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STATUS AND PROGRESS REPORT

MARCH 1966

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OAK RIDGE NATIONAL LABORATORY

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DATE ISSUED

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1966

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

REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

Fast Gas-Cooled Reactor Irradiation Tests. — Irradiation tests are being performed in the ORR poolside facility to support the development of fuel elements for a fast gas-cooled reactor. These capsule tests in a predominantly thermal-neutron spectrum are intended principally to investigate fuel-cladding reactions. The General Atomic fast gas-cooled reactor design (GCFR) is used as a reference in the selection of test parameters, and the test specimens are supplied by General Atomic.

The irradiation of the third capsule, designated P4B-3, is nearing completion, and a fourth, similar, element designated P4B-4 is ready for insertion in the test facility. Both capsules contain two fuel pins. The lower specimens are 4 in. long and are of the fuel-supported, clad type. They contain solid 0.31-in.-diam UO_2 pellets in a 0.015-in.-thick Hastelloy X cladding. The upper fuel pin is of the "flexcan" type, in which the external pressure is hydraulically transmitted through a specially deformed flexible unfueled upper section of the cladding to sodium, which fills the internal voids of the pin. By this means the collapsing forces from external pressure on the cladding are substantially eliminated. The upper element is about 12 in. long. A 4-in. length is fueled with solid pellets; the remaining length constitutes the flexible section. The cladding is 0.010-in.-thick type 304 stainless steel.

The present element is being irradiated at linear heating rates of about 16 kw/ft in the upper pin and about 13.5 kw/ft in the lower pin. The cladding temperature of both pins is about 660°C. The external pressure is 1000 psig. Capsule P4B-3 specimens have been subjected to both thermal cycles (660 to 300°C) and pressure cycles (1000 to 100 psig) at scheduled intervals during the seven-week irradiation period. Capsule P4B-3 will be replaced April 11 by capsule P4B-4, which will operate under the same conditions, except that the linear heating rate of both upper and lower pins will be about 15 kw/ft, and the cladding temperature will be about 705°C for the upper pin and 760°C for the lower pin.

GCR-ORR Loop 1 Irradiations. — The irradiation of experimental fuel assembly 15, which began July 12, 1965, will be terminated April 11, 1966.

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The test element consists of a mixed bed of loose pyrolytic-carbon-coated particles of different sizes and compositions [$(Th,U)C_2$, $(Th,U)O_2$, and ThO_2] in two graphite containers; the upper container is vented to the loop coolant. The upper-region central fuel temperature has been maintained at approximately 1400°C throughout the test. Burnup has reached 11.6% fission of initial heavy-metal atoms.

Fission gas release, expressed as R/B (ratio of release rate to birth rate), has risen rapidly since the burnup reached 10%. The ^{88}Kr release has increased from 2.4×10^{-5} to 1.1×10^{-4} and ^{87}Kr release from 1.3×10^{-5} to 7.3×10^{-5} in going from to 10 to 11% heavy-metal burnup. Measurements for determining the character of the nongaseous activity circulating in the loop have been made. The results indicate the prime activity source to be activated elements from stainless steel in particulate form that are probably corrosion products.

Upon termination of experiment 15 the loop in-core section is to be replaced as a precautionary measure. The in-core section cannot be examined, and the loop has operated continuously for about five years with an accumulated exposure of about 1×10^{22} neutrons/cm^2 (>0.1 Mev).

Compatibility of BeO and Graphite Under Irradiation. — An irradiation test for determining the compatibility of BeO with graphite at elevated temperatures was completed in December 1965, and postirradiation examination has begun. The experimental assembly consisted of BeO and graphite samples nested together, and the areas of contact were known for some samples. The assembly was irradiated for nine months. During the first four months the temperature was 1500°C, but it then slowly dropped, to 1280°C at termination of the irradiation. The fast-flux exposure was about 1.4×10^{21} neutrons/cm^2 (>0.18 Mev).

Postirradiation visual observation of the parts gave no indication of reaction between the BeO and graphite. The graphite parts experienced uniform linear shrinkage of about 1%. The BeO underwent a small amount of swelling, of the order of 0.4 to 0.8%.

Heat Transfer Analysis of Irradiated Fueled-Graphite Spheres. — Data from irradiation tests of 6-cm fueled-graphite spheres were analyzed to obtain thermal conductivities. The values for the fueled core are satisfactorily described as functions of temperature over the range of experimental temperatures (950 to 1350°C) by an equation of the form

$$1/k = a + bT ,$$

where k is thermal conductivity in w cm^{-1} (°C)$^{-1}$, T is temperature in °C, and a and b are constants that appear to depend somewhat on starting materials and fabrication conditions. Three of the instrumented fuel elements with machined shells showed significant decreases in thermal conductivity after the first 10 to 20 days of irradiation. This is attributed to differential shrinkage of the fueled core away from the machined shell.

Values of the constants a and b, for use in the equation above, have been calculated for each 6-cm instrumented sphere in the ORR poolside irradiation series.

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REACTOR FUELS AND MATERIALS

Nondestructive Test Development. — We have continued to improve the design of our phase-sensitive eddy-current instrument for detecting flaws, measuring thickness, and measuring conductivity in metal objects. It now operates at 12 different frequencies from 1 to 5000 kc. The total harmonic distortion is less than 0.5% on most ranges for an output of 1.5 w. This reduction in harmonic distortion allows a greater range of compensation for distance between probe and specimen. The direct-current drift of the instrument has been considerably decreased so that the long-term drift (1 week) represents a 5% conductivity change and the short-term drift (1 hr) represents only a 0.3% change. Until the latest modifications, an oscilloscope was necessary for establishing proper operation. We can now quickly set up the instrument with only a few simple adjustments, using the meter on the instrument. These modifications have significantly increased the range and ease of use of the phase-sensitive eddy-current instrument.

Mechanical Properties. — Structural alloys suitable for application at elevated temperature are being developed for resistance to elevated-temperature irradiation embrittlement. Previously (ORNL-3896, p. 3), laboratory heats of type 304 stainless steel modified with 0.2% Ti showed much better postirradiation ductility than that of unmodified material. A finely dispersed precipitate caused the improvement by preventing accumulations of helium in the grain boundaries. We have now tested two commercial heats of the modified alloy, one similar in composition to our material and the other richer in carbon (0.05% rather than 0.02%). After a preirradiation anneal for 1 hr at 1038°C, both showed better postirradiation ductility than the unmodified alloy but fell considerably short of our laboratory heat. Electron microscopy showed that the heat treatment failed to form the desired intragranular precipitate in the commercial heats.

We are now trying several different heat treatments to develop an intragranular precipitate in the commercial heats. Annealing for 1 hr at 1050, 1100, or 1150°C produces a clean structure with all the carbon in solution. Small strain fields appear when the material is annealed 1 hr at 900°C. Aging for 10 and 100 hr at 900°C or 100 hr at 700°C does produce the precipitate in the low-carbon alloy. Similar aging treatments are now in progress for specimens of the high-carbon alloy.

POWER REACTOR FUEL PROCESSING

Aqueous Process Development: General. — Development of mechanical, nonaqueous, and aqueous steps for head-end processes designed to convert power reactor fuels into aqueous solutions suitable for subsequent fuel decontamination and recovery by the well-established methods of solvent extraction is continuing. During the past period the major efforts were on (1) further development of the grind-leach process for graphite-base HTGR fuel, (2) chemical decladding studies on sintered-aluminum-product-clad (SAP-clad) UC fuel from the HWOCR, (3) hot-cell studies on tritium

and rare-gas fission product release from stainless-steel-clad and Zircaloy-2-clad oxide fuels using the chop-leach process, and (4) feed preparation studies for the Acid Thorex solvent extraction process.

Aqueous Process Development: Grind-Leach Process. — A grind-leach process is being studied as a possible alternative to the burn-leach process for high-temperature gas-cooled reactor fuels that contain pyrolytic-carbon-coated UC_2-ThC_2 or UO_2-ThO_2 particles either as dispersed or as consolidated fuel-bead compacts in graphite. In the grind-leach process, the fuel is crushed to −6 mesh and subsequently ground to −140 mesh to break all the pyrolytic carbon coatings on the fuel particles. The ground fuel is then treated with 13 M HNO_3–0.05 M HF. The undissolved graphite is filtered and washed to permit complete removal of the dissolved uranium and thorium. Recent washing tests, made with ground and leached General Atomic–Peach Bottom ThC_2-UC_2 graphite fuel compacts (3% U, 14% Th, 83% C) showed that extended washing of the leached graphite residue (−140 mesh) is required for maximum thorium and uranium recovery. Subsequent measurements of the permeability of 1- to 2.5-ft-deep beds of the graphite residue to 0.1 M HNO_3 wash solution were well correlated by the equation $V = 0.90(\Delta P/L)(\bar{1}/\mu)$, where V is in gal hr^{-1} ft^{-2}, $\Delta P/L$ is in psi/ft, and μ is in centipoises. The bed depth L can be calculated assuming that the bulk density of the leached graphite is 0.80 g/cm^3. The ranges of experimental values for which the above equation was evaluated were: V, 6 to 80 gal hr^{-1} ft^{-2}; $\Delta P/L$, 6 to 28 psi/ft; μ, 0.32 to 0.96 centipoise. Addition of small amounts of filter aid (20 g of Dicalite 5000 per kg of graphite) increased the permeability by 25 to 70%.

Aqueous Process Development: Advanced Fuels. — Methods are being developed on a laboratory scale for processing fuels for heavy-water-moderated, organic-cooled reactors (HWOCR's). One reference HWOCR fuel is UC clad in SAP (a sintered dispersion of Al_2O_3 in aluminum). Decladding with caustic followed by dissolution in nitric acid appears feasible as a processing method for SAP-clad UC fuel. Tests have shown that during decladding the SAP dissolves rapidly and the unirradiated UC crumbles, but does not react, in the boiling 2 M NaOH–2 M $NaNO_3$ decladding reagent. To simulate the decladding-dissolution process, UC and SAP (at a UC/SAP weight ratio of 8) were digested for 4 hr in the boiling reagent to dissolve the SAP. Only 0.07% of the uranium was solubilized during the decladding step. The residual solids, unreacted UC, dissolved completely in boiling 5 M HNO_3 in 5 hr, yielding a solution 0.5 M in uranium and 2 M in HNO_3 that contained about 42% of the carbide carbon as soluble organic compounds. These organic compounds would probably have to be destroyed before the solution could be used as a satisfactory feed for a solvent extraction process.

Aqueous Process Development: Shear-Leach Process. — In this process, fuels with nitric-acid-insoluble cladding (stainless steel, Zircaloy-2, etc.) are sheared to expose the fuel oxide, metal, or alloy, which is then dissolved in nitric acid or fluoride-catalyzed nitric acid. The cladding is subsequently discarded as a metal waste. Current studies include (1) the repair and modification of the 250-ton shear gibs and liners for additional wear studies and (2) hot-cell determination of tritium and rare-gas fission product release in the shearing operation.

The shear liners were ground smooth to remove all previous scoring and galling marks and were reinstalled on the ram of the shear. Straightening and grinding of the type 420 stainless steel gibs is about 50% complete. A new gib was fabricated from D2 tool steel hardened to a Rockwell C of 59. This gib will be operated against a Stellite 6B liner to evaluate the wearability of this combination of materials. A 173-rod stainless steel Consolidated Edison core B prototype fuel assembly 105 in. long has been filled with porcelain pellets in preparation for performing additional intact shearing and wear tests.

In hot-cell experiments designed to measure the release of tritium and rare-gas fission products to the environment during shearing and subsequent dissolution of gas-tight, metal-clad oxide fuels, several stainless-steel-clad prototype Consolidated Edison fuel samples containing pellets (96% of theoretical density) of 96% ThO_2–4% highly enriched UO_2 irradiated to 25,000 Mwd/metric ton were sheared in special hot-cell equipment. During multiple shearing of the specimens into 1/2-in. segments, only 0.26% of the tritium and 1.07% of the ^{85}Kr, based on calculated fission yields, were released. When the exposed fuel core was dissolved in boiling 13 M HNO_3–0.1 M $Al(NO_3)_3$–0.05 M HF, an additional 0.26% of the tritium was released to the off-gas. The balance of the ^{85}Kr was released during dissolution but was not measured. During acid removal and boildown of the dissolver solution, about 64% of the original tritium was carried overhead. After rectification of the acid, this tritium would eventually be carried to a low- or intermediate-level waste stream. Extrapolation of these data indicates that 1 curie of tritium would be released in the head-end off-gas per metric ton of heavy metals sheared and dissolved. This would be 0.5% of the maximum amount of tritium that could safely be released to the atmosphere at 1/10 the MPC_A from a 6-metric-ton-per-day processing plant.

Similar experiments were also made with prototype PWR fuel specimens containing UO_2 pellets of 93% theoretical density clad in Zircaloy-2 and irradiated to 40,000 Mwd/metric ton. These fuel pins were also sheared into 1/2-in. segments, and the exposed fuel was dissolved in 4 M HNO_3. Shearing released 7.43% of the theoretical fission yield of tritium; however, only 0.13% of the total tritium was carried by the dissolution off-gas. Total release of tritium during the shearing and leaching of 1 metric ton of fuel would amount to about 37 curies.

Aqueous Process Development: Feed Adjustment Studies. — To determine quantitatively the amount and composition of residues produced in the head-end steps of the shear-leach process, the radioactive leach solution produced by dissolution of the Consolidated Edison core material irradiated to 25,000 Mwd per metric ton of uranium plus thorium (see previous section) was filtered, washed, dried, and analyzed. The filtrate was then boiled down to produce an acid-deficient Acid Thorex solvent extraction feed; in this step a second precipitate formed, and this was also filtered and analyzed. The leaching residue amounted to 0.58 wt % of the original fuel and contained insoluble thorium equivalent to a loss of 0.05%. The bulk of this incompletely analyzed residue contained silica, alumina, stainless steel corrosion products, and fission product Mo, Zr, Nb, Sb, and Ru. The boildown residue represented about 0.6 wt % of the original fuel and contained insoluble thorium equivalent to a loss of 0.05%, along with insoluble compounds of molybdenum and zirconium.

Fluoride Volatility Processing: Fluidized-Bed Decladding and Fluorination Tests. — In the volatility process for UO_2 fuels clad with Zircaloy or stainless steel, the Zircaloy is removed by reaction with HCl, or perhaps a mixture of HF and oxygen. The latter mixture is used for stainless-steel-clad fuels, converting the components to oxides. The decladding steps have previously been performed in a fluidized bed of alumina, but investigations were begun recently to determine the feasibility of using fluidized beds of "stainless steel fluorides" and ZrF_4 for fuels clad with stainless steel and Zircaloy respectively. Elimination of the alumina would decrease the volume of highly radioactive waste.

In a recent experiment in a 1-in.-OD fluidized-bed reactor, 13.3 g of UO_2 was oxidized to U_3O_8 at 550°C in a 45-g ZrF_4 bed. After fluorination for 1 hr at 450°C and then for 3 hr at 550°C, the residual uranium concentration was only 0.0074%, corresponding to a 99.97% removal, which is superior to that obtained with alumina beds. In similar tests with alumina beds, a final uranium concentration of about 0.03%, for a removal of about 99.9%, was obtained. Other test results indicated that when ZrF_4 is used the final fluorination to recover plutonium can possibly be done with a settled bed and consequently a much lower flow of fluorine. This prediction is based on the absence of sintering during uranium-volatilization tests when a settled bed of ZrF_4 was fluorinated at either 450 or 550°C. Plutonium retention in a settled bed has yet to be tested. If the ZrF_4 is formed in a two-zone reactor by contacting the Zircaloy cladding with HCl in the lower section, followed by reaction of the $ZrCl_4$ with HF in the upper section, the waste stream will contain only one major component, with a volume dependent on the weight of the cladding processed. This two-zone decladding scheme will also be tested.

In other tests with the 1-in.-OD reactor, decladding of stainless-steel-clad UO_2 with mixtures of HF and oxygen in a bed of pure stainless steel fluorides was successful and was somewhat faster than would be expected with a fluidized bed of alumina. Oxidation of the stainless steel was apparently promoted to some extent also by the presence of the solid fluorides. Volatilization of about 99.9% of the uranium from this bed was possible with 1 hr of fluorination at 450°C and 3 hr at 550°C.

The chromium component that volatilizes during the reaction of stainless steel with HF-O_2 may present a problem. It is volatilized in an undetermined chemical form when the stainless steel reaction products are contacted with fluorine at temperatures of 450 to 600°C. Unfortunately, the chromium compound formed has only moderate volatility at 100°C (the temperature of the off-gas lines), and most of it is deposited in the first section of the off-gas system. Subsequently, some chromium is transferred to the cold traps, and chromium in lines and cold traps may retain some of the plutonium fluoride.

Fluoride Volatility Processing: In-Line Analysis of Process Streams. — The need for continuous analysis of process gas streams as a means of providing process-control information has led to the investigation of spectrophotometers. A calibration test of the ultraviolet spectrophotometer intended for monitoring UF_6 content in the fluorination off-gas stream resulted in a molar absorptivity coefficient for UF_6 of 3200 $mole^{-1}$ cm^{-1} at 216 mμ. This value compares with a previously reported 3610 ± 1000 $mole^{-1}$ cm^{-1} read at 214 mμ on a different spectrophotometer (ORNL-3830, p. 89). Five tests were made at 60°C and 1 psia in a cell with a

0.5-mm path length and sapphire windows. Test concentrations ranged from 100 down to 37 vol % UF_6. The method appears promising for pilot-plant use.

A method for rapidly determining the plutonium content of the solid waste leaving the primary reactor would be useful in pilot-plant work. A count of the delayed neutron flux resulting from neutron irradiation of plutonium has been proposed for this purpose. A preliminary test was made by irradiating a mixture of Al_2O_3 and uranium oxide of known ^{235}U content; a count of 150 to 200 neutrons per milligram of ^{235}U resulted. The presence of 0.02% plutonium in the waste alumina bed (corresponding to about 97% recovery after a single pass) would yield about 40,000 delayed neutron counts per 20 sec from 1 kg of waste. A calculation of the neutron flux resulting from transplutonic elements present in the waste, from both fission and (α,n) reactions, indicates a flux of 10^7 to 10^8 neutrons per 20 sec from 1 kg of waste. The statistical variation of the background flux is thus of the same order of magnitude as the signal. These findings indicate that the method in its present form is not sensitive enough.

Fluoride Volatility Processing: Chromium Fluoride Studies. — In processing stainless-steel-matrix fuels by the fluidized-bed volatility process, chromium is carried with the UF_6 product gas stream partly as CrO_2F_2. Effective separation from UF_6 on the NaF sorption beds is dependent upon the difference in stability of the association complexes produced in these beds. We have therefore measured the dissociation pressures as an aid in selecting optimal operating conditions for recovering pure UF_6 from NaF sorber beds. Chromyl fluoride was synthesized by reaction of CrO_3 with CoF_3 and sorbed on a bed of activated NaF. Equilibrium pressures for the reaction

$$CrO_3(s) + 2/3\ CoF_3(s) \rightleftharpoons CrO_2F_2(g) + 1/3\ Co_2O_3(s)$$

and the dissociation pressures of the NaF-CrO_2F_2 complex were determined by transpiration with helium gas.

We found that the equilibrium pressure of CrO_2F_2 in the synthesis reaction could be expressed as log P (mm Hg) = $6.98 - 2890/T_{°K}$. Since the equilibrium constant K is equal to the CrO_2F_2 pressure in atmospheres, log K = $4.10 - 2890/T$. From this expression, the free energy of the synthesis reaction was calculated to be ΔF^0 (cal/mole) = $13{,}220 - 18.7T$.

At temperatures of 296 and 441°K, equilibrium pressures over the NaF-CrO_2F_2 complex were found to be 0.00153 and 0.00656 mm Hg respectively; dissociation pressure may therefore be expressed as log P (mm Hg) = $-0.865 - 579/T$. In contrast, the pressure of UF_6 over $2NaF \cdot UF_6$ is expressed by log P (mm Hg) = $10.88 - 5090/T$. From these expressions, note that the dissociation pressures are equal at 375°K; above 400°K the UF_6 pressure is more than tenfold that of CrO_2F_2, and, consequently, UF_6 may be desorbed from NaF in the usual purification procedures (about 573°K) without significant contamination by chromium.

Fluidized-Bed Volatility Pilot Plant (FBVPP). — The FBVPP will be installed in cells 1, 2, and 3 of Building 3019 to study the processing of spent UO_2 power-reactor fuel clad in either zirconium or stainless steel.

For phase 1 (decladding, converting to oxide, fluorinating, collecting UF_6-PuF_6 in cold traps), design of the process vessels, shielding, and fuel-charging equipment is nearly complete. Design continues on process piping, electrical requirements, other mechanical handling devices, instruments, and the in-cell ventilation system. Procurement of instruments, electrical gear, furnaces, filter elements, refrigeration units, shielding, and nickel and Monel plate, sheet, and tubing is proceeding actively. Fabrication or assembly of the fluorine compressor and its drive unit is very nearly complete, the internal fins have been welded to the half-shells of both cold traps, and welding together of the half-shells of one trap has begun. The pyrohydrolyzer, the surge tank and scrub tower for HF and fluorine disposal, and several of the primary and backup filters are being made. Fabrication of the primary reactor will start as soon as approval can be obtained to deviate from the material requirements of the Unfired Pressure Vessel Code; otherwise, new material must be ordered.

Some of the cells in Building 3019 are being cleared for installation of FBVPP equipment. The remaining equipment was removed from cell 3, and cell decontamination was started. Equipment removal from cell 2 continued. Equipment removal from cell 1-A is delayed, pending reduction of radiation level. Cleanup of cells 1 and 1-A continued.

A request was approved for $99,000 to modify the cell-ventilation system outside Building 3019 so that a pressure difference of −4.6 in. H_2O will exist at the air exit of cells 2 and 3. A preliminary proposal is being prepared.

As a part of process design for phase 2 (separating and purifying the UF_6 and PuF_6), a digital computer program was written to perform the calculations necessary in the analysis of a dynamic-batch-distillation—thermal-decomposition system. Decomposition of PuF_6 to PuF_4 was assumed to occur in a decomposer located in a loop off the fractionating tower, with no decomposition in the tower. For one set of conditions where NbF_5 was added as a reboiler heel (about 2.5 mole % of the original charge), results of calculations showed that the reboiler could be operated below 100°C. This low an operating temperature should result in very slow thermal decomposition of PuF_6 to PuF_4 in the reboiler and in the fractionating tower.

FUEL-CYCLE STUDIES

Thorium Dicarbide Studies. — Two different approaches to the preparation of ThC_2 are being studied. One is based on mixing ThO_2 and carbon sols, preparing a gel from the sol, and firing the gel in vacuum at temperatures in the vicinity of 2000°C to convert the ThO_2 to ThC_2. The other is based on heating ThO_2 gel microspheres in a bed of carbon black, terminating at temperatures in the range of 2150 to 2250°C. The mixed-sol approach leads to a porous ThC_2 product (<90% of theoretical density), whereas the ThO_2—carbon-black approach leads to a theoretically dense product. In the current stage of development, both ThC_2 products contain unconverted ThO_2. In the mixed-sol approach, there is also unreacted free carbon to the extent of 0.5 to 2%. The lower free carbon contents are

associated with samples fired in a vacuum, while the higher percentages are found in samples fired in argon. Although we do not believe that these small amounts of carbon are necessarily deleterious, we are seeking ways to reduce the amount.

In the ThO_2–carbon-black approach, we have mounting evidence that effects of small amounts of carbon left in the spheres from the alcohol used in the sphere-forming operation are different from the effects of carbon derived from carbon black. In general, it appears that residual carbon interferes with the conversion step and should be eliminated, if possible. Several approaches are being studied for its removal, but results are not yet completely analyzed.

Coated-Particle Development Laboratory. — The Coated-Particle Development Laboratory is for use in developing remotely operable methods of producing microspheres and coating them with pyrolytically deposited graphite. The microspheres may be oxides or carbides of thorium, uranium, or thorium-uranium.

Oxide microspheres are prepared by a method based on dispersing a hydrosol into droplets at the top of a column of immiscible organic liquid. The organic liquid extracts water from the descending droplets, resulting in gelled spherules which are recovered at the bottom of the column. They are then dried and fired to dense microspheres. Development work is being carried out to devise ways to remotely conduct sol feeding and dispersion, gel drying and firing, and column operation itself, which requires measuring the column inventory and regenerating and recycling the organic liquid. The microspheres being produced are used in pyrolytic-carbon coating studies. A continuous transfer system for moving gel microspheres from the sphere-forming column to the driers would be preferable to the present system, which operates intermittently; so far, however, attempts to operate continuously have been unsuccessful.

Coating studies were made to evaluate coating reproducibility on 1-kg batches, to study scaleup to 2 kg per batch, and to study coating at 2000°C for producing high-density coatings (2 g/cm^3). Substantial variations in coating thickness, in rate and efficiency, and in coating density were observed. The variations are not related in any obvious way to the measured coating conditions.

We are attempting to develop an in-line process monitor for measuring average carbon-coating thickness on small samples of ThO_2 microspheres. An x-ray attenuation method based on attenuation of ^{241}Am 60-kw x rays is being used. Americium-241 is a more stable source of x rays than a conventional source, but the low-intensity source in use necessitates a thin sample of microspheres, and it was feared that the resultant variation from one sample to the next might be intolerable. However, several loadings have been made with the thin-sample containers, and loading variations are negligible. Thus, it appears that ^{241}Am is suitable for use with 230-μ-diam particles coated with carbon and in 1/16-in.-thick containers.

Sintering Studies on Thoria and Urania Sol-Gel Microspheres. — An understanding of the drying and sintering behaviors of thoria and urania microspheres prepared by the sol-gel process is of importance because the density, carbon content, strength, and residual gas content of the resultant microspheres depend so strongly on the drying and sintering methods

used. During the past month we carried out additional experiments on various procedures. We studied the removal of the residual organic gelling alcohol by steam stripping a bed of wet gel microspheres, using a steam flow rate of about 2 g/min at 200 and 400°C for times ranging from 15 min to 2 hr. With steam at 200°C, the residual carbon was rapidly reduced to about 0.5 wt %, and no additional loss was observed, even after 2 hr of stripping. With steam at 400°C, the residual carbon content was reduced to less than 0.025% in both thoria and urania. Microspheres that had been stripped at 400°C for more than 1 hr showed severe cracking after exposure to moist air. Similar behavior has been observed before in sintering studies with thoria.

Extrusion Fabrication of Sol-Gel Materials. — We are investigating extrusion fabrication of small-diameter ceramic nuclear fuel pieces to find more economical means of remote fabrication of sol-gel-derived materials. The present method is based on extruding a clay of oxides prepared from sol-gel thoria or thoria—3% urania. The clays contain 10 to 12 g-moles of oxide per liter. Good extrusion and well-formed pieces have been observed. The principal problem has been with cracking of the fired bodies. It was first thought that by controlling the water content of the clays, the cracking might be reduced, but results so far have not been encouraging. It now appears, however, that additions of ThO_2 powder to the clay may reduce or eliminate the internal cracking. Initial work indicates that such additions may reduce the density obtainable in the fired product. Work is continuing to define the range of densities and the dimensional tolerances obtainable.

Vibrational Packing of Microspheres. — Packing microspheres of several diameters into tubes by low-energy vibration shows promise as a means of economically making fuel rods. Studies are being made with ThO_2 microspheres of several sizes. Important factors for optimum packing are the ratio of the diameters of the microspheres and the ratio of the diameter of the tube to that of the largest microspheres. Results so far indicate that a vibration frequency of 60 cps is satisfactory. The use of a follower rod increases the packed density from 82.5% of the theoretical density of the ThO_2 to 83.5% and prevents possible segregation of the different size fractions of microspheres under prolonged vibration times. However, the follower rod used so far has stuck in the tube occasionally, and unless this possibility can be eliminated, such a device may not be practical in a remotely operated facility.

^{233}U Storage and Distribution. — ORNL serves as a storage, purification, and distribution center for ^{233}U. At present, about 177 kg of ^{233}U is in storage, of which about 49 kg contains less than 5 ppm of ^{232}U. The principal isotopic contaminant of this 49 kg is ^{238}U, which varies from 6.6% to less than 2% in the various lots.

Both the 1 kg/day solvent extraction facility and the Kilorod batch dissolver were operated last month. Operation of the systems was good, although uranium loss to the product evaporator was high (1.3%). Reflux water to the evaporator will be increased to reduce this loss.

One of the borosilicate-glass Raschig rings used in the solution storage tank to prevent criticality was removed after three years of being covered with solution. It was analyzed for boron content to determine the loss, if any, and there was none.

Thorium-Uranium Recycle Development Facility. — This facility is being built to provide shielded space for the development of remote processing and fabrication procedures for Th-^{233}U fuels of various types. Sufficient shielding (5-1/2 ft of normal concrete) is being provided to accommodate highly irradiated fuel assemblies after a three-month decay period.

Building construction is now approximately 49% complete. Cell lining work is progressing satisfactorily and is up in all cells to approximately the first floor level. Painting of the exterior of the building has begun. Procurement of equipment is progressing satisfactorily. The shielding-door procurement problem appears to have been solved by a plan suggested by the door fabricator, and delivery dates can probably be met.

Drawings of liquid handling equipment are almost complete. Prototype equipment is being designed for handling a Grayloc clamp support, for bringing services from outside the cell through a 4-in. bent tube to an equipment frame, for washing precipitated urania by countercurrent decantation, and for separating microspheres from the drying alcohol.

Sol Preparation Studies. — A variety of reactor fuel materials can be prepared by the sol-gel process. These include thoria, urania, plutonia, and their mixtures. In present studies we are concentrating on an approach to preparing mixed oxides whereby sols are mixed, since this would be a very convenient approach to use with the variety of fuel compositions we are considering in our development program.

A small pilot-plant facility consisting of three glove boxes was assembled for the formation of plutonia sol, mixing it with urania sol, and forming 20% PuO_2–80% UO_2 microspheres. The first step in the sol-forming operation was carried out in this equipment to produce 50 g of plutonia sol. This sol was used to develop the required baking and redispersion steps, and to form experimental batches of PuO_2-UO_2 spheres.

Thoria sol derived from thorium hydroxide prepared by ammonia precipitation of thorium from nitrate solution was mixed with sol derived from thoria prepared by steam denitration of thorium nitrate, in an attempt to understand the effect of crystallite size on product properties. Crystallite size of the precipitated thoria is <30 A; that of steam-denitrated thoria is about 80 A. It was found that microspheres prepared from sol containing more than 5% of the <30-A-crystallite sol cracked or shattered on firing, and that the fraction of spheres which cracked increased with the percentage of this precipitated-thoria sol.

In other studies, porous thoria microspheres were prepared by mixing thorium nitrate solution with ammonium molybdate solution to produce a mixed sol. The sol composition was in the range Mo/Th = 0.4 to 0.6 and was used in forming gel microspheres which were fired in argon at 1300°C for 1 hr. Porosity of the ThO_2 product increased with increasing molybdenum content in the sol. Porous ThO_2–20% UO_2 microspheres were successfully prepared by essentially the same procedure.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — The reactor remains in a partially shutdown condition. Coolant salt circulated without interruption throughout the month, but the fuel loop was empty. Meanwhile we concentrated on

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finding out more about the nature of the trouble in the fuel off-gas system and on measures to cope with the problem.

The last unexplored restriction in the off-gas system, at the inlet end of the charcoal beds, was explained when we removed one of the inlet valves and found a deposit in the trim which could account for the excessive pressure drop.

Analysis of material found in the valves, filter, and flow restrictor was almost completed (see the table in the following subsection). It proved to be predominantly organic material, but some samples also contained salt with a composition like that of the vapor from the fuel. The organic generally had the appearance of dried varnish, although there were a few drops of viscous oil in the pressure control valve and the nearby filter housing. The total quantity observed was minute (a few cubic centimeters) and appeared only where the gas stream was highly turbulent or impinged on the surface. Activities associated with the deposits were mostly tellurium, iodine, and the daughters of xenon and krypton. Experiments showed that helium flowing from the off-gas line contained measurable concentrations of hydrocarbons, which increased by an order of magnitude when the reactor cell was allowed to heat up from 120°F to 170°F. (It was calculated that the off-gas holdup volume in the reactor cell heated up this much due to activity in the off-gas during the brief 1-Mw operation.)

We inferred from the evidence that the offending material was probably produced by radiation polymerization of oil vapors or liquid in the off-gas stream. We suspected that the oil came either from a residue in the holdup volume or from leakage into the fuel pump bowl which somehow escaped thermal decomposition. The oil level in the fuel-pump oil system had been measured each shift in the months preceding the shutdown, and the records showed there had been no gross leakage, but gradual leakage of 1 or 2 liters could not have been detected; therefore, we opened the off-gas line in the reactor cell near the pump bowl for inspection. There was some dust between the flange faces and a faint film of dust on the surfaces of the pipe. Analysis of the dust indicated that it was mostly frozen salt, but it also contained particles of organic material. There was no evidence of much oil leakage and decomposition in the pump bowl. After the inspection, helium was blown through the line at 15 times the normal velocity to remove any loose dust; little came out.

We concluded that a small amount of oil from the holdup volume or from the pump had caused the trouble and that we might continue to have small amounts of material in the off-gas. Therefore, we decided to install a trap in the off-gas line to remove particles, mist, and organic vapors. In addition, we replaced the five hand valves at the charcoal bed inlets with larger valves with on-off trim and installed a larger hand valve upstream of the trap. The trap consists of two sintered fibrous metal filters followed by a bed of Fiberfrax ceramic filters, which should remove essentially all liquid and solid particles. Vapors will be adsorbed on charcoal packed into 13 ft of 1-in. pipe.

The damaged motor on the drain-cell cooler was replaced, and the cooler was reinstalled. While the reactor cell was open for the off-gas line inspection, a slight drip of water was observed from one of the reactor-cell coolers. The cooler was removed, the leak was repaired, and the cooler was reinstalled.

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Chemical Development. — Additional specimens of varnish-like deposits were scraped from several locations within the MSRE fuel off-gas system where flow restrictions were previously noted. Examination of the solids with the petrographic microscope showed them to consist of amber to amber-red translucent amorphous phases. Spectrochemical, infrared-absorption, and gamma-activity analyses were also performed in attempts to determine the nature of the materials that produced the flow restrictions. Results are summarized in the table below.

Results of Analyses of Samples from MSRE Off-Gas System

Sample Location	Refractive Index	Species Identified		
		Spectrochemical	Gamma Scan	Infrared
Capillary tube flow restrictor in pressure equalizer line 521	1.520	Li, Be, Zr		
Housing for capillary tube flow restrictor	1.540			
Stem of pressure control valve, PCV-522, in main off-gas line 522	1.540	Be, Zr	^{89}Sr, ^{140}Ba, ^{140}La	
Seat from PCV-522	1.544	Be, Zr	^{89}Sr, ^{140}Ba, ^{140}La	
Oil removed from PCV-522	1.509		^{89}Sr, ^{140}Ba, ^{140}La	
Filter from line 522	1.524–1.526	Li, Be, Zr	^{103}Ru, ^{140}Ba, ^{140}La, ^{95}Zr	$-CH_2$, $=CO$, $-OH$
Stem of valve HCV-521 in main off-gas line at carbon bed	1.526	Be, Zr	^{132}Te, ^{140}Ba, ^{140}La, ^{95}Zr, ^{130}Cs	
Fuel pump oil	1.473			$-CH_2$

These and additional data indicate that the flow restriction was due partly to thermal decomposition and partly to cross-linked polymerization of hydrocarbon oil, possibly the fuel pump oil.

Laboratory experiments have been performed to simulate, in the absence of radiation, the effects of injecting MSRE pump lubricating oil (Gulfspin-35) into the pump bowl at a rate of 16 cm^3/day. Such a flow of oil was injected into a gas stream of 4 liters of helium per minute (MSRE off-gas rate), and the mixture was passed through a nickel cracking chamber at 600 to 640°C. A definite oil fog was produced at the exit, and an appreciable fraction of the oil was decomposed into lighter hydrocarbons.

Several different mechanical filtration media were tested for removing the condensed phases at room temperature; increasing efficiency was found in the order: coarse nickel wool, No. 1 steel wool, No. 00 steel wool, Pyrex glass wool. The Pyrex wool proved very effective without contributing any substantial pressure drop in the system.

Downstream of the mechanical filtration devices, tests were made of 1-in.-diam charcoal beds at 100°C (conditions calculated for MSRE) to see

whether they would effectively remove the higher-molecular-weight volatile hydrocarbons which might be expected to polymerize to gummy products under the effects of radiation. PCB-6 × 16 charcoal (the same as used in the MSRE off-gas beds) appeared very effective in that its capacity for higher hydrocarbons was saturated at only about 1 in. per 10 hr. Thus a proposed 13-ft bed for the MSRE, if exposed to the approximately 1000 ppm of mixed volatile hydrocarbons produced in our tests, would be expected to hold up the higher-molecular-weight hydrocarbons (above C-6) for some 1500 hr. Further tests are in progress with mechanical filters planned for use in the MSRE and with charcoal beds at lower temperatures.

Radiation Effects. — Tests of the heater-cooler assembly for the in-pile experiment revealed that the unit was unsatisfactory for in-pile use. This assembly was constructed using Zircaloy-2 cooling coils (1/4-in. by 0.049-in.-wall tubing), and the heating coils and tubing were attached to the autoclave by flame-spraying with Zircaloy-2. (Previous assemblies were flame-sprayed with nickel.) The Zircaloy-2 deteriorated when heated, possibly from oxygen contamination in the argon cover gas used in the tests. Also, cooling capacity appeared marginal.

Subsequently, the heater-cooler and the main autoclave body were redesigned to use stainless steel cooling coils and flame-sprayed nickel to attach cooling coils and heaters to the body. By placing the cooling coils adjacent to the main body of the autoclave, which was grooved to accommodate the coils, the total thickness of the assembly wall and heater-cooler jacket was reduced from ~9/16 in. to ~7/16 in. This unit has been fabricated for assembly into the thermal loop experiment.

Assembly of the auxiliary equipment for the experiment has continued with no undue delay. It is anticipated that all equipment needed for modifications to beam hole HN-1 will be available by April 10, when the modifications are scheduled to begin.

MSR Processing: Theoretical Density and Settling of Beryllides. — Among the possible methods of removing rare-earth fission products from the fuel salt of a molten-salt breeder reactor (MSBR) is precipitation as intermetallic beryllides. The collection of this precipitate by dissolving it in a molten metal phase or by filtering it has been considered. An alternative is sedimentation. Estimates of the theoretical densities of intermetallic beryllides of the type $LnBe_{13}$ were made for the range of lanthanides from La to Eu. Values of $\rho_{25°}$ calculated from the face-centered cubic lattice parameters ranged from 3.1 g/cm^3 for $LaBe_{13}$ to 3.39 for $EuBe_{13}$. Settling rates in molten Li_2BeF_4 at 600°C calculated from these data were 0.60 cm/min for 40-μ $LaBe_{13}$ particles or 0.036 cm/min for 10-μ particles. Thus, unless rather large particles are grown, the physical separation of the lanthanides by sedimentation of their beryllides may be too slow even if they are deposited in particles of near theoretical density.

MSR Processing: Removal of Fission Product Zirconium. — It has been demonstrated that beryllium metal may serve as a holding reductant to retain zirconium in the still pot as free metal during vacuum-distillation recovery of MSBR solvent salt. This possibility was suggested by results of a reduction-filtration experiment made at 1000°C, the proposed still-pot temperature, in which 91% zirconium removal [DF(Zr) = 11] was achieved by using only 16 times the theoretically required quantity of beryllium powder as reductant.

Analysis of Radioactive MSRE Fuels. — Fabrication of the apparatus for remotely analyzing radioactive MSRE salt samples for oxide content was completed. It was installed in cell 3 of the High-Radiation-Level Analytical Laboratory (Building 2026). Support equipment was placed in the cell access and operating areas. Fluoride salt samples and tin oxide standards were analyzed to check the apparatus and familiarize the HRLAL personnel with the method. The results were satisfactory, and the physical manipulations were performed adequately.

Laboratory tests of the apparatus for the determination of oxide in radioactive MSRE samples were completed. Individual components were tested exhaustively and, when necessary, were modified or replaced. A final check of the splitter and cell was made by injecting a known quantity of water into the flow system; quantitative recovery of water resulted. The entire system was then checked out by analyzing synthetic fuel-salt samples. Also, one flush salt sample was analyzed and found to contain 38 ppm oxide.

The apparatus was then installed in the HRLAL and was checked out by analyzing synthetic fuel salt samples, with satisfactory results. An additional reactor flush salt sample was analyzed and found to contain 25 ppm oxide. This value compares favorably with the previous value, 38 ppm, obtained on the bench top.

A method for verifying the performance of the capillary gas stream splitter and the water electrolysis cell in the oxide apparatus was developed. A tin capsule containing a known amount of SnO_2 is heated to 550°C in the hydrofluorinator as hydrogen is passed through the system. The SnO_2 is reduced to the metal, and the water formed passes on to the electrolysis cell. One run of an SnO_2 standard in the hot-cell facility has shown the oxide recovery to be quantitative, indicating that the equipment is performing satisfactorily.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. — The fuel element producer is now achieving his goal of two assemblies a month. With the exception of some small changes in welding parameters, to give improved dimensional stability, the fabrication procedures now appear to be acceptable. During this month the fuel plate rejection rate has dropped below 10%.

To improve the water gap spacing and to increase the rigidity of the fuel plates, it would be desirable to have a means of adding longitudinal spacers to the water channels. Such spacers would have to be firmly held by plates on each side if abrasion is to be avoided. Two techniques are being pursued for attaching such spacers. In the first, wires are drawn through a completed element; in the second, spacers are ultrasonically welded to a plate. With the first technique, wires are held in place by a groove cut into both plates by drawing a wire with an enlarged area or a carbide ball through the channel. The ball or enlarged area is 56 mils in diameter, resulting in a 3-mil groove. The spacer wire, which is 55 mils in diameter, is then drawn into the same groove. Such a technique has been demonstrated to work with single channels, and samples have been prepared for flow testing.

Design. — A poison injection system is being added to the primary coolant system for emergency control of the reactor. A solution of $Cd(NO_3)_2$ will be gravity fed into the suction manifold of the pressurizer pump, which in turn injects the solution into the circulating main coolant stream. The entire system will be located inside the reactor building, with the storage tank at the level of the control room. Provision for both manual and remote control of the injection valves is made. Manual operation will be at the valve location near the pressurizer pumps on the ground floor. Remote control would be from the control room level. Possibilities of inadvertent injection are being reduced in the control considerations.

Some portions of the target holders are being redesigned to provide a more rugged assembly, which provides added protection for the target rods during handling and loading operations. The six tubes supporting the target-support grid are replaced with a perforated cylinder completely encompassing the bundle of target rods, so that the individual rods cannot spill out the side of the holder when inadvertently lifted sufficiently to be dislodged from their locating sockets. This change is being accomplished without change of the metal-to-water ratio in the target region and is expected to provide a more uniform flux distribution. An extension of the upper end of the target holder provides protection for the target rods; it also provides an internal groove for attachment of a smaller handling tool, allowing improved visibility needed for safe handling.

Equipment is being fabricated for positioning vibration detection instruments immediately above the inner fuel element, in addition to the original location near the vertical center of the tower. This mounting eliminates the flow disturbances in the main coolant stream caused by the original instrument mounting. An additional facet of the next vibration test will be checking the effect of spring loading the tower against the top head of the vessel. Analysis of the previously measured movements of the tower indicates that they were caused by or allowed by an instability of the upper end of the inner fuel element, rather than being due to induced vibrations.

Reactor Analysis. — The HFIR is presently operating at 20 Mw, awaiting approval by the Advisory Committee on Reactor Safeguards (ACRS) to proceed with the planned escalation to 100 Mw. All of the "zero"-power experiments have been completed, and the reactor has been operated up to 20 Mw with no indication of significant difficulties.

Nuclear transient calculations have recently been made in which reactivity feedback from steam formation was included. The correlation used for steam void fraction is that proposed by Griffith, Clark, and Rohsenow (MIT). In this correlation it is presumed that the void fraction is proportional to the square of the hydrodynamic boundary layer and thus inversely proportional to the square of the coolant heat transfer coefficient; in addition, the void fraction is directly proportional to the difference between the total heat flux and the incipient boiling heat flux. If it is assumed that the coolant heat transfer coefficient does not increase significantly as the coolant film changes from the subcooled to the nucleate boiling condition for high coolant velocity (~50 fps) conditions, then the steam void fraction becomes approximately inversely proportional to the coolant heat transfer coefficient and thus inversely proportional to the coolant velocity.

In the HFIR analog transient analysis the fuel region was divided into five axial regions, with proper weighting factors for power density and reactivity worths. Results of the calculations show that steam voids are very effective in terminating HFIR excursions when the coolant velocity is about 10% of normal. For full flow conditions the effect is very small.

According to the above correlation the steam void fraction is also inversely proportional to the bulk water subcooling (ΔT_s). Thus, reducing the system pressure will improve the void feedback. However, for small ΔT_s (less than about 30°F for HFIR) the above correlation is no longer applicable, and a relatively complicated one is required. Furthermore, reducing ΔT_s reduces the burnout heat flux. Obviously, the determination of an optimum or even near-optimum condition would be extremely difficult in view of all the uncertainties associated with the present state of the art. Thus there are no plans at this time to pursue this investigation further. Instead, it is concluded, as before, that within the range of credible accidents the HFIR is quite safe, and more specifically that the HFIR can withstand the maximum credible reactivity accident of 1.3% $\Delta k/k$ on a 30-msec ramp with essentially no damage to the fuel elements and flux-trap target.

In connection with the mechanical integrity of the fuel element, the entire heat transfer and mechanical analysis is being reviewed in order to incorporate new information. A specific field of interest is that associated with the axial buckling of the fuel plate. At present we are using a much refined experimental apparatus, compared with that previously used, for investigating buckling. Thus far the buckling wavelength appears to be about twice the length of the plate rather than about one-tenth of the length of the plate as had previously been determined; furthermore, the plate deflection is always in the same direction, a very favorable situation. However, because of the infancy of our program these results must be considered to be very tentative.

Since there is a definite possibility that the problem of fuel plate buckling as a result of thermal and/or radiation damage could be more severe than anticipated, the incorporation of longitudinal spacers in the coolant channels is being considered. The present program includes an investigation of fabrication techniques and heat transfer characteristics.

Safety Analysis. — Work is in progress to obtain answers to a series of questions submitted by the ACRS following their February meeting with members of the HFIR project. These questions relate to the consequences of reactivity accidents more severe than those previously considered. The problems posed may be divided into four areas as follows:

1. A study of the various mechanisms which may either enhance or inhibit the magnitude of a nuclear excursion.
2. An investigation of the role of the metal-water reaction in this connection.
3. Estimation of the mechanical energy partition following a reactor excursion.
4. Calculation of the effect of these reactivity accidents on the integrity of the secondary containment system.

The first area has been investigated in detail using a somewhat more sophisticated reactivity model than that previously employed; however,

this model is not capable of taking into account the consequences of core disassembly. At present efforts are being made, based upon rather broad physical principles, to establish an upper limit to the magnitude of possible energy releases.

The Al-H_2O reaction has been investigated in detail, and it has been determined that this reaction is of little consequence unless the initiating nuclear excursion releases 175 Mwsec or more. Calculations of the effect of various energy releases on the pressure vessel are under way. Application of these results, however, must await the completion of the work in the first area.

The ability of the containment structure to withstand pressure has been recalculated, and once the initiating accident is established it will be possible to estimate the consequences.

Instrumentation and Controls. — An all-solid-state electrometer amplifier was designed, tested, and installed in the HFIR faulty-fuel-element detector system. This amplifier, designated Q-2637, is capable of operation at much lower currents than the amplifier it replaces, Q-2602. The amplifier utilizes a dual-differential insulated-gate field-effect transistor for the high-impedance input stage and a small differential operational amplifier for loop gain. It is capable of operating at input current levels as low as 10^{-12} amp by selecting appropriate feedback resistance.

Checkout of the entire system continued.

Reactor Operations. — Investigation of the reactor physics characteristics of the reactor facility at "zero" power was completed, and power level was raised to 2.5 Mw on January 26 and to 20 Mw on January 29. The "first production" element, made by the Metals and Ceramics Division, is being used for the initial power escalation. A total of about 600 Mwd of energy has been accumulated on this element to date.

The reactor was shut down February 27 for about two weeks for maintenance and to modify and recalibrate the safety system heat-power instrumentation, the performance of which was thereby significantly improved.

In attempts to remove the outer fuel element, it was found to be stuck to the fuel grid. Rather than apply a large force to the element to separate it from the grid while in the reactor vessel, it was decided to remove the fuel grid and the fuel element together and separate the two in the pool. This was done quite satisfactorily with no damage to either component. The cause of the sticking is believed to be the very close tolerance between the fuel element and the fuel grid. This condition will be corrected by modifying the fuel element end adapter.

Many shutdowns and startups have been conducted for training purposes and to evaluate the performance of the instrumentation and controls. Several minor changes have been made to control systems to provide greater flexibility and a smoother operation in the manipulation of the controls.

All of the auxiliary systems have performed smoothly, and the entire system appears to be adequately shielded for 100-Mw operation, based upon the surveys at 20 Mw.

The experimenter's collimator and shield plugs have been installed in all four of the beam holes. ANL made gamma intensity and neutron spectra measurements at the HB-2 and Hb-3 beam tubes during the escalation of power to 20 Mw.

PHYSICAL RESEARCH

Reactor Operations. — The ORR operated approximately 75.8% of the time during January and February, and the first six capsules of ^{241}Am were removed from the reactor and stored for decay in a special storage rack equipped with an off-gas line.

The new quick-opening cover on the ORR has made it possible to open the tank to begin refueling in about 5 min, compared with about 35 to 40 min previously.

The BSR operated a total of 187 hr, mostly on day shift, this period. Excavation for the new BSR 2-Mw cooling system was started in the control-room annex, where the cooling-water lines must pass under the floor. A dust-tight partition was erected to protect instruments and equipment, and normal operation was continued in spite of this work.

Waste Disposal. — The construction of the 600-gph, intermediate-level waste evaporator was completed in March. Testing of the equipment is now in progress, and full-scale operation is scheduled for April. At that time the disposal of liquid wastes in the soil, the disposal method used at ORNL for 12 years, will be discontinued.

Hot-Cell Operations. — The second ORR-AGCR capsule (P4-B2) was disassembled and examined in detail. A BeO-graphite compatibility test (Mark II) was disassembled and examined in a dry box (in-cell) maintained at a dew point of less than −40°F. Nine other fueled capsules, including two for General Electric, were disassembled and examined in detail. Two ETR-irradiated capsules containing carbides of Ti, Ta, W, Nb, and Zr were examined; one ETR-irradiated experiment containing INOR-8 tensile specimens was disassembled. One UO_2 meltdown experiment was disassembled and examined.

A capillary tube and a pressure-relief valve used as a flow restrictor from the MSRE were examined for plugging; also, a filter element was examined for plugging. Several radioisotope (^{242}Cm, ^{244}Cm) source cans were metallographically examined for corrosion and compatibility. About 350 tensile specimens and 75 impact specimens of various materials were tested; the creep-test machines continued in full operation.

PHYSICS AND MATHEMATICS

Theoretical Physics: Scattering of Electrons, Protons, and Deuterons from ^{12}C. — The relation between the inelastic scattering of high-energy electrons and impulse approximation scattering of protons from light nuclei was investigated for the excitation of collective levels described in a shell-model picture. A search code was developed to find "best fit" form factors for the electron scattering to define the nuclear transition density for proton scattering. Calculations were performed for the first 2^+ and 3^- levels of ^{12}C. The data for the two types of probes were found to be consistent in regard to enhancement and angular distribution for

both transitions. The sensitivity of the comparison was tested by expressing the proton interaction in terms of three different sets of nucleon-nucleon phase shifts. The available data did not allow a choice of a preferred phase-shift set.

The available data for the elastic scattering of deuterons by ^{12}C for energies from 3 to 34 Mev have been analyzed in terms of the optical model, including spin-orbit coupling. Two potentials are found, with a fixed real part and imaginary parts whose strength and radius depend linearly on the energy, which give a good account of all this data. There is some difficulty with the choice of diffuseness for the imaginary potential. The real potential is compared with the predictions of a simple model. The inelastic scattering to the 4.43-Mev state is also considered.

Theoretical Physics: A Smooth Two-Nucleon Velocity-Dependent Repulsion. — A velocity-dependent potential of the form

$$V(r,p) = -V(r) + p^2 e^{-sp^2}\ \omega(r) + \omega(r)\ p^2 e^{-sp^2}$$

has been used to fit the singlet even and odd (0 to 310 Mev) phase shifts for elastic nucleon-nucleon scattering. This exponential velocity dependence replaces the hard core by a repulsion which is significantly softer than the case of quadratic ($s = 0$) velocity dependence used by Green [Nucl. Phys. 33, 218 (1962)]. The applicability of ordinary many-nucleon perturbation theory for this interaction is being tested by calculation of the first- and second-order energy per particle of nuclear matter. Preliminary calculations indicate that the second-order Goldstone terms in nuclear matter are about a factor of 4 smaller than Green's potential. Harmonic oscillator matrix elements of this potential can be evaluated quite simply, and we plan to eventually use this force in Hartree-Fock calculations of spherical nuclei. Since the potential readily separates into relative x, y, and z coordinates, it should also be fairly simple to do Hartree-Fock calculations of deformed nuclei.

Low-Energy Neutron Time-of-Flight Spectroscopy. — The energies and intensities of the gamma rays emitted following thermal-neutron capture in 5-g samples of tungsten enriched in ^{182}W (94.4%), ^{183}W (86.2%), ^{184}W (94.3%), and ^{186}W (97.2%) have been measured with a 6-cm^3 lithium-drifted germanium crystal. For each isotope an energy range from 50 kev to the neutron binding energy was covered, with a resolution of 5 kev at the low energies, increasing to 10 kev at the high energies.

Gamma-ray energies and intensities are being used in conjunction with the (d,p) results of Erskine [Phys. Rev. 138, B66 (1965)] to study the level structure of ^{183}W, ^{185}W, and ^{187}W. Many new levels have been found, including the $J = 1/2$ member of the 1/2 [510] band in ^{185}W (at ≈ 15 kev) and ^{187}W (at 147 kev). The structure of these levels in terms of Nilsson single-particle states and the rotational model is being investigated.

Compilation of Charged-Particle Cross Sections. — Nuclear cross sections for charged-particle-induced reactions of the nuclei Li, Be, and B have been prepared in tabular and graphical form. This compilation

includes experimental cross-section data available in the literature before January 1, 1964, for nuclear reactions of the type A(a,b)B at all energies, where $M_a \geq$ one nucleon mass. Nuclear reactions involving mesons in the exit channel are not included. This collection supplements a previous compilation, Los Alamos Scientific Laboratory Report LA-2014. A large amount of the cross-section data is not presented either in tabular or graphical form in order to limit the size of the present compilation to a manageable one.

High-Voltage Experimental Program: Proton Strength Functions for $89 < A < 130$. — The complex nuclear potential, by virtue of its imaginary part, predicts a cross section σ_c for compound nuclear formation for incident protons. If the proton energy is well below the top of the Coulomb barrier and yet well above the threshold for neutron emission, σ_c becomes nearly equal to the (p,n) cross section. The external Coulomb field causes the (p,n) excitation functions to vary strongly with both proton energy and atomic number, but corrections can be made in order to reduce these functions to slowly varying proton strength functions. Hopefully, these strength functions can be described by a reasonable complex potential. Predictions for 5-Mev protons, based on Perey's [Phys. Rev. 3, 745 (1963)] optical model, give a minimum in the strength function near A = 90 and a maximum due to p- and f-wave resonances near A = 125.

The (p,n) cross sections have been measured with uncertainties of less than ±10% for 3- to 5.5-Mev protons on 25 nuclei with A = 89 to 130. The resulting strength functions have been compared with optical-model predictions for Perey's geometry for various real and imaginary well depths. These comparisons show that a single set of parameters will not give the observed variations with energy and with A. A real well depth of about 57 Mev gives the best overall agreement and produces the observed minimum near A = 90; however, if one adjusts the imaginary well depth to about 6 Mev in order to produce the observed strength functions near A = 90, then one finds too much energy dependence near A = 105. An imaginary well depth of about 20 Mev works better near A = 105. These results suggest that the imaginary potential should be modified in order to account for nuclear deformations near A = 105.

High-Voltage Experimental Program: Investigation of (p,n) Reaction Mechanism via Isospin Analog Resonances. — The excitation functions and the angular distributions of neutrons produced by the reactions $^{88}Sr(p,n)^{88}Y$, $^{89}Y(p,n)^{89}Zr$, and $^{89}Y(p,n)^{89}Zr^*$ ($1/2^-$, 590-kev state) in the vicinity of well-established d-wave resonances in the compound systems, which are isospin analogs of low-lying $d_{5/2}$ neutron single-particle states, were investigated in detail. Neutrons from the (p,n) reactions were detected by the time-of-flight method. The pulsed proton beam was provided by the ORNL 5.5-Mv Van de Graaff accelerator. The excitation functions show a characteristic asymmetric energy dependence [Phys. Rev. 137, B535 (1965)] about the resonance energy, and the neutron angular distributions measured on resonance as well as off resonance are symmetric about 90°. Two features make it possible to study some aspects of the reaction mechanism in detail: (1) the d-wave resonances are well isolated and are superimposed on a smoothly varying nonresonant cross section, and (2) selective enhancements of the neutron partial cross sections are

brought about by the decay of the analog states. The experimental angular dependences of the enhanced cross sections are compared with the theoretical results which are calculated from the isospin resonance theory of Robson [Phys. Rev. 137, B535 (1965)] and the generalized Hauser-Feshbach theory with an appropriate set of optical-model potentials. The results show that the statistical compound nucleus gives a good description of the reaction mechanism.

Spectroscopy Research. — It has been standard practice to compute the values of atomic energy levels by an iterative method based on the recurrence of common differences between levels of the same parity. The levels as determined by this method are subject to accumulative errors. The method of least squares has been avoided because it would appear from the formal statement of this solution that the computer memory requirements and computational time are prohibitive. A practical solution to the least-squares adjustment of atomic energy levels has been obtained. This solution is particularly compatible to computation by a digital computer, since it is conservative of total storage space and computational time. A typical array of 185 energy levels adjusted to fit 1122 transitions required a fast-memory storage of approximately 5400 words and used 70 sec of computational time.

The obvious advantage of the least-squares adjustment of energy levels is the simultaneous adjustment of all levels. This eliminates the possibility of accumulative errors in other methods. In addition, the method yields an estimate of the errors of the adjusted results, which is most useful as a criterion for selecting possible new levels to be added to the classification of the spectra. Along this same thought, the standard deviation of the fit, σ, is a characteristic number of a given set of measurements. It can serve as a criterion for selecting physically real arrays from fortuitous arrays. Irrespective of these advantages, the least-squares solution is simply a very convenient method for the computation of energy levels from a given set of classified transitions.

CHEMISTRY

Effects of Radiation on Analytical Methods. — In the study on the effect of radiolysis on the acid chloride solutions used for extractions in the TRU program, it was postulated that the mechanism involved the formation of nascent chlorine. If this were the case, it would be probable that many metals normally insoluble in HCl could be attacked by such a medium under conditions of radiation. To check this possibility, foils of several different metals were irradiated in concentrated HCl. The metals subjected to this test were Au, Nb, Pt, Ta, W, and Zr. Tantalum was found to be the most resistant to attack, showing no measurable weight loss, although colorimetric determination showed that a few micrograms were present in solution. The rate of dissolution of the other metals was measurable and in each case is far in excess of any rate shown by blank runs in HCl not irradiated. The rates of dissolution in milligrams per square meter of foil per 10^6 rads were found to be as follows:

Au, 530; Nb, 79; Zr, 430; Pt, 98; W, 760. The rates of dissolution in HCl of varying concentrations showed the same dependence of HCl concentration as does the formation of chlorine. The rates of metal dissolution are large enough to cause interferences in electrometric or spectrophotometric procedures.

Separations Chemistry Research: Extraction Equilibria. — In continued study aimed at resolving apparent anomalies in extraction equilibria with alkylamines (ORNL-3869), the apparent aggregation number $\bar{n}$ of tri-n-octylamine bisulfate (TOAHS) in dry benzene increased from 2.3 at 0.01 m to 3.3 at 0.1 m and 3.9 at 0.4 m, suggesting a mixture of dimers and tetramers. (Through this range, $\bar{n} = 1 \pm 0.1$ for both free-base TOA and normal-sulfate TOAS.) The stoichiometric activity coefficient γ_{TOAHS} dropped steeply from 1 at infinite dilution to 0.15 at 0.1 m and to 0.05 at 0.4 m TOAHS. Water concentrations in undiluted $TOA \cdot 0.765 H_2SO_4$ deviated noticeably from Henry's law, the activity/mole fraction quotient a_{H_2O}/X_{H_2O} increasing steadily from about 0.8 at a_{H_2O} = 0.1 to nearly 1.3 at $a_{H_2O} = 1$. The corresponding activity of the amine sulfate mixture, taken as the solvent, dropped smoothly and with slight curvature from 1 at $a_{H_2O} = 0$ to 0.14 at $a_{H_2O} = 1$.

Separations Chemistry Research: Biochemical Separations. — The preparation of highly purified samples of specific transfer ribonucleic acids is receiving continued attention under the Biochemical Separations Program. Previously, commercial E. coli B "soluble" RNA has been the source, and the chromatographic preparation of $tRNA^{Phe}$ and $tRNA_4^{Leu}$ has been described (ORNL-3917). Under the Biological Macromolecular Separations Technology activities, in the Chemical Technology Division Unit Operations Section, the procedure for preparing mixed tRNA's (ORNL-3845) has been greatly scaled up, and large quantities of mixed tRNA's from E. coli W have been made available. The fractionation of this material on the reversed-phase chromatographic columns is being investigated, and a map is being constructed of the positions in the chromatogram of all the tRNA's at three pH levels. Differences between tRNA's from E. coli B and from E. coli W, as detected by chromatographic behavior, appear in general to be minor. However, there appear to be two phenylalanine tRNA's in E. coli W, which are only partially resolved on the reversed-phase column. Attention will be directed toward methods of resolving them.

It has been found that the reversed-phase chromatography columns can be operated at much higher flow rates, five to ten times those previously used, without loss of chromatographic resolution, thus drastically reducing the time required to perform an experiment.

Volatility Studies: Sorption of PuF_6 by NaF. — One aim in volatility processing is to find a sorbent from which PuF_6 can be desorbed easily, as in the UF_6-NaF system. While static and some dynamic tests indicate that sorption of PuF_6 on the fluorides of elements in groups I-A and II-A proceeds quite satisfactorily, desorption has been unfeasible so far.

The results presented here indicate that the difficulty in desorbing PuF_6 lies in the tendency of the plutonium to form stable complexes having plutonium oxidation numbers less than 6. Next, we will test other sorbents and conditions to promote better desorption.

The most complete evaluation of the desorption problem was with NaF as the sorbent. This evaluation consisted in (1) determining the efficiency of sorption of PuF_6 passed through beds of NaF at temperatures from 20 to 700°C; (2) determining the degree of desorption from the NaF in a series of 2-hr isothermal tests at 20 to 700°C, and in one 10-hr test at 600°C; (3) determining the oxidation number of the plutonium as a function of temperature of the NaF bed.

In the sorption tests, where about 50 mg of plutonium (as PuF_6) was passed in a stream of fluorine (60 ml/min) through a 2-g bed of NaF in 15 min, sorption was more than 99% complete at temperatures of 100 to 700°C, but about 50% complete at 20°C.

Desorption was insignificant from a 2-g bed of NaF in a stream of fluorine (also 60 ml/min) at all temperatures tested in 2-hr tests; in a 10-hr test at 600°C, only 0.31% of the plutonium had desorbed, leaving a green residue.

Study of the oxidation number of the plutonium was done by oxidimetric (with ferrous sulfate) and iodometric titrations, infrared absorption, and x-ray diffraction.

The green solid produced by the 600°C reaction was identified as Na_3PuF_7, since the diffraction pattern was almost identical with that for Na_3UF_7. This reaction product released no iodine; however, for the 350°C product and those produced at lower temperatures, iodine was released, with the maximum release for the 100°C reaction product equivalent to an oxidation number for the plutonium of about 4.75. The infrared study of the 100°C reaction product also showed slightly greater concentrations of tetravalent plutonium than of hexavalent. The 600°C product is green, as already mentioned; while the 300°C product is green-brown, and the 100°C product is reddish brown.

Chemical Engineering Research: Stacked-Clone Contactor. — The stacked-clone solvent extraction contactor, by virtue of its rapid control response, low solution holdup, and high throughput, is potentially useful for processing small inventories of solutions which may be highly radioactive. The contactor has previously been operated exclusively with the aqueous phase continuous. Recent tests have proved that organic-continuous operation is not only possible but may even be desirable, since operation at low flow ratios (A/O) is now possible. Using the system 0.08 M HNO_3—Amsco at 40°C, the 11-stage contactor was operated with (1) all 11 stages aqueous-continuous, interface in end section (normal operation); (2) interface controlled by a "floodometer" at the fourth stage, stages 4 to 11 aqueous-continuous; (3) interface controlled by a "floodometer" at the seventh stage so that stages 1 to 7 were organic-continuous; (4) all stages organic-continuous, with the interface in a disengagement section at the raffinate end. Equipment modifications have been made to allow the organic feed to enter the raffinate end section and be picked up by a stronger induced underflow from the end clone.

Flooding data under these conditions show throughputs at flow ratios (A/O) of 1/2 and 2 to be 6.3 and 4.9 liters/min respectively. Aqueous-continuous operation yielded flow capacities of 3.7 and 5.7 liters/min respectively.

Mechanisms of Separations Processes. — The electrolysis of water to produce hydrogen is being studied as a means of utilizing large blocks of cheap electrical power available from proposed dual-purpose desalination reactors. Investigations are continuing on an electrolysis cell incorporating porous nickel electrodes (85% void, 0.030 in. thick) with a 10-mil asbestos membrane serving as the electrolyte reservoir.

So far, the conclusions are that this new type of electrolysis cell shows very good current efficiencies at high current densities and that off-peak power could be used without decreasing cell efficiencies. Significantly, this small cell has been operated intermittently for about three months without requiring a new membrane or electrodes.

A 60-cps sine-wave current interrupter was constructed to measure electrode polarization voltage and the IR drop across the asbestos membrane. These measurements permit evaluation of electrode potentials without the complicating effects of electrolyte and membrane resistance. The cell is subjected to a half-wave rectified current pulse from a 60-cps ac source. During the current-off period a capacitor is charged to the polarization voltage of the cell, and this voltage is read out in a high-impedance circuit connected to a recorder. During the current-on period, the IR drop between the electrodes is measured across a positively biased transistor with the charge on the capacitor now bucking out the polarization voltage of the cell.

Measurements of cell voltage were made with both dc and half-wave rectified ac power to the cell, and cell temperatures up to 90°C. At higher current densities, slightly lower cell voltages were obtained with ac power; at a current density of 1243 ma/cm^2 and 90°C, the cell voltage was 2.08 for dc and 2.05 for ac power. Current efficiencies, measured by determining the volume of hydrogen evolved over a 5-hr period, were 95.7% for dc and 94.4% for ac at this current density. Current measurements were made across a standardized shunt, and half-wave current measurements on the recorder were calibrated by the use of an integrating digital voltmeter. It was found that recorder readout of the voltage drop across the shunt was 2% high, probably due to the electronics of the recorder being affected differently by the strange wave form. There was a linear relation between recorder readout of shunt voltage and the voltage as measured by the integrating digital voltmeter; therefore, corrections are easily made.

Polarization voltage and IR drops, measured at 90°C and at a current density of 1243 ma/cm^2, were 1.71 and 0.34 v respectively. The reversible cell voltage at this temperature is 1.175 v. Daily startup and shutdown of the laboratory cell has not visibly affected cell efficiency.

Thermochemistry and Calorimetry. — The heat capacity of K_2ReI_6 was measured from 7 to 300°K. Anomalous behavior is exhibited over the temperature range 190 to 240°K which resembles that shown by K_2ReBr_6 over the more narrow region of 200 to 220°K. Anomalously high heat capacities

are observed in this region only when the K_2ReI_6 has been cooled to ~170°K and spontaneous heat evolution at this temperature is allowed to go to completion. In contrast to the similar behavior shown by K_2ReBr_6, however, subsequent coolings to 170°K do not diminish this heat effect. A lambda-type heat-capacity anomaly with a maximum at 24.0°K (Néel temperature) was observed which represents the transition from the paramagnetic state to the ordered antiferromagnetic state below this temperature.

Organic Chemistry: Organic Compounds in Nuclear Technology. — 2-Ethyl hexanehydroxamic acid was prepared and was tested as a model of the interfering complexing agent that we expect to be formed from hydrocarbon diluents and nitric acid in solvent extraction processes. 2-Ethyl hexanehydroxamic acid has interesting properties as an extractant for some elements. Distribution coefficients, E_a^o, vary inversely with the concentration of nitric acid in the aqueous phase. The specificity of the reagent is strikingly demonstrated by the equilibria found with 2 M nitric acid. The distribution coefficients for zirconium and hafnium are 20 and above, while those for cobalt, thorium, and uranyl are 0.05 and less.

High-Temperature Aqueous Solution Chemistry. — The electrical conductances of aqueous sodium chloride solutions were measured at temperatures from 0 to 800°C, at pressures from 1 to 4000 atm, and at concentrations to 0.1 m. From these results it was shown that sodium chloride, although a very strong electrolyte at 25°, associates to some extent at temperatures above 500°C at densities from 0.4 to 0.7 g/cm^3. Association constants for the formation of the neutral sodium chloride species, $NaCl^0$, were calculated to the extreme conditions of temperature and pressure. For comparison, measurements on 0.01 demal (~0.01 m) potassium chloride solutions were made to 800°C and 4000 atm; these measurements showed small differences in conductance, chiefly due to the difference in mobility between the potassium and sodium ions. Interpretation of the several extensive investigations on sodium chloride, potassium bisulfate, potassium sulfate, and sulfuric acid, carried out in this program, has revealed simplicities in aqueous media at high temperatures that are not found at low temperatures. For example, the limiting equivalent conductances of NaCl and $KHSO_4$ are essentially independent of temperature between 400 and 700°C (if kept at a constant density in the range between 0.5 and 0.8 g/cm^3), although the limiting equivalent conductances of strong electrolytes increase by factors of 7 to 10 between 25 and 300°C at constant density.

Physical Chemistry of Molten Salts: Iodide Chemistry in Molten Fluorides. — The removal of the 6.7-hr fission product ^{135}I from a molten-salt fuel would reduce the amount of daughter ^{135}Xe, a major fission product poison formed in the fuel. In measurements which are to be continued, it has been found that iodide (the chemical form of iodine expected to be present) can be readily removed from $LiF\text{-}BeF_2$ melts upon sparging with mixtures of $HF\text{-}H_2$ by the reaction

$$HF(g) + I^-(d) \rightleftharpoons F^-(d) + HI(g), \quad Q = (P_{HF}/P_{HI})[I^-] \text{ kg/mole} .$$

The experiments were performed at 470 to 650°C in a nickel vessel provided with a gold-lined gas exit tube to prevent reaction of HI with metal surfaces at the lower temperatures. Although the material balances are not yet satisfactory, the results indicate that the above reaction is the principal one which occurs. The decrease of iodide in the melt is related to the equilibrium quotient, Q, and the number of moles of HF passed per kg of melt, n_{HF}/W, by

$$\ln ([I^-]/[I^-]^0) = -Q(n_{HF}/W) .$$

For $2LiF\text{-}BeF_2$ the present incomplete results give $\log Q = -1.094 + 2.079 \times (10^3/T)$ with error limits of ±15% in Q. From this and the available thermochemical data for HI(g) we have calculated the following expression for E^0:

$$1/2\ I_2(g) + e \rightleftharpoons I^- , \quad E^0 = -0.345 + 0.002\ (T/10^3) ,$$

vs the reference half-cell reaction

$$HF(g) + e \rightleftharpoons F^- + 1/2\ H_2(g) , \ E^0 = 0 .$$

With increasing BeF_2 content of the coolant, Q appears to go through a maximum near 41.5 mole % BeF_2, indicating a maximum in the activity coefficient ratio $\gamma_{I^-}/\gamma_{F^-}$.

Physical Chemistry of Molten Salts: Method for Predicting Thermal Conductivity of Molten Fluorides. — A new equation for estimating the thermal conductivity of fluoride melts has been derived. The equation is

$$k = (13/V^{2/3})\ \mu ,$$

where k, thermal conductivity, V, molar volume, and μ, velocity of sound, are all in cgs units. The molar volume of the melts can be estimated from the simple rule of additive molar volumes; the velocity of sound can be estimated from thermodynamic parameters. Measurement of thermal conductivity of molten fluorides ordinarily has an uncertainty of 25%; the estimated values are believed to be accurate to within this uncertainty. The equation given above was obtained by empirically adjusting a relationship originally derived by Bridgman [Proc. Am. Acad. Arts Sci. 59, 162 (1923)].

Isotope Chemistry. — A potentiometric method was developed for the microtitration of dibenzoylmethane and other weak acids in pyridine or ethylenediamine. Various polarized electrodes were investigated; anodized platinum and cathodized antimony microelectrodes responded best. Sharp end points were obtained for most acids with a titrant consisting of tetrabutylammonium methylate dissolved in a 90% benzene—10% methanol solution. Several compounds, whose analyses were not previously feasible, such as cyanoacetamide, acetylurea, 2,3-butanedione, sym-diphenylurea, malonamide, dimethylglyoxime, and 2-nitropropane, were titrated successfully.

TRANSURANIUM-ELEMENT PROCESSING

Process Development. — In continued efforts to find nonfluoride dissolution methods for refractory PuO_2, it was found that plutonia is dissolved rapidly and completely by anhydrous HCl gas in molten KCl-$AlCl_3$ at 500°C. In a similar experiment without HCl, a plutonia dissolution rate of only 8% per hour was obtained.

If fluoride catalyst is used for dissolving TRU targets that contain PuO_2, it will be necessary to reduce the fluoride concentration to less than 5 ppm prior to subsequent processing in Zircaloy-2 equipment. Fluoride removal by hydroxide precipitation, filtration, and redissolution was investigated with simulated dissolver solution that contained thorium as a stand-in for plutonium. In experiments with and without aluminum, fluoride was reduced to 3 ppm by two precipitation cycles. Fluoride decontamination factors of about 54 and 14 were obtained for the first and for the second cycle.

In a study of the purification of plutonium in HCl during TRU processing, the stability of tetravalent plutonium and its anion exchange behavior in concentrated HCl are being investigated. Tetravalent plutonium was found to be completely stable in both 6 and 8 M HCl at room temperature (by spectrophotometric analysis), and there was no evidence of disproportionation after heating to 75°C for 1 hr. For the anion exchange investigation, more than 99% of the plutonium was loaded on Dowex 1-8X resin at 60°C from either 6 or 8 M HCl feed solution. After loading, the column was washed with 10 column volumes of 6 or 8 M HCl without incurring significant plutonium losses (about 0.1%). Plutonium can be stripped from the column either with dilute acid or with concentrated acid containing sufficient reductant to reduce the plutonium to the trivalent state. Since significant decontamination from ruthenium and zirconium can be expected by the latter method, NH_4I and ascorbic acid were evaluated as reductants for plutonium. Both 8.0 M HCl—0.1 M NH_4I and 6 M HCl—0.1 M ascorbic acid were effective in stripping plutonium; however, NH_4I is preferable, since recoveries of 99% or more were obtained in both experiments.

A program to prepare and classify the solid compounds of berkelium has been started. The dioxide and sulfate have been prepared on a microgram scale, and these compounds are being characterized by x-ray analysis. The dioxide has a fluorite cubic structure, and the lattice parameter was found to be 5.33 A. Characterization of the sulfate structure has not yet been completed. Additional studies will include preparation and characterization of berkelium chlorides and fluorides and the hydrogen-reduced oxide.

Curium Recovery Facility. — A new tantalum mixer-settler rack was installed in the facility, and a third ^{242}Cm processing campaign was started. Five Martin targets were dissolved, yielding about 6-1/2 g of ^{242}Cm. The curium was purified in the solvent extraction system by the Dapex process and then the Tramex. Approximately 6.2 g of ^{242}Cm was recovered in the final Tramex product solution. The overall gross-gamma decontamination factor was about 500.

Experimental Engineering Studies. — The new tantalum mixer-settlers for the Curium Recovery Facility were tested before installation in the hot cell. They have deeper chambers and tandem impellers to improve the balance between mixing and pumping. The hydraulic performance with the Tramex flowsheet was good at design flow rates except for an intermittent flow of raffinate and/or strip solution in the waste organic. A slight modification of piping was made to eliminate this. The average steady-state entrainment of strip in the waste organic was 1%. The individual stage efficiency, measured by use of nickel chloride, averaged 75% for extraction at 1300 rpm and 94% at 1440. The overall efficiency, however, was less than 60%, which must have resulted from operating too close to the flooding point (the organic flow was 40% greater than design to avoid "pinching" in the loaded organic). The average stripping efficiency was 75% at 1000 rpm and 78% at 1220. The loss was less than 0.5% after five stages.

Hydraulic tests of the 3/4-in.-diam glass pulsed columns were made by using the modified Pharex flowsheet, in which the solvent is 0.5 M 2-ethylhexylphenylphosphoric acid in Amsco. Severe flooding, caused by phase inversion, and plate wetting occurred. Cleaning of the plates with HNO_3-HF, alcohol-HCl, or a detergent was only temporarily beneficial. Batch testing of the system showed very slow phase separation for an organic-continuous dispersion. Treatment of the solvent with activated alumina greatly improved the rate of phase separation and permitted operation of the columns at 16 ml/min (each phase) for both extraction and strip at 25°C. This is twice the design flow rate.

HFIR Target Fabrication Development. — Equipment in cubicle 1 is to be used in April to inspect 18 HFIR target elements which have been irradiated at Savannah River for one year. We will inspect for straightness, visible damage, and corrosion damage and to ensure that the six 25-mil holes in the hex sheath are clean. The integrity of the mechanical attachment of the can to the rod will be determined. These inspections will be required before the elements can be installed in the HFIR. This new task has rearranged and accelerated the schedule for cubicle 1 to meet the April deadline. Four items of special equipment must be designed and built to perform some of the inspection required on these 18 rods. Fabrication and testing of these devices are scheduled to be completed by April 1.

TRU Operations. — Testing and checkout of the Transuranium Processing Plant chemical-process and target-fabrication equipment are still in progress. Tests with the dissolver indicated that simple heat balances could be used to measure dissolution rates within ±50% in the range of 1 g of aluminum per minute, and thus indirectly provide a means of ensuring a low hydrogen concentration (less than 4%) in the vapor space. Operation of the pellet-fabrication equipment in cubicle 3 was satisfactory during the fabrication of 40 pellets. The office and maintenance addition to the building has been completed and is now occupied by Transuranium Project personnel.

METALLURGY AND MATERIALS

Physical Properties Studies. — Richter and Kohlhaas [Z. Naturforsch. 19a, 1632 (1964)] claim that the thermal conductivity of iron deviates significantly from a smooth function of temperature near the Curie point (766°C): from −6.7% at 763°C to +8.4% at 768°C. We measured the thermal conductivity of our high-purity iron and obtained 11 data points between 754 and 775°C. We confirmed our previous measurements and did not confirm the changes reported by Richter and Kohlhaas, which we believe to be a manifestation of their equipment and method of operation.

We completed measurements in the range −200 to 20°C on a platinum specimen obtained from the National Physical Laboratory. This is the specimen on which they obtained thermal conductivity data from 25 to 1000°C, which indicated virtually no temperature dependence of the thermal conductivity. Recent measurements by others on platinum indicate a positive temperature dependence and at 1000°C deviate 15% in value from the NPL values. Our low-temperature results disagree with the NPL values by 5%, confirm to within 1.5% the positive temperature dependence, and indicate that a minimum in the thermal conductivity occurs at about −50°C.

Direct Observation of Lattice Defects. — To improve our understanding of deformation of metals with the body-centered cubic crystal structure, which include the important refractory metals, we are studying dislocations in tungsten. Dislocations are line defects; their mobility accounts for the ductility of metals.

We tried three etching methods to bring out dislocation pits on tungsten samples: (1) use of Wolff solution (2 parts saturated $CuSO_4$ and 1 part concentrated NH_4OH), (2) use of 15% $K_3Fe(CN)_6$–5% NaOH, and (3) electrolytic etching in 2% NaOH. Methods 2 and 3 gave similar results; the pits had sharper corners and were more densely distributed than the ones formed by method 1. Judging from the densities, method 1 seems to give a more realistic picture of dislocation distribution in tungsten. However, to check further, we are using transmission electron microscopy to correlate the etch pits with the dislocations in thin specimens. The evidence so far shows a poor correlation between the pits and dislocations in specimens prepared by methods 2 and 3. We have not yet examined specimens etched in Wolff solution.

We completed compression tests at two temperatures on one batch of polycrystalline tungsten specimens. The deformation modes at room temperature are twinning and slip with transgranular cracks; the only mode at 250°C is slip. The details of deformation will be examined by etch pitting. Also, we are preparing single-crystal specimens for similar study.

Physical Ceramic Studies. — To obtain a better understanding of the mechanical behavior of such ceramic materials as ThO_2 and UO_2, we are continuing to study the mechanisms by which materials with the fluorite structure deform under load. The sliding of grain boundaries observed in deformed polycrystalline fluorite-structure ceramics was reproduced in a UO_2 bicrystal. Pores were opened along the sliding boundary, which is consistent with the intergranular void formation and sliding that

occur when polycrystalline material is deformed. A UO_2 bicrystal that had been displaced slightly along the boundary was examined by electron transmission microscopy. The boundary contained numerous kinks, and dislocations were denser near the boundary than within the grain. Electron microscopy of an as-grown boundary from the same sample revealed that the boundary was initially smooth and did not contain kinks.

CONTROLLED THERMONUCLEAR RESEARCH

The DCX-1 Injection-Accumulation Experiment. — During the first two-thirds of this report period the DCX-1 facility was utilized by the Beam-Plasma Interaction Group to study experimental scaling relations of the Burnout series. The findings are in the subsection of this report entitled "Properties of Plasmas Created by Beam-Plasma Interaction."

Experiments using the end-wall stabilization technique (ORNL-3917, p. 32) have recently been extended well into the lossy region in order to see whether stabilization would be effective in reducing losses. With the end walls at ineffective positions, the instability drove proton losses that held the central fast-proton density to 1.7×10^8 cm^{-3}, a factor of 12 below stable density. The proton energy spread under these conditions was 60 kev (full width at half maximum). These values agree with earlier measurements made with the usual grounded end walls. When the end walls were positioned (axially and azimuthally) for optimum stabilizing effect, the following observations were made: (1) The amplitude of the ion cyclotron fundamental was reduced by factors of 3 to 10, but was not completely quenched. The effect on higher harmonics was difficult to assess but was much less pronounced. (2) The energy spread scaled approximately like the fundamental amplitude. The lowest value was 45 kev, still much larger than the 11-kev spread inherent in the analyzer system. (3) The proton density remained loss dominated at the original value (±10% fluctuations) in spite of the above-mentioned reductions in other instability effects.

It was possible, moreover, to find positions of the end walls which enhanced losses (reduced density) by a factor of 2 with an accompanying factor of 3 increase in average fundamental rf amplitude and a 90-kev energy spread. These studies are continuing with modified end-wall structures.

Properties of Plasmas Created by Electron-Cyclotron Heating. — The INTEREM facility modifications are nearly completed, and the machine is in operation. About 8 ma equivalent of 20-kev H^0 beam current has been injected into the machine. The cooled silicon barrier detector on the south side of the machine has been used to measure the energy of the emerging charge-exchange neutrals. This detector, with the associated field-effect transistor input amplifier, has a resolution of 2.5 kev for the incident particles. To date, no energy spread has been detected in the particles trapped by the electron-cyclotron plasma or trapped by gas. The detector has shown that there are three beam components injected in

the machine. Most of the beam consists of 20-kev H^0 atoms, but there are small fractions of 13-kev H^0 and 40-kev H^0. These are produced from H_3^+ and H^+ by conditions in the source region and drift tube.

Another cooled silicon barrier detector on the top of the machine has been used to look at the long-decay-time component of the neutral current after beam turnoff. The observed effect is now smaller than when last reported, and this may be due to the lower microwave power now available. To date, we have been able to get only 29 kw continuous wave of microwave power into the cavity, whereas the long tails previously reported were obtained with 34 kw continuous wave or more. The facility has been operated under power at a central pressure of 2×10^{-6} torr. However, at this low pressure less microwave power can be fed into the cavity due to the appearance of reflected power bursts. The cleaning of the microwave cavity has produced the desired effects. The previously observed condition in which the discharge would establish itself on diffusion pump oil "varnish" from the walls does not occur now.

Properties of Plasmas Created by Beam-Plasma Interaction. — An attempt was made to use the DCX-1 apparatus to generate the fully ionized plasma of the burnout experiments. DCX-1 has a number of desirable features not present in either Burnout IV or V. It has a pumping system of large gas-handling capacity, plasma is easily accessible for diagnostics, and it is a large machine. The experiments were beneficial, but an unforeseen, unfavorable combination of characteristics of the apparatus prevented us from obtaining as high a power density in the plasma, and therefore we had a lower plasma temperature, than obtainable in either Burnout IV or V.

The unfavorable combination involved the location of the pumps, the fact that the pumps are made of a magnetic material, and the geometry of the vacuum wall of the machine. In particular, the small diameter of the outboard-coil spool cans relative to other DCX-1 cross-sectional dimensions proved to be a serious limitation on the size and placement of the reflecting electrodes. A further complicating feature was that the magnetic-field axis does not seem to be coincident with the geometrical axis of the machine.

We believe that this combination of characteristics prevented us from obtaining the pressure gradient so necessary for high power densities in the plasma. Langmuir probes gave an ion density of 10^{12} cm^{-3} and an electron temperature of 20 ev. Radio-frequency emission from the plasma was essentially "white" up to 4 kMc, the limit of the observation.

In the hot-electron-blanket experiment, the intermediate-energy electron group has been studied in more detail. Since this group of electrons is present when the hot x-ray-producing group is absent, the intermediate energy must be less than or equal to about 20 kev. Assuming that the drift time of the electrons around the annulus is less than or about equal to the relaxation time for scattering out the mirrors, a lower energy limit of about 2 kev is found. Studies of the electron distribution impinging on a probe located at midplane yield a mean energy of about 1.8 kev. If we accept the value of 2 kev, the density of the intermediate electron group is calculated to be between 1 and 5×10^{11} cm^{-3}.

Further experimentation with iron shielding in the anode region has permitted operation of the hot-electron blanket at a midplane magnetic field in excess of 3 kilogauss — over a factor of 2 increase. A new anode design is being tested in order to attempt operation at higher magnetic fields.

Ion Source and Ion Beam Technology. — With the energy for the DCX-3 injector increased from ≈ 7 kev to ≈ 15 to 20 kev, the injector has been reevaluated. After studying several possibilities, the system that looks most promising consists of accel-decel electrodes located very close to a magnetic solenoid (lens), a plasma-shaping coil around the anode cup, and a no-PIG (horizontal) epoxy insulator arrangement. This system is now being used for injecting 80- to 100-kev protons (hundreds of milliamperes) axially into DCX-2. During a recent maintenance period on the DCX-2 generators, the injector was moved into the 100-kev laboratory facility, and preliminary evaluations were made for low-energy beams. An all-copper duoplasmatron ion source was used for 10- to 25-kev H^+ ions, and a four-electrode version of the duoplasmatron was used for 30- to 40-kev H_2^+ ions. A preliminary criterion for evaluation was the beam available through a 2.5-cm aperture located 120 cm from the lens. The performance of the system is illustrated by the following typical results:

Total Beam (ma)	Ion Species	Energy (kev)	Beam Through 1-in. Aperture (ma)
100	H^+	20	35
100	H^+	15	25
100	H_2^+	40	45
100	H_2^+	30	40
145	H_2^+	40	60

These results do not necessarily indicate a limitation in total beam nor the ultimate performance. A new system similar to the end injector is being built for a more detailed study in this energy range.

Theoretical Plasma Physics. — An infinite uniform monoenergetic proton plasma of energy $E = mc^2\gamma > mc^2$ in a uniform magnetic field B is subject to electrostatic instability arising from relativistic dilatation of the gyrofrequency $\Omega = e|B|/mc\gamma$. Even highly nonrelativistic cases (20 and 300 kev) of fusion interest appear susceptible. Unstable frequencies are very near Ω and its multiples, unstable wavelengths λ are near gyroradius ρ_g or submultiples, and growth rates are of the order of a few percent of Ω. The propagation direction is exactly perpendicular to B, so stabilization by electron motion along B (electron Landau damping) is inoperative, as is stabilization by introduction of the effect of finite length in the B direction. At certain values of λ/ρ_g, the

threshold proton density is infinite, while between these values, stabilization results when the density exceeds a small critical value ($\omega_{pe}/\Omega \approx$ a few percent). The instability persists when the electrostatic approximation is relaxed by calculating fluctuation fields from a wave equation instead of from Poisson's equation. Quenching of the instability by giving the protons a square distribution f(E) of energy is very slight, even for $\delta E/E \cong 50\%$. Since the dilatation of the gyrofrequency is energy dependent, an ion damping akin to the electron Landau damping occurs when df/dE is nonzero over a range of E. Numerical calculations of this effect are in progress. (A heuristic estimate suggests that a Gaussian distribution with $\delta E/E \approx 5\%$ should suffice to stabilize a fusion plasma, so that reactors as currently projected would not be subject to this instability.)

Atomic Cross Sections. — Studies have continued of the population of excited states of hydrogen molecules formed by the electron capture of H_2^+ and H_3^+ in hydrogen gas. For both H_2 and H_3 the fraction in highly excited electronic states was less than that for H^0 formed by protons capturing an electron. We had previously postulated that the highly excited states of the molecules were comparable to the "Rydberg" states of the hydrogen atom; however, these measurements indicate that the states cannot be described in such a simple manner.

A time-of-flight spectrometer has been fabricated to confirm the Franck-Condon principle for the dissociation of H_2 by electron impact. The spectrometer determines the velocity spectrum of H^0 arising from the repulsive levels of H_2. The resolution is sufficient to provide separation of masses up to 200 amu. Modifications of the electron gun are being made to increase the signal-to-noise ratio.

In an attempt to provide an absolute detector of low-energy H^0 (1 to 10 kev), a secondary-emission plate was used to intercept an H^0 beam. Secondary electrons arising from particle impact were accelerated to 10 kev and detected by a silicon surface-barrier detector. It was anticipated that with the resolution obtainable with silicon detectors in this energy range we could determine the shift in the peak pulse height of the secondary electrons with a change in H^0 energy. Instead, the spectrum at a given H^0 energy was composed of seven peaks corresponding to groups of one, two, three, etc., electrons arising from each incident H^0. Although this technique offers little as an absolute particle detector, it may prove useful in the detailed investigation of secondary electron emission.

BIOLOGY AND MEDICINE

RADIATION GENETICS

Human Cytogenetics. — Biological testing of the anticipated GT-11 S-4 experiment has been completed. Qualification and predelivery acceptance testing of the hardware have been completed, and the flight hardware and required documentation have been delivered to NASA. A

complete replication of the GT-3 S-4 experiment, in which the "flight" device was subjected to a Gemini-Titan vibration and acceleration profile simulating the GT-3 flight, has been successfully completed. The resulting cytological material is being studied.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — The major achievement in the program on treatment of radiation injury in mice was the demonstration that 90-day mortality after foreign bone marrow transplantation could be reduced to less than 6% by proper selection of age, number, and day of injection of marrow cells as determined by a series of factorial design studies. This value is well within the range of mortality obtained with genetically identical bone marrow. From the same factorial design studies one can also see how to get the greatest 90-day mortality. This latter information is particularly important in knowing what conditions to avoid in transplanting foreign bone marrow.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation: Semiconductor Detectors. — Recently, the production of germanium gamma-ray detectors from commercially prepared germanium crystals was made difficult owing to unpredictable variations in the lithium drift rates. Some as-received ingots of germanium showed diffusion constants only about 0.001 of the usual values. Since the drift times for large-volume detectors became too long to be practical, the lithium precipitation process in germanium was studied to determine why the germanium was nondriftable. The lithium precipitation kinetics in 13 germanium ingots, including both zone-leveled and pulled crystals from several commercial sources, were examined. It was concluded that the low lithium drift rates often encountered in commercial germanium crystals are the result of dissolved oxygen. Oxygen atom concentrations of the order of 10^{14} cm^{-3} or higher can seriously reduce the diffusion constant.

Basic Instrumentation: Charge-Sensitive Preamplifier. — Since an energy resolution of 0.1% can be obtained with semiconductor detectors, more sophisticated preamplifiers and amplifiers are required for experimental studies. A transistorized charge-sensitive preamplifier consisting of a wide-band (gain-bandwidth product $\approx$ 2 gigahertz) low-noise charge-sensitive input section and an output cable-driving section was developed for use with semiconductor radiation detectors. At room temperature the noise line width of the preamplifier is about 0.8 kev full width at half maximum for germanium detectors and 0.06 kev/pf (RC-RC filter), and the charge gain drift with temperature is less than 50 ppm/°C. The output section will linearly drive ±2 v into a 93-ohm load with an output pulse rise time of 25 nsec with negligible overshoot.

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