation screening test.

All test data to date indicate that the mercury impregnation test for graphite in the MSRE graphite specification is an adequate substitute for a salt-permeation test.

Instrumentation Development. – Development of a differential scanner-alarm system, for scanning main system piping during heatup, is continuing. A circuit was developed which eliminates spikes produced by double-pole switching of the grounded-junction thermocouples. The random-noise spikes observed with the mercury switch occurred only during the "break" portion of the scanning cycle. The use of "make-before-break" contacts should eliminate this trouble.

Work is continuing on the development of a float-type continuous level transmitter for measurement of level in molten fuel and coolant salts. The differential transformer which had performed satisfactorily during the initial tests developed excessive zero and span shifts when it was inserted in an Inconel protective housing and operated at design temperature. These shifts were due to high-resistance shorts between the windings and ground and between primary and secondary windings. Further investigation revealed that the shorts were produced by condensation of an electrically conductive material which had evaporated from a Fiberflax insulation used in the transformer assembly. The effect had not previously been noticed, since the transformer was not enclosed and the material could escape. The trouble was eliminated by baking the assembly before inserting it in the housing.

Attempts to develop a satisfactory method of attaching Inconel-sheathed mineral-insulated thermocouples to Inconel pipes and vessels by gold-nickel field-brazing techniques have so far been unsuccessful, and this approach will be abandoned. However, satisfactory attachments have been made with Heliarc welding techniques. The use of welded attachments to the thin-wall tubing of the MSRE radiator was judged to be acceptable, since the welds cannot be inspected. A mechanical method of attaching thermocouples to the radiator tubing is being developed, and a test rig is being designed and constructed for testing the mechanically attached thermocouples under conditions simulating the MSRE radiator installation.

Reactor Analysis. — Preliminary results were obtained from simplified reactor kinetics calculations of the response of the MSRE to step reactivity insertions. Peak pressure rises of 64, 13, and 1.8 psi were calculated for step reactivity insertions of 0.8, 0.6, and 0.4% respectively. A rapid (~20 cps) oscillation was observed in the calculated pressure response, having a frequency characteristic of the liquid in the pipe between the core and pump bowl.

Additional multigroup multiregion calculations were performed (with the IBM 7090 program MODRIC developed at K-25) to determine the effects of Li^7 purity on fuel inventory. It is estimated that a reduction in Li^7 purity from 99.997% to 99.990% will increase the system U^{235} inventory from 58 kg to 67 kg.

The shielding requirements for the off-gas line and charcoal bed have been estimated for a 10-Mw reactor power level, assuming 100% removal of the fission gases xenon and krypton from the bypass stream in the pump bowl. For a 5000-liter(STP)/day sweep-gas flow rate, and a 50-gpm bypass flow rate, the equivalent shielding required to reduce the dose rates to 1 mr/hr was 12 in. of lead or 5 ft of ordinary concrete for the line and 16 in. of lead or 7.5 ft of ordinary concrete for the charcoal bed.

REACTOR SAFETY

Release of Fission Products on Out-of-Pile Melting of Reactor Fuels. — The effect of burnup on the diffusion of fission products from reactor-grade UO_2 sintered compacts into pure helium was investigated recently by using two types of fuel (PWR and EGCR) having irradiation levels of 1000

and 4000 Mwd/ton. A study of diffusion from trace-irradiated UO_2 was completed earlier. The examination of this effect is an extension of the parametric study program dealing with the release of fission products from overheated power reactor fuels. Previously reported data on melting of UO_2 indicated significant enhancement of release with increased burnup.

The diffusion data showed the expected increased diffusion of the volatile fission elements iodine, tellurium, and cesium with increased burnup, but also an unexpected reduced release of ruthenium. Most of the strontium release data indicated no significant difference between the highly irradiated material and the trace-level material. Only a small increase in the total rare-gas release was observed as an effect of burnup in the range investigated; however, a variation in the proportion of the gas released during heating and cooling was noted which appeared to be characteristic of the different fuel types. The data in the following table, arranged according to type of fuel and irradiation level, are typical of data obtained at other temperatures. The samples were heated for 5.5 hr in tantalum by rf induction; helium flow was 50 cc(STP)/min.

Fission Product Diffusion into Helium

Fuel Type	Irradiation Level (Mwd/ton)	Per Cent Released in 5.5 hr						
		Rare Gases	I	Te	Cs	Ru	Sr	Zr, Nb
				At 1400°C				
PWR	Trace	0.81	5.4	4.0	3.9	0.02	0.02	0.001
PWR	1000	0.5	1.6	1.2	0.5	0.0007	0.06	0.15
EGCR*	1000	0.83	0.9	0.8	2.6	0.001	0.12	
PWR	4000	6.1	23	16	21	0.006	0.08	0.1
				At 1600°C				
PWR	Trace	2.7	2.1	6.5	12.1	1.7	1.5	0.13
PWR	1000	0.4	5.5	1.1		0.025	0.23	0.07
EGCR*	1000	2.6	3.7	12	12	0.008	2.0	0.8
PWR	4000	14	25	48	43	0.2	3.0	0.35

*Large cooling peak noted in gas release.

Release of Fission Products on In-Pile Melting of Reactor Fuels. — Test materials from the first experiment on the release of fission products on the in-pile melting of reactor fuels, described in a previous report (ORNL-3062, p 11), were cut up and examined in the hot cells. The

UO_2 specimen was in two pieces, one section remaining seated in the ThO_2 pedestal, the second piece leaning against a ThO_2 cylinder which surrounds the specimen. The other components, that is, the ThO_2 and the ZrO_2 cylinder, were intact but cracked in several places. A central void surrounded by long grains was observed in the piece of UO_2. The void did not extend through the ends of the specimen. The outer wall of the specimen was characterized by sharp-pointed, pyramid-shaped crystals. This piece is being examined further to establish the mechanism of formation of the void and the crystals and to estimate the maximum central temperature in the specimen. The components of the experiment have been transferred to analytical hot cells, and radiochemical analyses are being performed to establish a UO_2 and fission product balance.

Assembly of components for the second experiment on the release of fission products is 90% complete. This experiment is identical with the first, except that the specimen is 25% enriched rather than 3%.

Modifications of the reactor facility to allow more rapid insertion and retraction of the experimental equipment are complete. Full insertion now requires approximately 4 min.

Design work is in progress to make use of the F-3 position in the ORR so that samples of reactor fuel can be irradiated to specific burnup levels for later fuel meltdown experiments. This work is approximately 35% complete.

Deposition and Removal of Fission Products from Gases. — The experimental investigation of radioactive aerosols was continued. Measurement of the distribution of radioiodine in gas diffusion cells is expected to yield information on the effects of condensation nuclei and other impurities in gases on the transport of radioactive fission products through gas cleaning systems.

Assembly is 80% complete on equipment for the study of the efficiency of iodine absorbers in steam-air systems.

Nuclear Safety Pilot Plant. — A preliminary study was made of a nuclear safety pilot plant to perform experimental investigations of various aspects of fission product release from molten fuel materials and to simulate maximum credible reactor accidents. Problems concerning the location of the plant have been investigated, and information on several available sites has been assembled. The two most acceptable sites are the B cell in Building 7500 (HRT) and a new site in Melton Valley approximately halfway between the HRT and the MSRE. The B cell has existing air-handling equipment and shielding, but its height might need to be increased. Cost estimates will be made on both the B cell and the new site.

FUNDAMENTAL HEAT TRANSFER AND FLUID DYNAMICS

Thermal Transient Studies. — Instrumentation has been developed for recording temperature fluctuations at the inner surface of a 2-in.-diam heated stainless steel pipe through which water is circulated in turbulent flow. Transients as low as 0.5°F can be recorded with an adequate signal-to-noise ratio in the frequency range from 0 to 1000 cps. Methods for spectral analysis of the random signals are under investigation. In preliminary runs at a Reynolds modulus of 25,000, temperature fluctuations

up to ±3% of the temperature difference from the wall to the mixed-mean fluid temperatures have been observed at a position 50 diameters from the tube entrance. By way of comparison, the maximum surface-temperature fluctuation which might be expected to occur according to eddy penetration theory is ±7%. No significant change in the amplitude or frequency of the fluctuations was observed when partially deaerated water was used instead of air-saturated process water.

Vortex Heat Transfer Studies. – Measurements of the friction factor for swirl flow of water in tubes (0.13 in. $\leq d_i \leq$ 0.25 in.) have been continued for tubes having full-length internal twisted tapes (twist-diameter ratios of 2.3 to 8 per 180° turn) and tube surface roughnesses of 19 to 270 μin. The new data confirm the existence of a transition phenomenon for vortex flow similar to that which occurs in axial flow. For Reynolds moduli greater than 1.5×10^4 (based on inside tube diameter, axial velocity, and fluid bulk properties) friction factors could be differentiated according to tape twist ratio and surface roughness. For Reynolds moduli less than 6×10^3, the friction factor appeared to depend clearly only on the tape twist ratio. The former situation would be characteristic of turbulent flow; the latter suggests laminar flow. In the intervening Reynolds modulus range (6×10^3 to 1.5×10^4), the data showed typical transition scatter, with friction factors ranging from values given by an extrapolation of the laminar-flow curve to those given by an extrapolation of the turbulent-flow curve.

The generalized additive prediction method for the burnout heat flux with the convective flow of subcooled, wetting liquids has been tested by using all available information on burnout (~1100 data points). These data result from measurements on axial, swirl, and cross flow in circular, annular, rectangular, and rod geometries over a wide range of the experimental variables (pressure, 4 to 3000 psia; subcooling, 1 to 506°F; velocity, 0.05 to 174 fps) with a multiplicity of fluids (water, ethylene glycol, diphenyl, monoisopropyldiphenyl, nitrogen tetroxide, ammonia, and hydrazine). Burnout heat fluxes ranged from 0.1×10^6 to 37.4×10^6 Btu hr^{-1} ft^{-2} in these experiments. The additive-method predictions deviate from the reliable water data by an average of 13.7% and from the nonaqueous data by an average of 14.7%.

MARITIME REACTORS PROGRAM

Physics Studies. – Although the main study to compare Zircaloy and stainless steel fuel containers in the NS "Savannah" core I is complete, supplementary calculations were continued. Core reactivity and control-rod worth were evaluated as a function of Zircaloy fuel-container thickness and were found to be rather insensitive to the thickness within the range 120 to 140 mils. This thickness range brackets the Zircaloy fuel-container wall dimensions obtained from various stress-analysis calculations. Also investigated was the radial power distribution in a Zircaloy fuel-container core containing two zones – a 4.2 wt % enriched inner fuel zone and a 4.6 wt % enriched outer fuel zone. As had been found in the one-zone comparison, the Zircaloy fuel container tended to flatten the radial power distribution relative to a stainless steel fuel container.

Previous one-zone burnup investigations for core I were extended to two-zone burnup investigations. The calculated lifetime of a two-zone core I (4.2 wt % inner zone and 4.6 wt % outer zone) was found to be only about one month longer than the lifetime of a single-fuel-zone core I (4.2 wt %). Replacing the stainless steel fuel container with a Zircaloy fuel container in this burned-up core I was found to increase core reactivity by about 5.8% Δk, which allows further burnup of core I fuel elements. The additional lifetime provided by replacing the stainless steel fuel container by a Zircaloy fuel container in a burned-up core I was found to produce a total core I fuel element lifetime close to the lifetime calculated for core I fuel elements in a Zircaloy fuel container throughout the entire burnup period.

Water Chemistry. — The possibility of using magnetite (Fe_3O_4) as a filter and ion exchange medium in pressurized-water-reactor primary systems is being investigated. Water samples from the ORR pressurized-water loop which had been previously filtered through a 0.45-μ micropore filter were contacted with finely divided magnetite in batch equilibrations and in column experiments. Preliminary results show that a large portion of the nonfilterable (probably ionic) water activity was removed by the magnetite. The feasibility of using a magnetic field as the means for supporting a magnetite filter bed was indicated by other tests in which such beds were easily supported by the field of an electromagnet.

ARMY POWER REACTORS

Postirradiation examination has been completed of five miniature absorber specimens containing 20, 30, or 40 wt % Eu_2O_3 dispersed in and clad with stainless steel. The specimens were exposed in ETR process water to doses ranging from 7.0 to 14.6×10^{20} thermal neutrons/cm^2 (unperturbed). As reported previously, the samples were dimensionally stable. Metallography has revealed no evidence of irradiation-induced changes.

Corrosion tests were conducted in water at 570°F on irradiated and unirradiated control specimens with the Eu_2O_3—stainless steel dispersion intentionally exposed. Little swelling was observed in specimens containing 20 wt % Eu_2O_3 during an 88-hr test, but thickness increases of 25 to 34% were observed in the specimens with 40 wt % Eu_2O_3. Corrosive attack appeared to be slightly enhanced by irradiation.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Radiation Damage: Advanced Engineering and Development. — Radiation Effects in Uranium-Doped Zirconia. — A new study was made to determine the mechanism of the radiation-induced phase transformation, monoclinic to face-centered cubic (fcc), in ZrO_2. The most important aspect of these experiments was the effort to separate the effects of fast neutrons, fission fragments, and uranium chemical impurities. This was accomplished by (1) carefully doping chemically pure ZrO_2 samples with selected concentrations of enriched and depleted uranium, (2) irradiating these samples in closely monitored thermal-neutron and fast-neutron environments and

correlating the phase changes observed with the specific particle events, (3) irradiating ZrO_2 obtained by oxidizing Zircaloy-2 that was free of fissionable impurities with a dose greater than 6×10^{20} fast neutrons/cm^2, and (4) subjecting a large group of reactor-grade zirconium and its alloys, selected at random, to neutron activation analysis for uranium and thorium impurities.

The results of the investigation indicate that (1) fission-fragment fluxes associated with 10^{15} to 10^{16} fissions/cm^3 produce a monoclinic to fcc phase change in ZrO_2, and uranium in ZrO_2 in concentrations less than 0.5 wt % does not act as a nucleation or stabilizing atom in the process, (2) the stability of the phases of ZrO_2 under reactor irradiation depends entirely upon the action of a fission-fragment flux of sufficient magnitude, and where conflicting data have been reported it is believed that an unknown presence of fissionable impurities would resolve these differences, (3) fast neutrons cannot induce the phase transition at doses up to 6.3×10^{20} neutrons/cm^2, and (4) in 19 analyses for uranium and thorium in randomly selected reactor-grade zirconium and its alloys, fissionable impurities were found in every case. Natural-uranium concentrations varied between 1.8 and 6.3 ppm, and thorium concentrations varied between 2.0 and 50.0 ppm.

Radiation Damage: Advanced Engineering and Development. — $Au_{0.82}Hg_{0.18}$, a Transmutation Alloy. — Single-crystal spheres of gold (< 0.015 in. in diameter) were subjected to thermal-neutron fluxes of sufficient magnitude to produce 18 wt % mercury in the gold by transmutation at ~85°C. These irradiations took place in the ORR. A single crystal results which is a solid-solution alloy; under normal conditions it cannot form below ~400°C.

Power Reactor Fuel Processing: Mechanical. — Mechanical dejacketing of the SRE core I, NaK-bonded stainless-steel-clad uranium fuel, was successfully completed. A total of 1780 kg of uranium slugs, 0.75 in. OD and 6 in. long, was dejacketed, steam-cleaned, recanned in aluminum, and stored to await shipment to the Savannah River Plant for processing. The over-all average processing rate, based on actual operating time, was ~6 kg of U per hr. The temperature of a can of 12 slugs stored in air was 2.5°F above the storage-room temperature of 87°F.

In shearing studies, preliminary tests of an outer gag (fuel assembly clamp) faced with rubber and a metallic inner gag contoured to the shape of a sheared assembly were very encouraging. The stepped moving blade of the shear has now made about 800 cuts against stainless-steel-jacketed porcelain-filled mark I prototype assemblies with no signs of wear.

Preliminary design of the shear-leach complex auxiliary equipment is almost complete. Design of the electrical control circuit for manual and automatic operation of the conveyor-feeder and leacher was completed.

Power Reactor Fuel Processing: Graphite-Base and Uranium Carbide Fuels. — In experiments with unirradiated 1.5-in.-diam Pebble-Bed Reactor fuel spheres, > 99% of the uranium and the thorium were recovered by leaching with boiling 90% HNO_3 when neither the spheres nor the fuel particles were coated with SiC. Uranium recoveries from spheres coated with pyrolytic carbon were high only after the spheres were roughly crushed. Both SiC-coated and pyrolytic-carbon-coated particles were inert to 90% HNO_3.

In engineering-scale tests uranium loss was not acceptable in semicontinuous runs in which −4 mesh graphite-base fuel containing 2.6% U was

leached in 90% HNO_3 at 60°C. The leaching cycle consisted of a 2-hr batch leach followed by a 4-hr leach in flowing acid, and a final water wash. Uranium losses to the residue were 1.5, 1.6, 0.61, and 0.56% at respective flow rates of 0.52, 0.91, 1.52, and 2.21 bed volume changes per hour.

In studies on the application of the chloride volatility method to processing of graphite-base fuels, chlorine at 750°C removed 97% of the U_3O_8 from 7% uranium-graphite specimens in 1 hr. Further treatment at this temperature had no effect. Treatment of one sample at 750°C with mixed Cl_2 and O_2 containing 30 vol % O_2 volatilized 98.4% of the uranium in 20 min. The graphite did not ignite.

In scouting studies on hydrolysis of uranium carbide containing considerable amounts of U_2C_3 (some UC_2 and either uranium metal or UC), the gaseous products at 80°C consisted of 58% hydrogen, 2% methane, 26% ethane, and a complex mixture of 24 additional saturated and unsaturated hydrocarbons.

Power Reactor Fuel Processing: Zirconium-Containing Fuel. — Continuous dissolution of Zircaloy-2 in refluxing 3 M HNO_3–0.4 M HBF_4–0.6 M Cr(III)–0.4 M Cr(VI)–0.46 M Zr in a titanium dissolver appears practicable. Dissolution rates were 10 mg cm^{-2} min^{-1} and corrosion rates were 0.0 mil/month in short tests. Addition of aluminum nitrate to complex the fluoride ion gave a stable product solution containing 0.36 M Zr. With the dissolvent 16 M HNO_3–2.6 M F–0.025 M HBF_4–0.4 M Zr the titanium corrosion rate was also 0.0 mil/month, but Zircaloy-2 dissolution rates were only 3 mg cm^{-2} min^{-1}.

Power Reactor Fuel Processing: BeO-UO_2 Fuels. — The initial dissolution rate of sintered 5% UO_2–95% BeO in HF-HBF_4 solutions containing up to 5 M HBF_4 increased from 0 to 1.4 mg cm^{-2} min^{-1} as the HF concentration was increased from 0 to 15 M, regardless of the HBF_4 concentration. GCRE fuel pellets, 70% UO_2–30% BeO, dissolved completely in < 14 hr in boiling 8 M HNO_3 containing 0.5 to 0.8 M NaF. The final solution contained about 4 g of uranium per liter.

Power Reactor Fuel Processing: Corrosion Studies. — Welded specimens of Haynes alloy No. 25 exposed to boiling 5 M HNO_3–2 M HCl for 264 hr were corroded at maximum rates of 0.31 mil/month, compared with 3.1 to 10.0 mils/month for Haynes 21 exposed for 192 hr. Grain-boundary attack was observed on both alloys. The poor behavior of Haynes 21 is believed to have been due to the mill anneal given this alloy.

In N_2 saturated (at 25°C) with CCl_4, Haynes 25 was corroded at rates varying from about 600 mils/month at 727°C to 10 mils/month at 580°C. The plot of log corrosion rate vs 1/T was very nearly linear. The corrosion appeared to be uniform at all temperatures.

Titanium was corroded at a maximum rate of 0.03 mil/month in 144 hr exposure to boiling 21.5 M nitric acid containing 50 g of uranium and 20 g of graphite per liter.

Power Reactor Fuel Processing: Solvent Extraction Studies. — The flowsheet used for processing MTR fuel in the Idaho Chemical Processing Plant includes an aluminum nitrate solution scrub. Substitution of a 4 M HNO_3 scrub improved decontamination in laboratory experiments, but modification of the present plant equipment would be required for equivalent results in the plant.

The amount of protactinium removed from nitric acid solutions containing protactinium at a concentration of 6×10^5 counts min^{-1} ml^{-1} by 15-min batch contact with 10 g of 100 to 200 mesh unfired Vycor glass per liter increased from 82% in 0.1 M HNO_3 to a maximum of 94% in 6 to 8 M HNO_3. With 6 M HNO_3, adsorption was more than 90% complete in the first 5 min. The presence of 50 g of Th per liter decreased the adsorption from 0.1 and 8 M HNO_3 to 45 and 86%, respectively, but increased the adsorption from acid-deficient solutions from 0 to 45%.

Radioactive Waste Treatment and Disposal: High-Activity Waste Calcination. — In nine calcination runs in engineering-scale (8-in.-diam, 90-in.-high) stainless steel calcination pots, with both batch and continuous evaporation of feed, simulated Purex and TBP-25 wastes were fed, after evaporation, to the calciner at average rates of 25 to 31 liters/hr when filling the pots to 90% of capacity. In two runs where organic phosphates were present, average feed rates were a factor of 4 lower because of excessive foaming in the calciner.

Oxides from TBP-25 waste were incorporated into glassy materials at 875 to 1000°C by addition of phosphate with borate and lead oxide fluxing agents. The solids, 2.36 to 2.84 g/ml density, represented waste volume-reduction factors of 7.2 to 8.4. The condensates from batch evaporations of TBP-25 waste made 2 M in sodium hypophosphite contained 0.48 and 0.71% of the original ruthenium; less ruthenium, 0.68 vs 2.18%, was released when the feeds were made 0.25 M in PbO than when they were made 0.25 M in $Pb(NO_3)_2$. The chief gaseous product from the reaction between iron or aluminum nitrates (1 to 2 M) and phosphorous acid (1 M) appears to be nitric oxide. The best catalysts found for the reaction between nitrate and phosphite ions were silver, mercuric, and palladium ions. Vapor-liquid equilibrium conditions were investigated for simulated TBP-25, Darex, and Purex waste solutions as a function of acid and salt concentrations, and densities of the solutions were measured as a function of temperature and pressure.

The economics of disposal of high-activity waste in salt is being studied for (1) liquids in rooms with recessed floors, (2) pot calcination vessels in racks above the floor, and (3) pot calcination vessels in holes in the floor. The gross mine-space area required for case 1, calculated by the infinite-slab-heat method, varied from 67 to 10 acres/yr for decay times of 0.33 to 30 yr.

Radioactive Waste Treatment and Disposal: Low-Activity Wastes. — An additional demonstration run on process waste water in the semi-pilot-plant facility gave effluent with < 10% of the MPC for > 1500 bed volumes of waste treated. Total hardness in the clarifier overflow averaged 20 ppm. The early breakthrough in an earlier run is believed to have been due to carbonate deficiency in the feed, which resulted in saturation of the resin with calcium. This will be forestalled in the pilot plant by continuous analysis of the effluent for hardness.

Installation of the 10-gpm low-activity-waste pilot plant is complete, including an enclosed type 3 FLC Oliver vacuum filter substituted for an Eimco-Burwell plate-and-frame filter to concentrate the sludge effluent from the clarifier. Approximately 300 hr of nonradioactive processing was done with ORNL tap water, with the objectives of orienting personnel, optimizing operating procedures, and testing equipment.

Radioactive Waste Treatment and Disposal: Disposal in Deep Wells. — A detailed analysis of the grout sheets formed at the site of the second fracturing experiment will require a survey of the deviation from vertical of the test wells, and arrangements for this survey have been completed.

Negotiations with Westco Research, a subsidiary of the Western Company (Fort Worth), have been completed, and a proposed cost-plus-fixed-fee subcontract (No. 72X-75828) has been submitted to the AEC for approval. The scope of work to be performed under this subcontract involves the development of a self-solidifying waste mixture for use in the proposed ORNL Fracturing Disposal Pilot Plant.

Radioactive Waste Treatment and Disposal: Disposal in Natural Salt Formations. — Time-space-temperature profiles were obtained around two isolated cylindrical Calrod-type heaters, placed in a pillar and in the floor of the Hutchinson mine, at distances of 2, 4, 8, and 16 ft on lines perpendicular to the heaters with power input held constant at 200 w. Values of temperature rise were obtained in three different runs, with the rock salt allowed to return to ambient temperature after each run. Scatter in the experimental measurements does not permit an accurate determination of thermal conductivity and diffusivity of rock salt, but the values appear to approximate 3.0 Btu hr^{-1} ft^{-1} $(°F)^{-1}$ and 0.10 ft^2/hr respectively. These values are in good agreement with those reported for single, pure salt crystals. There is little difference apparent in the heat transfer capabilities of the two locations, even though considerable amounts of shale and anhydrite are present in the floor, while the pillar is nearly pure salt.

An equation for the temperature distribution about a finite-length cylinder in an infinite array of cylinders has been developed and is being programmed for the IBM 7090 computer.

Radioactive Waste Treatment and Disposal: Clinch River Study. — Core samples of bottom sediments taken at ten equally spaced intervals along five cross sections, Clinch River mile (CRM) 4.7, 7.6, 11.9, 15.3, and 19.2, were submitted to the U.S. Geological Survey for size distribution analyses. Results of the analyses indicate that (1) the sediments vary in texture from silty loam to loam and (2) the sizes are classed as well-graded clay to the upper range of sand sizes (median diameters ranging from 20 to 39 μ). Average clay content is about 14%, and sand content varies from 30% at the lowest cross section to 40% at the uppermost cross section.

Results of a dispersion test, with Au^{198} as a tracer, were as follows: (1) White Oak Creek water is uniformly dispersed in width and depth of cross section at CRM 17.1, (2) travel time from the mouth of White Oak Creek (CRM 20.8) to Centers Ferry (CRM 4.5) is 30.2 hr, and (3) the dilution factor is approximately 5000. Flow conditions under which the test was run were 7 cfs from White Oak Creek and 8000 cfs in the Clinch River, with the level of Watts Bar Reservoir held between 740 and 741 ft above mean sea level.

Radioactive Waste Treatment and Disposal: Fundamental Studies of Minerals. — Ion exchange sites of vermiculite and montmorillonite were saturated with the cations K, Na, Li, Ca, Al, and Ba. Barium-vermiculite and potassium-montmorillonite sorbed the highest amounts of cesium. By

slurrying 1-g equivalents of the saturated minerals with 20 ml of 1.5 M $NaNO_3$ solution containing 25 mg of Cs per liter, barium-vermiculite and potassium-montmorillonite sorbed 2.7 and 1.6 times more cesium, respectively, than their natural counterparts after over 150 hr of contact. Potassium-montmorillonite reached equilibrium within 1 hr after contact, while barium-vermiculite did not reach equilibrium even after 190 hr. The c spacings were partially collapsed in both cases; however, the exchange capacity of the saturated forms remained approximately the same as that of the original minerals.

PHYSICAL RESEARCH PROGRAM

PHYSICS AND MATHEMATICS RESEARCH

High-Energy Electronuclear Machines. — Successful operation of the eight-sector electron cyclotron was reported previously. Full design energy (500 kev) was achieved with minor adjustment of the magnetic-field shape at the center of the cyclotron. Since the initial operation, the Analogue was dismantled and is being reassembled in its permanent location. Relocation and reassembly are 95% complete. A small error was discovered in the calculation of the currents for the main average and trimming coils; it resulted in magnetic field errors of a few tenths of a per cent. Calculation of the correct coil currents is in progress; changes resulting from these calculations are expected to reduce the threshold voltage and to improve performance of the Analogue at full energy.

Basic Studies of Cryogenic Electromagnets. — A superconducting solenoid was designed, fabricated, and test-operated; it produces a magnetic field of 32.5 kilogauss at a critical current of 19 amp. The coil was wound from 1800 ft (0.5 lb) of 0.011-in. Nb–25% Zr wire; it has a 2-in. OD, a 0.5-in. ID, and is 1.25 in. long. The critical current at which the superconducting solenoid "goes normal" at 4.2°K is somewhat less than would be expected from the following results of tests of the same wire in the 3.2-Mw High-Field Solenoid:

Field, kilogauss	0	10	25	50	70	80
Critical current, amp	200	75	45	25	12	0

The High-Field Solenoid now produces up to ~100 kilogauss in a 1-7/8-in. bore; the windings are water-cooled copper. A 0.007-in. wire (Nb_3Sn coated onto platinum wire) tested in the field of this magnet carries a current of 15 amp at zero field and 10 amp at 93 kilogauss.

Scintillation Spectrometry and Instrument Development: Recoil-Free Nuclear Resonance Gamma Scattering in Total Reflection from Fe^{57} Mirror. — Measurements were made on the reflectivity R of an Fe^{57} mirror for the 14.4-kev gamma rays from a Co^{57} source (unsplit line) as a function of source velocity v and (small) glancing angle θ. The technique of the Mössbauer effect, combined with that of total reflection, permits the study of coherent nuclear resonant gamma scattering through the region of nuclear anomalous dispersion by Doppler shifting of the source frequency. The observed fine structure depends on the chemical and magnetic environment of those Fe^{57} nuclei which lie in a very thin surface film of thickness equal to the gamma penetration depth. According to theory, $R(v,\theta)$ depends on the complex forward-scattering amplitude $f(v) = f_e + f_n(v)$, with superposed contributions from electronic and nuclear scattering. The function $f_n(v)$ is given by a multilevel Breit-Wigner expression. Numerical computation of $R(v,\theta)$ is in progress. From a partially

oxidized mirror of 89.8%-enriched Fe^{57} (300 A thick) evaporated on a glass optical flat, large (40%) changes in R(v) were observed. The most prominent feature at $\theta = 1.7 \times 10^{-3}$ is a double valley with minima at 0.09 and −0.01 cm/sec. For $\theta = 3.1 \times 10^{-3}$, R(v) exhibits several minima as well as a peak at 0.6 cm/sec. The observed scattering resonance pattern is attributed to FeO and Fe_2O_3.

Scintillation Spectrometry and Instrument Development: A Mössbauer-Parity Experiment with Ni^{61}. — Cobalt-61 decays by beta emission to the 71-kev state of Ni^{61}. After the beta decay the Ni^{61} nuclei are left as a polarized ensemble, which may be described by a statistical tensor

$$T_{ik} = \delta_{ik} + \frac{v}{c} M_{ik} .$$

The hyperfine-structure spectrum of the following 71-kev gamma radiation is, because of the parity-violating term $(v/c)\ M_{ik}$, different for left- and right-handed polarization. The off-diagonal elements of M_{ik} yield interference terms between different hyperfine-structure lines.

The Mössbauer technique (nuclear resonance scattering on magnetized nickel) allows measurement of the polarization dependence of the hyperfine-structure spectrum. A comparison between theory and experiment gives the absolute sign of the nuclear magnetic moments involved.

High-Voltage Experimental Program. — A new nanosecond pulser has been installed in the 3-Mv Van de Graaff accelerator. The equipment has been designed around a duoplasmatron ion source so that larger currents can be delivered in the pulses. A system of Lissajous sweeping is being used to eliminate retrace pulses as well as all pulses of H_2^+ and H_3^+ ions. Some difficulty has been encountered in optically matching the pulser ion to the accelerator tube. The equipment is presently delivering target pulses with a height of 1 ma and a width of 4 nanoseconds. Performance is improving as outgassing proceeds.

The yield curve for the reaction $Mg^{26}(\alpha,n)Si^{29}$ has been measured with good resolution by means of alpha particles with energies of approximately 3 to 5 Mev; the graphite sphere was used as the neutron detector. The effects of some 40 levels are evident in this region of excitation of the compound Si^{30} nucleus (13.2 to 14.9 Mev).

Multiparticle Medium-Energy Nuclear Physics: The 63-Inch Cyclotron. — The angular distributions of alpha particles from the reactions $Li^6(N^{14},\alpha)O^{16}$ and $Li^7(N^{14},\alpha)O^{17}$ were measured for transitions to the ground state of the residual oxygen nuclei. Pronounced forward maxima were observed in both cases, and in the Li^6 reaction a broad secondary maximum appears at 110° center-of-mass. The shapes of the angular distributions indicate that direct processes are important in these reactions. Several cross sections for nitrogen-induced compound-nucleus reactions in Cl^{35} and K^{39} were measured.

Multiparticle Medium-Energy Nuclear Physics: The 86-Inch Cyclotron. — The energy spectra of alpha particles from (p,α) reactions on targets in the iron and in the zirconium region were examined. A silicon surface-barrier counter was used as the E counter, and a thin proportional counter

was used as the dE/dx counter. A few low-lying levels were resolved. Comparison of the (p,α) cross sections with (p,t) and (p,d) cross sections leading to the same final state indicates that the (p,α) cross section is roughly one-tenth that of the (p,t). It is plausible that the low-lying levels are excited by the pickup of two neutrons and a proton from shell-model states. At slightly higher excitation, a continuum which is forward-peaked in angle and has a maximum well above the Coulomb-barrier energy appears in the spectra for the targets in the zirconium region. This is not yet understood. An attempt is being made to apply the tentative explanation that this represents all the states which can be reached either by a three-particle pickup or by an alpha-particle knockout in which the incoming proton is captured.

During the past two months the following isotopes were produced: Be^{7}, Na^{22}, Sc^{44m}, Co^{57}, Zn^{65}, As^{74}, $Rb^{83,84,86}$, Sr^{85}, Zr^{88}, I^{130}, Cs^{132}, W^{181}, and Bi^{207}. The problem of oxidation of scandium metal target contained in an aluminum packet was resolved by placing a magnesium wire in contact with the scandium metal strips; this produces an electromotive cell in which the magnesium wire is oxidized first and the resulting Sc^{44m} activities remain in metallic form. Several irradiations performed in this fashion enabled the Princeton University group to evaporate Sc^{44m} metal in their apparatus. A gas target of normal krypton was irradiated at 13 Mev with a beam current of ~16 μa. The resulting $Rb^{83,84,86}$ activity was found at the cold surfaces of the water-cooled target assembly. The released krypton gas showed negligible activity several days after irradiation, and ~97% of the rubidium activity was removed by washing the assembly with warm distilled water. A solid tantalum target was irradiated at an average beam current of ~1600 μa for the production of W^{181}.

Electronuclear Machines: The Isochronous Cyclotron. — Fabrication and testing of the valley and the harmonic coils were completed, and the coils were installed on the pole tips in preparation for the second stage of magnet measurements. The vacuum chamber was closed with temporary cover plates, and the complete system was vacuum-tested. The magnet measurement equipment was modified, to accommodate the new design of the valley coils, and reinstalled. Flowmeters and safety devices were installed in the many water-cooled lines, and the water flow was checked in all coils. Installation of the 19 AVF magnet power supplies was completed; regulation of the valley-coil supply was checked out, and the harmonic-coil supplies are being checked. One set of ten trimming coils (for one pole tip) was completed; the second set was wound and is being welded in its stainless steel "can." These coils will be installed before the third stage of field measurements begins.

The 48-sector shorting plane which moves along the dee stem within the dee-stem housing was assembled. Components for the dee and the transition piece are nearing completion; they should soon be ready for installation on the dee stem. One half of the water-cooled liner was completed and vacuum-tested; the other half is being fabricated. The amplifier mock-up (through the amplifier driver) was installed on the full-scale rf model; measurements are being made to verify the parameters of the coupling network. The crowbar protective equipment and circuitry for the 6949 power amplifier were designed; fabrication is 30% complete.

Analysis of the results of the first stage of magnetic-field measurements indicates a very low first-harmonic content; the small correction required near the maximum radius will be provided by the harmonic coils. In beam extraction studies, the interaction of a sharp first-harmonic "bump" in the magnetic field with the 3/3 resonance of the azimuthally varying field was investigated and found to be in agreement with approximate formulas. Coils for producing the bump were designed and are being fabricated.

Stable Isotope Development. — Test runs using tin and lead as feed materials are in progress on the prototype 255° separator (inhomogeneous magnetic field). Samples of Sn^{119} from two test runs have been assayed at 93% isotopic purity, which compares with the previous maximum of 86% in a conventional calutron with homogeneous magnetic field. A Pb^{204} content of 67% (maximum calutron isotopic purity is 70%) was obtained even though the ion exit slits were misaligned during the test run. Detailed measurements of the magnetic field with the recently installed digital data system will be made in order to evaluate the corrections needed on the magnetic shim contours to produce optimum beam resolution.

The effect of changes in arc-chamber geometry on defocusing of the ion beam when the ionic sound frequency of the source plasma is in resonance with the cyclotron frequency of the beam is being investigated cooperatively with members of the Thermonuclear Division.

A study has been made of the theory, construction, and operating characteristics of a small-scale mass filter, under direction of K-25 personnel, in order to evaluate the application of this type of device for isotope separations.

Uranium (quantities of approximately 50 g) is routinely converted from UF_4 to metal by use of the conventional calcium and iodine reduction technique. By use of a smaller reduction bomb and associated ceramic liners, it has now been possible to produce yields of up to 60% with 5-g quantities. Modifications in the reduction procedure are planned in order to increase the yield when using such reduced amounts.

Special Separations. — Second-pass separations to obtain at least 1 g each of Sm^{148} and Sm^{150} with an isotopic purity of 99.8% or greater were made, using 18 g of samarium enriched to 96.6% Sm^{148} and 11.8 g of samarium assaying 95.4% Sm^{150}.

By use of the recycle technique, the original materials were processed through the calutrons twice, and an estimated 1.349 g of samarium assaying 99.96% Sm^{148} and 1.716 g of samarium with an Sm^{150} content of 99.97% were collected.

CHEMISTRY RESEARCH

Thorium Oxide Studies. — Determination of pycnometric densities of code P-82 thoria pellets in H_2O indicated that irradiation has no particular effect on pellet density and that long-term autoclaving under water at 250°C causes some of the internal pores to become surface-connected. The density of the as-received pellets was 9.16 g/cc. After three months of irradiation in the LITR (7×10^{16} fissions/g), pellet densities were 9.45 and 9.34 g/cc before and after drying respectively. Corresponding

values for the control pellets were 9.68 and 9.40 g/cc. The density of pellets autoclaved only two days at 250°C and not dried was 9.25 g/cc.

The surface area of the irradiated pellets was 1.48 m^2/g, as opposed to 0.01 m^2/g obtained with the original pellets. However, in a 1-hr wear test (standard spouting-bed test) the irradiated pellets lost only 0.013% by weight, less than one-tenth the weight lost by the original pellets. The surface area after the wear test was 1.54 m^2/g.

The crystallites in thorium oxide powders irradiated dry in the LITR (at < 300°C) appeared to be approaching an equilibrium size of 500 to 600 A (10^6 to 10^7 Th atoms), as measured by x-ray diffraction line broadening. The original crystallite sizes of the oxides fired at 650, 800, 900, 1100, and 1500°C were 120, 220, 500, 1140, and > 2000 A respectively. After an exposure of 1.5×10^{18} fissions/g the crystallite sizes were 265, 400, 560, 540, and 850 A respectively. Annealing the materials fired at 1100 and 1500°C did not change the line broadening. Therefore 10^6 to 10^7 thorium atoms appear to be displaced by each fission in the thorium oxide.

Equipment Decontamination. — Oxalate-peroxide mixtures, with either acetate or citrate as buffers, decontaminated stainless steel from Cs^{137} by factors of 1000 to 2000 in 20 min at 95°C. The production of hazardous peracetic acid during the use of oxalate-acetate-peroxide mixtures for decontamination was negligible. The stainless steel samples were contaminated with carrier-free Cs^{137} under simulated EGCR conditions by exposure to cesium vapor volatilized at 750°C in helium from a mixture of $Cs^{137}Cl$ and metallic calcium. The peroxide mixture at 85 to 95°C was a good low-corrosion rust remover and decontaminant for miscellaneous rusty hand tools, but large quantities of rust shortened the life of the solution to about 2 hr.

In initial tests on decontamination from ruthenium oxalate—peroxide, 1 and 2 hr at 60°C and pH 2.5 was equivalent to 0.5 and 1 hr, respectively, at 95°C and pH 4.3. At 65°C and pH 2.5, corrosion was severe. In decontamination of the Pebble-Bed Reactor, pyrolytic-carbon-coated UC_2 dust will apparently have to be removed solely by detergent action, except where dissolution in fused $KOH-KNO_3$ is practicable.

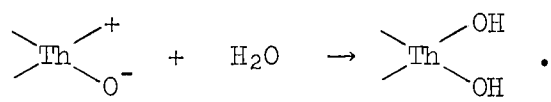
Radiation Chemistry of Organic Moderators. — Purified biphenyl samples in evacuated Pyrex tubes were heated in furnaces controlled at 475, 525, and 550°C. The samples at the lowest temperature showed no visible signs of decomposition until 120 hr had passed. At the end of approximately 3.5 hr, the samples at 525 and 550°C had begun to decompose, for they were slightly discolored yellow. By the time the samples at 550°C had been exposed for 4.75 hr, they were badly decomposed, forming a dark brown to black viscous mixture.

Products of the biphenyl pyrolysis and radiolysis, ranging from permanent gases to polymeric materials, will be identified quantitatively by means of gas chromatography. Synthetic mixtures of compounds which possibly would result from the decomposition have been used to determine the proper type of columns for these separations. A 12-ft column of Apiezon N (30 wt %) with 2% Carbowax 6000 was found to be quite satisfactory for resolving a seven-component mixture of benzene and its derivatives dissolved in acetone, using temperature programming between 75 and 200°C. Similarly, a 12-ft column of 20 wt % lithium chloride on Chromosorb P performed well by resolving a benzene solution of phenylcyclohexane, biphenyl, diphenylmethane, o-terphenyl, and p-terphenyl in 15 min by temperature programming between 200 and 350°C. In addition, hydrogen,

nitrogen, and oxygen were resolved at 5°C with no difficulty on a 10-ft column of molecular sieve 5A.

Surface Studies: Chemical Development. — Three calorimetric determinations of the integral heat of wetting of ThO_2 at 25°C yielded values of 924.8, 929.2, and 923.4 ergs/cm^2. The thoria used was 650°C-fired, oxalate-precipitated material with a surface area of 14.7 m^2/g (N_2 adsorption). Pretreatment consisted of heating at 300°C for 24 hr at a pressure of less than 10^{-5} mm Hg.

If 118 ergs/cm^2, the surface enthalpy of water, is subtracted, and a chemisorption reaction with a heat of 20 kcal/mole is assumed, the surface required is 17.2 A^2 per molecule of H_2O adsorbed. Thoria is face-centered cubic with an a_o value of 5.586 A. If each surface thorium atom can accommodate one molecule of water, the area available is 15.6 A^2 per molecule of H_2O. The agreement is sufficiently good to justify continued belief that the principal reaction is simply the hydration of the surface oxide groups:



Instrumentation for Analytical Chemistry Research. — Studies have been continued with the ORNL model Q-1988-ES controlled-potential and derivative polarograph on methods of measurement and on the interpretation of dc derivative polarograms. The forms of the experimental derivative polarograms were compared by several methods and found to be in very close agreement with that predicted by the theoretical equation for reversible, diffusion-controlled processes. Conditions necessary for obtaining derivative polarograms of theoretical form have been delineated. Overlapping polarograms, both regular and derivative, of solutions containing Pb(II) and Tl(I) were resolved mathematically by means of simple simultaneous equations for the purpose of comparing the resolution obtainable with regular and derivative polarography. Proportionality constants used in the equations were evaluated by a standard addition technique rather than from the slopes of calibration curves. The qualitative resolution of derivative polarograms was demonstrated as being superior to that of regular polarograms; furthermore, the increments resulting from standard additions are greater, a fact which is particularly desirable in making quantitative measurements. Peak and wave heights are equally reproducible. Studies were made to determine the relationship of peak height to concentration (with unvarying dropping mercury electrode characteristics) for the reduction of a number of ionic species at concentrations ranging from 10^{-6} to 10^{-3} M. For some systems, strict linearity is not obtained; however, peak height is directly proportional to wave height. With ORNL model Q-1988-ES and Q-1988A polarographs, dc derivative polarograms can be recorded whose forms are not influenced by instrument characteristics.

Analytical Chemistry Research. — In a continuation of work previously reported on the determination of antimony by controlled-potential coulometric titrimetry, a procedure has been developed for the estimation of Sb(V) or the amounts of Sb(V) and Sb(III) in mixtures thereof. This procedure involves the stepwise electrolytic reduction of Sb(V) to Sb(III)

and the further reduction of Sb(III) to metallic antimony, which amalgamates with the mercury anode. In order for the electrolytic reduction of antimony to proceed satisfactorily, a high concentration of acid and of chloride (approximately 6 M) must be maintained in the supporting electrolyte. A series of standard solutions containing Sb(V) and Sb(III) in varying proportions were analyzed by this procedure with a relative standard deviation of approximately 0.5%.

Aqueous Solution Chemistry. — The development of a new analytical separations scheme based principally on ion exchange was started. The object of the scheme, which is to be applicable to essentially all the metallic elements, is to divide them into suitable groups and subgroups. During a feasibility study, it was shown that logical groups can be obtained at high, medium, and low ionic strength with cation and anion exchange resins. These preliminary groups were tested with a considerable number of representative elements.

A new separations technique based on cation exchange and concentrated electrolyte solutions was extensively studied and found to lead to a large number of novel sparations.

Chemical Physics: Neutron Diffraction. — An x-ray diffraction study of water at 25°C shows that each oxygen atom has approximately five oxygen neighbors between 2.80 and 3.00 A, with additional neighbors at about 3.80 A. This result suggests that the atomic arrangement is a somewhat expanded ice-like framework in which some of the cavities contain interstitial water molecules.

The automatic neutron diffractometer, which collects three-dimensional, single-crystal neutron diffraction data on instruction from a computer-prepared punched tape, is in operation at the ORR. It measures about five reflections per hour in routine use.

Transuranium Elements. — Results of fire-hazard tests on o-xylene in a glove box indicated that a simple water-sprinkler backup system will be adequate for fire suppression. In a test for explosive conditions at a box temperature of 100°F (open-cup flash point of o-xylene is 75°F), no audible explosions occurred, but the pressure rose 7 to 8 in. H_2O in < 1 sec. The fires snuffed out, even when a 5-cfm purge and/or an open glove port existed.

In extraction of americium from 11 M LiCl into 30% Alamine 336 in diethyl benzene at 25°C, equilibrium was achieved within 10 sec. However, in scrubbing the pregnant organic with 11 M LiCl, about 1 min was required at 25°C and 10 sec at 60°C. An 0.81-in.-ID pulsed extraction column with nickel as a stand-in for actinides showed up to four stages in a length of 25 in.

Plutonium for the neutron cross-section program will be purified by dissolving highly irradiated Pu-Al alloy in nitric acid and isolating the plutonium on anion exchange resin. From the column effluent, 6 M HNO_3–0.75 M $Al(NO_3)_3$ containing Am, Cm, fission products, Na, Fe, Ni, and Hg, the Am, Cm, and rare earths can be recovered by anion exchange resin from neutral aluminum nitrate solution. Synthetic column effluent evaporated to a temperature of 140°C and diluted with water was acid-free and was stable at room temperature with up to 2.7 M $Al(NO_3)_3$. In tests at 70°C with 15 column displacement volumes of feed containing europium tracer on Dowex 1-10X (50 to 100 mesh) resin, europium losses from 2.7 M $Al(NO_3)_3$ feed were 23.2% for a flow rate of 4 ml cm^{-2} min^{-1} and 0.12% for a flow

rate of 2 ml cm^{-2} min^{-1}. At a flow rate of 2 ml cm^{-2} min^{-1}, europium losses were 0.12, 0.27, 2.20, and 16.2% for $Al(NO_3)_3$ feed concentrations of 2.7, 2.3, 2.0, and 1.8 M respectively.

Separation of americium and curium by chromatographic elution from Dowex 50-10X (100 to 200 mesh) resin with 0.5 M ammonium α-hydroxyisobutyrate at pH 4.0 improved with increasing resin volume. With a 5-ml column, 70% of the curium was free of americium, and with a 250-ml column, 95% was free of americium.

Target Preparation. – The direct deposition of 18-kv boron ions on metal backings is being studied. An alpha grid ion source and receiver have been modified so that an external gas feed (BCl_3) and a rotating target holder in the collector can be used. The target holder consists of a 2-in. hexagonal bar with a 1-in. water passage through the center. Fourteen metal disks are mounted on each of the six sides of the holder. This technique will permit the direct deposition of separated isotopes on a total of 84 disks per calutron run. Attempts will be made to check uniformity of the deposition by microscopy and interferometric techniques.

Electron bombardment equipment in the 24-in. evaporator has been used successfully to evaporate a 300-μg/cm^2 film of Ni^{60} on a gold backing. Iron foils, 1 mg/cm^2, and platinum foils, 3 mg/cm^2, have been rolled.

METALLURGY AND MATERIALS RESEARCH

Research and Development on Pure Materials. – The modified furnace for producing single crystals of LiF was calibrated with respect to its temperature distribution in the 2-in. region occupied by the newly inserted baffle. Thermal gradients of 10 to 15°C/cm, needed for the most effective crystal growth, were observed throughout this region.

A furnace designed to enhance natural-convection circulation of solvents up to 1200°C in closed capsules has been constructed to make use of temperature solubility coefficients and to avoid strong concentration gradients which inhibit crystal growth. Short (24- to 48-hr) tests have been performed with ThO_2, UO_2, ZrO_2, and MgO in molten fluoride solvents. The maximum crystal size was increased by a factor of 10 and the finer starting particles were dissolved. Similar results were obtained with Cu_2O in molten PbO.

Seeds of ZrO_2 and ThO_2 have been grown in place on platinum stirring rods in molten sodium tetraborate at temperatures between 700 and 1200°C. This may eliminate the need for mechanical attachment of very small seeds, usually a substantial problem.

Reactions at Metal Surfaces. – The study of the role of strain in the mechanisms of oxidation of metals has been continued with further investigations of strain-induced changes in the optical properties of thin oxide films on copper single crystals. The original Drude equations for the relative amplitude reduction and phase retardation on reflection of polarized light from a film-covered surface were modified to take into account the existence of a strain-induced gradient in the refractive index of the oxide. These equations were programmed for machine computation. Although the precise form of the refractive index gradient has not yet been established, preliminary results indicated that for very thin Cu_2O films the real part of the average index of refraction for

Cu_2O was initially larger than its bulk value but decreased rather rapidly toward the bulk value once a critical film thickness was surpassed.

Fundamental Investigation of Radiation Effects in Solids: A Primary Defect Structure of Silica and Its Relation to the Concentration of OH Bonds. — Observations on three different silicas (Corning 7940, Corning 7943, and Amersil O.G.1) showed that the intensity of the 5.9-ev band (the E_1' center) for a constant Co^{60} gamma-ray irradiation was correlated with the intensity of the 2.7-μ band. The 2.7-μ band has been attributed to the O-H stretching mode. The Corning 7943 silica had the weakest 2.7-μ band and the most intense 5.9-ev absorption.

The Corning 7943 was heated in H_2O vapor at 900°C for increasing periods of time, and the intensity of the 5.9-ev band for constant irradiation was plotted vs the intensity of the 2.7-μ band. The relation derived from this plot was $\eta_{E_1'} + A\eta_{2.7\mu} = b$, where $\eta_{E_1'}$ is the initial concentration of the E_1' centers, and $\eta_{2.7}$ is the concentration of H^+ and $O\text{-}H^-$ introduced by the heat treatment. This result suggests that the H^+ and $O\text{-}H^-$ that diffuses into the silica occupies E_1 sites. Either the E_1 sites are no longer present or they can no longer trap a single electron, thereby forming E_1' centers.

Irradiation of the Corning 7943 at 78°K by 50-kv x rays shows two rates of growth of the 5.9-ev band, a rapid initial phase and a slower second phase. The concentration after irradiation with 50-kv x rays at 78°K for a dose of $\sim 2 \times 10^{18}$ photons was $\sim 2 \times 10^{19}$ centers/cm^3. No indication of saturation was evident. An extrapolation of this data suggests a saturation concentration > 1%. It is suggested that the E_1 center is a fundamental characteristic of the silica structure and that H or O-H can occupy the site of the E_1 center in such a way as to increase the energy required to form it.

Fundamental Investigation of Radiation Effects in Solids: Yield Stress of Copper Crystals. — Copper crystals, 99.999% pure, were deformed in tension with an Instron tensile tester, and the dislocation density and arrangement in the crystals was determined before, during, and after the deformations by use of an etch-pit technique. For crystals of low initial dislocation density, it was found that a large amount of disolcation multiplication occurred prior to yielding. Experimental relationships of dislocation density vs applied stress and vs shear strain were determined. It was found that the yield stress was not related to the initial dislocation density or arrangement. It is suggested that the yield stress is determined by the stress necessary to enable the gliding dislocations to break through impurity-atom barriers in the crystal.

CONTROLLED THERMONUCLEAR RESEARCH

Plasma Physics and Arc Research. — The use of Langmuir probes for density measurements has been proven accurate to within better than a factor of 2 for the deuterium arc plasma, even though the probe casts a very definite electron shadow toward the anode and a more diffuse shadow toward the cathode. This has resulted in more confidence in the electron temperature measurement of $T_e \doteq 40$ ev. The tail of the

energy distribution measured by the probe is a good fit to an exponential, as expected from the Maxwellian distribution predicted from Spitzer's calculations.

The ion impurity temperature has been found to be proportional to the electron current through the arc. The ion temperature was found to decrease as the impurity level is increased, but the normal background gas level of impurity is imperceptibly low. The ion temperature has been measured at the center of the gas arc facility and ~50 cm away (at the cathode). No (< 2 to 3 ev) change in the ion temperature was observed.

In comparison with the preliminary predictions from the Rose model for the arc, (1) the density was predicted to decrease toward the cathode but was found to be constant or to increase; (2) the temperature was predicted to increase toward the cathode for both electrons (slight) and ions, but the ion temperature was found to be constant.

DCX-1 Facility. — The ion source and the accelerator tube were disassembled and cleaned following a break in the water jacket of the ion-source diffusion pump. The east inboard coil, which had a partial short circuit, has been replaced. The source was reinstalled along with the spare accelerator tube, and the injection system was completely realigned. The bakeout of the injection system is in progress and is almost complete.

Further vacuum tests have been performed; they started after the apparatus had been opened to the atmosphere for some mechanical operations. With a 3-1/2-hr bakeout at 175°C the diffusion pumps produced a base pressure in the liner region of 1.4×10^{-6} Torr (ion gage reading). Adding liquid nitrogen to the liner cold traps reduced this to 2.5×10^{-7} Torr. Evaporating titanium onto the water-cooled liner produced a base of 3.5×10^{-8} Torr, and cooling the liner with liquid nitrogen produced 1×10^{-8} Torr. On and off cycling of the liquid-nitrogen cooling and the titanium evaporators further reduced the pressure to 5.4×10^{-9} Torr. Other vacuum tests are in progress.

Acceptance tests were performed on the Veeco mass spectrometer, which met all performance specifications. These included a mass range of 2 to 50, a resolution of 50, and sensitivity to a partial pressure of 1×10^{-10} Torr (at mass 28).

Studies of the energy distributions of the escaping slow plasma with the slow-ion spectrometer and biased Faraday collectors are being made, and a series of rf measurements are also in progress.

DCX-2 Facility. — Construction of the DCX-2 facility is essentially complete. The vacuum system was assembled and pumped to 5×10^{-6} mm Hg by using only half the diffusion pumps and despite a few troublesome seals. The accelerator tube and manifold has been under vacuum at pressures of $\sim 3 \times 10^{-7}$ mm Hg and has held voltage up to 630 kv. Mechanical alignment of the coils is proceeding, and initial field-shape measurements will shortly be carried out. The injection duct coils are completed, and the entire duct should be available for use during the next report period. The control room is essentially complete, and most interlocks required for safe operation of the facility have been checked.

Vacuum. — Accelerated decomposition tests for a variety of pump oils have been completed. Hexaphenyl polyether, pentaphenyl polyether, Octoil, Octoil-S, and Convoil were studied. The polyethers at 400°C in glass showed little or no discoloration at 200 to 400 hr, but appear to be somewhat light-sensitive. The samples of Octoil, Octoil-S, and Convoil were

decomposed extensively in 1 to 4 hr at 400°C. All the tested oils at 250°C appeared to be unchanged after many hundreds of hours.

Further tests of nude gages have been performed in a system with Octoil rather than Convoil as the diffusion pump fluid. A Bayard-Alpert type of ion gage was modified by addition of a metal screen in place of the glass envelope, and its behavior has been studied.

A titanium evaporation rate monitor was constructed and tested. Further experiments to measure sticking fractions of gases on vapor-deposited metal film have been carried out.

BIOLOGY AND MEDICINE PROGRAM

ENVIRONMENT RADIATION STUDIES

Terrestrial Ecology: Evaluation of Fission Product Distribution and Movement in White Oak Drainage System. — The rate of horizontal groundwater movement in the bed of former White Oak Lake has been measured by the auger-hole method. This method consists in augering a cavity below the water table, allowing it to fill with water, lowering the water level in the hole by pumping, and then observing the rate of rise. Measurements in the upper lake bed show horizontal permeability values of 0.5 and 2.6 ft/day. In the lower lake bed the rate was observed to range from 0.2 to 2.2 ft/day. Additional tests are planned to determine vertical permeability of the lake-bed soil.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND INSTRUMENTATION

Internal Dose Estimation. — Studies of the distribution of uranium in rat kidney at the microscopic level were carried out to obtain more information relating to the phenomenon of prolonged retention of radioactive uranium by kidneys of animals given intravenous administration of a large mass of uranium. The distribution of uranium in the cortex and medulla of rat kidney has been determined at different times after injection and for different injected amounts of uranium by counting tracks observed in autoradiograms. The data from these counts, made by visual method, corroborate the earlier results obtained by radiochemical methods, showing that, with injected activity constant, the greater the amount of natural uranium injected, the greater the fraction retained over prolonged periods of time.

Data on the nonuniform distribution factor obtained from 18 animals indicate that the cortex contained ~1.4 times the average amount in the kidneys.

Studies of leaching of uranium from kidney tissue into formalin fixative and dehydrating solutions indicated that very little uranium was leached.

In observing anatomical changes, measurements of diameters of tubules from kidneys of uranium-injected rats did not significantly differ from controls or correlate with the amount of uranium injected. The diameters of the glomeruli, however, decreased with increasing levels of uranium injected.

TRAINING, EDUCATION, AND INFORMATION PROGRAM

Professional Health Physics Training. — A three-month training period in applied and research health physics was completed by 20 AEC Fellowship students. Twelve of the Fellows received extensions in order to complete the research and thesis requirements for the master of science degree. Of this number, three elected to remain at ORNL to pursue research, and nine returned to Vanderbilt University. Currently, 17 AEC Fellows are enrolled at Vanderbilt University for the 1961–1962 Health Physics Fellowship program.

The Education and Training Section has been responsible for the coordination of programs for both foreign and American visitors. Included are one Brazilian, one Australian, two Frenchmen, two Japanese, and fourteen Americans.

A. L. Mohan from the Atomic Energy Establishment, Trombay, Bombay, India, has completed a one-year training period in health physics. Samuel Helf, Picatinny Arsenal, New Jersey, is presently assigned to the Health Physics Division for one year of training. The programs of these visitors were coordinated by the Education and Training Section.

ISOTOPE DEVELOPMENT PROGRAM

Neutron Activation Analysis Research. — In an investigation concerned with the use of short-half-life radionuclides in neutron activation analysis, methods which do not depend on chemical separation have been employed for determining nanogram amounts of Se, Hf, F, Nb, Y, and V in sample materials by use of neutron-induced, short-lived radionuclides. In each instance, gamma-ray scintillation spectrometry has been used to complete each analysis. When the flux is 6×10^{13} neutrons cm^{-2} sec^{-1} and 100 counts are taken in measuring the gamma radiation, the limits of measurement in nanograms are: Se, 0.2; Hf, 0.02; F, 50; Nb, 5; Y, 200; V, 0.08.

Methods of analysis based on neutron radioactivation have been used to establish the concentration of trace amounts of stable As, O, Br, and Mn in Tc^{99} metal and also stable Ba and Na in Sr^{90} and solutions of other fission products. In this work no interferences were encountered from the radioactive materials initially present in the samples. In the analysis of Tc^{99}, 1 nanogram of As and 10 nanograms each of Br and Na were determined by the use of these methods. Concentrations of oxygen in Tc^{99} of the order of 3% have been measured by the use of the neutron reactions $Li^6(n,\alpha)H^3$ and $O^{16}(H^3,n)F^{18}$. In the analysis of Sr^{90} and solutions of fission products it was found that at least 100 nanograms of Na^{23} and 10 nanograms of Ba^{138} could be estimated by neutron activation.

A neutron radioactivation method of analysis has been developed for the determination of trace quantities of phosphorus in silicon or silicon-bearing materials, utilizing the neutron reaction $P^{31}(n,\gamma)P^{32}$. The P^{32} decays with a half-life of 14.3 days and emits 1.7-Mev beta radiations in its decay. The method consists in irradiating the samples for a short time — no longer than 20 min — at a flux of at least 5×10^{13} neutrons cm^{-2} sec^{-1} and subsequently processing the samples for P^{32}. The beta radioactivity of the P^{32} is measured by a beta counter. When the particular period of irradiation specified is used in the determination of

phosphorus in materials containing large amounts of silicon, interference due to the reactions $Si^{30}(n,\gamma)Si^{31} \xrightarrow[2.6h]{\beta^-,\gamma} P^{31}(n,\gamma)P^{32}$ is minimized. During this irradiation period, less than 10^9 atoms of P^{31} will be produced from the decay of Si^{31}. It is now possible to detect and determine 3.5 nanograms of P^{31} per gram of silicon by this method.

Radioisotope Research and Development. – The 256-w (thermal) SNAP-7A heat source was completed. The source consists of 40,500 curies of $Sr^{90}TiO_3$ in 24 pellets encapsulated in Hastelloy C. The similarly sized SNAP-7C source (described in ORNL-3190) has been inserted in a thermoelectric converter in the customer's plant and is producing about 10 w of electricity, which powers weather instruments and a radio transmitter. Radioisotope sources suitable for standards have been prepared by adsorbing the desired source material on Decalso (a sodium aluminum silicate) and then fusing on a Mullite backing. The sources are very insoluble and are resistant to abrasion.

A system suitable for handling the uranium recovery from HRT fuel shows a gamma decontamination factor of $> 10^7$. The procedure involves a forward extraction into 30% tributyl phosphate and a back extraction of fission products out of the organic phase.

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