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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MAY 1966

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

May 1966

DATE ISSUED

June 6, 1966

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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CONTENTS

REACTOR DEVELOPMENT..... 1
GAS-COOLED REACTOR PROGRAM..... 1
Fast Gas-Cooled Reactor Irradiation Tests..... 1
GCR-ORR Loop 1 Irradiations..... 1
Mathematical Simulation of Coated-Particle Behavior..... 2
Fission Product Deposition..... 2
REACTOR FUELS AND MATERIALS..... 2
Zirconium Metallurgy..... 2
Nondestructive Test Development..... 3
REACTOR COMPONENTS..... 3
Reactor Controls Development..... 3
POWER REACTOR FUEL PROCESSING..... 4
Aqueous Process Development: General..... 4
Aqueous Process Development: Grind-Leach Process..... 4
Aqueous Process Development: Burn-Leach Process..... 5
Aqueous Process Development: Advanced Fuels..... 6
Fluoride Volatility Processing: Decladding of Zircaloy-Clad UO_2 Fuel..... 6
Fluoride Volatility Processing: Small-Scale Engineering Studies of Fluidized-Bed Process..... 7
Fluoride Volatility Processing: Corrosion Problems Related to the FBVPP..... 7
Fluidized-Bed Volatility Pilot Plant (FBVPP)..... 8
FUEL-CYCLE STUDIES..... 9
Urania Sol Preparation Studies..... 9
Firing Studies on Sol-Gel UO_2..... 9
^{233}U Storage and Distribution..... 9
Gas Evolution from Sol-Gel Oxide Fuels..... 10
Thorium-Uranium Recycle Facility..... 10
Preparation of ThC_2 Microspheres..... 11
Coated-Particle Development Laboratory..... 11
MOLTEN-SALT REACTOR PROGRAM..... 11
Molten-Salt Reactor Experiment..... 11
Metallurgy..... 12
Chemical Development..... 13
Radiation Effects..... 13
Analysis of Radioactive MSRE Fuels: Analyses of MSRE Samples..... 13
Analysis of Radioactive MSRE Fuels: Determination of Oxide in MSRE Fuel..... 14
HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT..... 14
Metallurgy..... 14
Component Development and Testing..... 15
Operations..... 15
Instrumentation and Controls..... 16

PHYSICAL RESEARCH..... 17
Reactor Operations..... 17
Waste Disposal..... 17

Hot-Cell Operations 17
PHYSICS AND MATHEMATICS 18
Theoretical Physics: Elastic and Inelastic Scattering of Heavy Ions 18
Theoretical Physics: Optical Model for 30-Mev Proton Scattering 18
Theoretical Physics: Comparison of the Effective Two-Nucleon Interaction in 2 $s_{1/2}$, 1 $d_{3/2}$ Shell Nuclei with a Surface Delta Interaction 18
Low-Energy Neutron Time-of-Flight Spectroscopy 19
Low-Temperature, Nuclear and Solid-State Physics 19
High-Voltage Experimental Program: Charge-State Distribution of Channeled 40-Mev ^{127}I Ions in Gold Single Crystals 19
High-Voltage Experimental Program: The $^{14}C(^{3}He,p)^{16}N$, $^{14}C(^{3}He,d)^{15}N$, $^{14}C(^{3}He,^{3}He)^{14}C$, and $^{14}C(^{3}He,\alpha)^{13}C$ Reactions at 2.0, 4.0, and 5.0 Mev 20
CHEMISTRY 20
Effects of Radiation on Analytical Methods 20
Separations Chemistry Research: Biochemical Separations 21
Separations Chemistry Research: Solvent Extraction 22
Foam Separation Studies 22
Volatility Studies 22
Chemistry of Carbides and Nitrides 23
Chemical Engineering Research: Stacked-Clone Contactor 23
Chemical Engineering Research: Macromolecular Biological Separations 24
Chemical Physics: Microwave and Radio-Frequency Spectroscopy 24
High-Temperature Aqueous Solution Chemistry: Solubility of Magnetite 25
High-Temperature Aqueous Solution Chemistry: Isopiestic Studies 25
Physical Chemistry of Molten Salts 26
Isotope Chemistry 26
TRANSURANIUM-ELEMENT PROCESSING 27
Curium Recovery Facility 27
Experimental Engineering Studies 27
TRU Operations 27
METALLURGY AND MATERIALS 28
Physical Properties Studies 28
Physical Ceramic Studies 28
Structure of Metals 28
Solid Reaction Studies 28
CONTROLLED THERMONUCLEAR RESEARCH 29
The DCX-1 Injection-Accumulation Experiment 29
Properties of Plasmas Created by Electron-Cyclotron Heating 29

[redacted]

Properties of Plasmas Created by Beam-Plasma Interaction..... 29
Ion Source and Ion Beam Technology..... 30
Theoretical Plasma Physics..... 30
Atomic Cross Sections..... 30

BIOLOGY AND MEDICINE..... 31
SOMATIC EFFECTS OF RADIATION..... 31
Medium(Low)-Level Long-Term Effects of Radiation..... 31
Gnotobiotics..... 32
Physiology of Blood Platelets..... 32
RADIATION GENETICS..... 33
Human Cytogenetics..... 33
COMBATING DETRIMENTAL EFFECTS OF RADIATION..... 33
Mammalian Recovery..... 33
RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION..... 33
Basic Instrumentation..... 33

OPEN LITERATURE PUBLICATIONS..... 34

VISITORS..... 37

[redacted]

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MAY 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 the General Atomic fast gas-cooled reactor design (GCFR). Test specimens are supplied by General Atomic. The third capsule (P4B-3) in this series, which included the first "flexcan" fuel element and a fuel-supported clad element, is undergoing postirradiation examination. Visual examination of the irradiated specimens showed that the general appearance was not appreciably changed from the preirradiation condition. Sections of the flexcan specimen are being examined metallographically; no evidence of reaction between the cladding and the sodium has been found, nor has grain growth of the UO_2 been observed in either element.

The fourth capsule in this series, designated P4B-4, contains two fuel specimens similar to those in capsule P4B-3. Irradiation began April 16, 1966, and is continuing according to planned conditions, except that the cladding temperature of the fuel-supported specimen is about 55°C below the design value. The specimens have been subjected to 21 thermal cycles and 5 pressure cycles with no apparent ill effects. We are considering extending the irradiation period to two ORR cycles.

GCR-ORR Loop 1 Irradiations. — Experimental fuel assembly 15, which was removed from the loop on April 10 after an irradiation period of about nine months, is being subjected to postirradiation examination. The test fuel element consisted of a mixed bed of 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 was vented to the loop coolant. The outer surface of the upper graphite container was roughened in appearance, presumably as a result of corrosion by the coolant. However, the coatings on the fuel particles in this container retained their original smooth surfaces and were apparently in good condition. A preliminary inspection revealed no broken fuel particles and no evidence of significant segregation of the different kinds of particles.

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Analysis was completed on the second sample of loop coolant. The May-pack technique for determining the character of the nongaseous activity circulating in the loop was used. The results substantiated those from the previous measurement, which indicated that the principal sources of activity were activated elements from stainless steel in the form of particulates that were probably corrosion products.

Mathematical Simulation of Coated-Particle Behavior. — We completed the metallographic examination of coated particles irradiated in the first ETR X-basket experiment. From the correlation of these results with the behavior predicted by the mathematical model, we conclude that there are at least three independent criteria for successful performance of pyrolytic-carbon-coated fuel particles, as follows:

1. Void volume equivalent to about 2% of the fuel volume per atom percent burnup must be provided by the inner coating and/or fuel kernel to accommodate fuel swelling, fission-gas release, and fast-neutron-induced shrinkage of the outer coating.
2. The outer layer, which serves as a pressure vessel, must not be subject to fission recoil damage. The inner coating must not be severely contaminated by fuel and must retain its shape and be thick enough to absorb all damage from fission fragments.
3. The outer coating must be mechanically decoupled from the inner layer so that recoil-induced shrinkage of the inner layer does not impose an excessive stress on the outer coating.

As predicted by our mathematical model, the best types of coated particles in this experiment were the two batches of UO_2 coated with porous inner layers and high-density outer layers. The remaining eight batches were designed to give a direct comparison of isotropic outer coatings having densities of approximately 1.8 g/cm^3 with high-density granular outer coatings (~2.05 g/cm^3). In every case the granular outer coatings performed better; that is, they showed lower rates of failure.

Fission Product Deposition. — We are developing techniques for macroscopically examining fission product deposition on a container surface by radioautographic means. In preliminary tests, clear deposition patterns were observed on commercial x-ray films when the film was placed in contact with surfaces upon which iodine had been previously deposited. Calibration curves were developed for interpreting the density of the exposed film in terms of the quantity of fission products deposited per unit surface area. Attempts will be made in future tests to relate unusual deposition patterns to defects on the container surface.

REACTOR FUELS AND MATERIALS

Zirconium Metallurgy. — We are studying the changes that occur in the preferred orientation of polycrystalline α-zirconium during pure shear as part of a study of the development of texture during fabrication. In the high-shear regions, the basal poles of the highly deformed grains are always oriented at 40 ± 5° to the normal to the shear plane and 55 ± 5° to the shear direction, regardless of the original angles that the

principal texture of the specimen made with the shear plane and shear direction. In the deformed regions near the shear plane, the basal poles are located on an essentially direct path from their original position to the final position. The position occupied along that path depends directly on the amount of deformation undergone.

We developed a method for determining the orientation in space of the basal pole of any grain of polycrystalline zirconium. Three simple measurements are made on a single surface of the specimen with a polarizing metallurgical microscope fitted with certain standard accessories. The method is being used to determine the microtexture of fabricated polycrystalline specimens in increments of 10- to 20-μ-thick layers. We fitted a polar chart recorder to the microscope to allow direct recording of the basal pole position of each grain, giving a direct plot of the pole figure in polar coordinates.

Nondestructive Test Development. — Our study of x- and gamma-ray attenuation for the evaluation of fuel materials and fuel elements is continuing. Variation in transmitted intensity is monitored with a scintillation detector and may be related through proper calibration to the property of interest.

We are developing attenuation techniques for determining the average thickness of pyrolytic carbon coatings on small batches of ThO_2 particles. Initial investigations on 230-μ-diam particles, both bare and with a 95-μ coating, showed that a through-transmission x-ray system would be feasible. To relate radiation attenuation to coating thickness, we examined batches of 230-μ-diam particles coated with carbon to some 14 different thicknesses ranging from 13 to 172 μ. Rectangular Plexiglas containment cells held the particles in the x-ray beam. An NaI(Tl) crystal coupled to a photomultiplier detected the transmitted radiation. A digital voltmeter with a five-print-per-second digital printer for direct instantaneous readout measured the photomultiplier output. The signal can be processed through the averaging circuitry of our scanner for a printout of the average voltage developed over a particular scan length.

This technique will be quite useful as an on-line process monitor for coating thicknesses for both the Thorium Utilization and the Gas-Cooled Reactor Program.

REACTOR COMPONENTS

Reactor Controls Development. — Statistical analysis of the fluctuating current from a neutron-sensitive ionization chamber offers a means of rapidly (5 to 10 min) determining subcritical reactivity. We are currently investigating the problems encountered with on-line negative-reactivity-measuring devices for determining the subcritical reactivity of a power reactor. Two phases of this program were studied: an analog computer study of a simulated situation, and an analysis of signal-to-noise ratios under high gamma background conditions.

An analog computer study of a simulated instrument system is of real value, because it eliminates the need of costly reactor experiments,

while allowing variation of such parameters as type of neutron detector, placement of the detector, type of reactor, and the value of negative reactivity. In this application, the neutron-induced current fluctuations in an ionization chamber placed near a nuclear reactor were simulated on an analog computer. Preliminary results show that for a realistic situation (simulation of negative reactivity measurements at the HFIR), a negative reactivity of about −1 dollar can be measured with an accuracy of approximately 3% in a measurement time of only 6 min.

An analysis was made to derive equations required to quantitatively evaluate the degradation effect of the gamma-flux background on the measurement of negative reactivity. In power reactors, subcritical reactivity measurements by neutron noise analysis must be made in the presence of intense gamma radiation from residual fission products. In this case, an additional background noise is superimposed on the ionization chamber neutron noise. It was first found that, in absence of gamma radiation, the ratio of the signal to the detector neutron noise is almost proportional to the square of the reactor multiplication factor. In the presence of gamma rays, the degradation of the signal-to-noise ratio is proportional to the ratio of the gamma flux to the neutron flux; and as long as the gamma flux in roentgen-per-hour units is less than the neutron flux in nv units, the gamma degradation is not appreciable for optimized ionization chambers. Since the neutron flux is itself proportional to the reactor multiplication factor, the ratio of the signal to the detector noise background is therefore proportional to the third power of the reactor multiplication factor (inverse of subcriticality). Consequently, if the gamma background is intolerably high for a given subcriticality, the situation will be improved drastically for a slight increase in reactivity.

As a result of this analysis, an investigation was made to obtain a detector with optimized characteristics for subcritical reactivity measurements in high gamma backgrounds. A commercially available RSN-76A boron-lined ionization chamber filled with ^{4}He at 2 atm performs better than any other ionization chamber of comparable sensitive volume.

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) hot-cell studies on the burn-leach process, also for graphite-base HTGR fuel; and (3) chemical decladding studies on sintered-aluminum-product-clad (SAP-clad) UC fuel from the HWOCR (Heavy-Water-Moderated Organic-Cooled Reactor).

Aqueous Process Development: Grind-Leach Process. — Fine grinding followed by acid leaching is being evaluated as a method for processing

graphite-base HTGR fuels that contain coated fuel particles. Both engineering- and laboratory-scale tests are being made with Peach Bottom carbon-coated ThC_2-UC_2 fuel particles in a graphite matrix. So far, the best method found for grinding HTGR fuels is rough crushing to −4 mesh in a hammer mill followed by roll-crushing to reduce the particle size to −140 mesh. Grinding this fine ensures rupture of all of the particle coatings. Performance tests with a new 12-in.-diam by 4-in.-wide double roll crusher, designed and built at ORNL, generally met or exceeded expectations. The −4 mesh fuel from the hammer mill required three passes between the rolls, at successively closer roll spacings (40, 3, and 2 mils) for best results. The grinding rate for the final 2-mil roll spacing was 5 to 10 kg/hr; the rate was limited to a value which would cause less than 1 mil measured roll deflection. Allowable grinding rates for the first two passes were 25 to 50 kg/hr.

Acid leaching of the new roll crusher product for 6 hr in refluxing 15.8 M HNO_3 resulted in recovery of 99.7% of the uranium and 99.4% of the thorium. Leaching of prototype Peach Bottom fuel roll-crushed to −140 mesh is also being studied on a laboratory scale. Satisfactory uranium and thorium recoveries can be achieved by using boiling 13 to 16 M HNO_3 as the leachant. The presence of HF as a dissolution catalyst is not required for fuel containing coated carbide particles. In experiments where the fuel was leached for 5 hr and the residue washed three times with water, the uranium recoveries increased from about 99.6 to 99.8% as the HNO_3 concentration was increased from 2 to 16 M; the corresponding increase in thorium recovery was from about 98 to 99.2%. Uranium and thorium recoveries of about 99.9% were achieved by leaching the crushed fuel twice for 5 hr with boiling 13 M HNO_3. Multiple leaching was more effective than a single 20-hr leach. After leaching for 20 hr with boiling 13 M HNO_3, uranium and thorium recoveries were 99.6 and 99.1% respectively. In all cases the product solutions were about 0.05 M in Th + U. The amounts of soluble organic compounds in the product solutions (derived from the carbon in the ThC_2-UC_2 particles) corresponded to 20 to 50% of the carbide carbon. These organic compounds would probably have to be removed or destroyed before the product solution could be used as a feed for a solvent extraction process.

Aqueous Process Development: Burn-Leach Process. — The first of a series of hot-cell evaluations of the combustion-leach process for graphite-base fuels was run after several weeks of cold tests with unfueled graphite and unirradiated fuel. In this process, HTGR compacts (pyrolytic-carbon-coated UC_2-ThC_2 particles dispersed in a graphite matrix) are crushed to −4 mesh and then burned in a fluidized bed of 60- to 120-mesh alumina using air and/or oxygen at 750°C. After burning, the ThO_2, U_3O_8, and nonvolatile fission product oxides are leached from the alumina bed with fluoride-catalyzed nitric acid to produce a feed solution from which thorium and uranium can be recovered by conventional solvent extraction.

A major concern with this process has been the possibility of producing burner off-gas contaminated with gross quantities of volatile fission product oxides, particularly ruthenium and cesium, which would require extensive off-gas cleanup. To minimize fission product volatility,

a technique was developed whereby the fuel is burned at a uniform rate at 750 ± 25°C with air/oxygen mixtures regulated to provide about 90% oxygen utilization. Fission product oxide particulates, alumina, and carbon particles entrained in the off-gas stream are trapped on a micrometallic filter inside the burner, which rapidly becomes coated with carbon fines.

In the first run, 100 g of crushed HTGR fuel, irradiated to 8000 Mwd/metric ton and decayed about one year, was burned in 2 hr at 750°C. Operation was quite smooth as indicated by gas-chromatographic and infrared analyses of the off-gas for O_2, CO_2, CO, and N_2. Preliminary data from this run indicate that fission product contamination of the off-gas after filtration was very slight. The gross-gamma decontamination factor across the micrometallic filter was estimated to be 2×10^6, which is considerably higher than anticipated.

Aqueous Process Development: Advanced Fuels. — Methods for processing fuels for HWOCR's are being developed on a laboratory scale. The HWOCR fuel now receiving the most attention is UC clad in SAP (a sintered dispersion of Al_2O_3 in aluminum). A process that appears feasible for this type of fuel consists in removing the SAP cladding by dissolving it in NaOH solution and then dissolving the hydrous UO_2 residue in nitric acid in preparation for decontamination and recovery of the uranium by solvent extraction. This method has the advantage that, during the decladding step, the UC is hydrolyzed, releasing the carbide carbon primarily as methane. Thus, only traces of organic compounds are found in the solvent extraction feed solution.

In experiments with simulated fuel (UC/SAP weight ratio about 10), about 90% of the aluminum matrix of the SAP was dissolved in 4 hr in boiling 2 M NaOH when the initial NaOH/Al mole ratio was 3 to 4. Less than 0.006% of the uranium was lost to the decladding solutions. After decladding, the residues were dissolved in boiling 5 to 15 M HNO_3 to yield solutions that were 0.5 M in uranium. Any aluminum remaining from the decladding step was dissolved by the nitric acid along with the uranium oxide. Less than 2% of the original carbide carbon was found in the final solutions.

Fluoride Volatility Processing: Decladding of Zircaloy-Clad UO_2 Fuel. — In the fluidized-bed volatility process, the proposed method for removing Zircaloy cladding is to contact it with HCl to form volatile $ZrCl_4$. The $ZrCl_4$ vapor is then transferred through the off-gas system to a pyrohydrolysis unit for conversion to ZrO_2. This scheme requires that the off-gas system be heated to above 300°C and that there be a separate reactor for pyrohydrolysis. An alternative scheme that retains the zirconium in the fluidized bed as ZrF_4 was tested but was not satisfactory.

The alternative recently tested included contacting the Zircaloy with HCl in a two-zone fluidized bed, with the HCl entering the lower section of the bed. The $ZrCl_4$ formed in the lower half of the bed was then contacted with HF in the upper section to form ZrF_4. Tests of this scheme in a 1-in.-OD fluidized-bed reactor revealed two major difficulties. Decladding rates under similar conditions were about five times as fast for an alumina bed as for a ZrF_4 bed. This indicates that ZrF_4

interferes with the reaction between zirconium and HCl. Also, during decladding at a relatively high rate, serious sintering was observed even when the temperature was below 500°C. Sintering apparently will occur when the concentration of $ZrCl_4$ in the gas phase is greater than 0.4 vol %. Since ZrF_4 does not sinter at such low temperatures, the most likely explanation is that a low-melting eutectic occurs in the system ZrF_4-$ZrCl_4$ and that this liquid phase leads to the observed sintering.

In future tests, removing Zircaloy cladding with HF-O_2 in a bed of ZrF_4 will be compared with results obtained for an alumina bed.

Fluoride Volatility Processing: Small-Scale Engineering Studies of Fluidized-Bed Process. — To establish a reference point for evaluating the effectiveness of fluidized beds, one complete process experiment was made without a bed. A segment of a tubular fuel element (Consolidated Edison type) containing four 12-in.-long, 0.3-in.-ID stainless steel tubes filled with UO_2 pellets was contacted with 40-60 vol % HF-O_2 at 550°C for 3 hr and was then fluorinated for 3 hr at 450 to 500°C. The sum of the uranium accounted for after the run was 99% of that charged; of this only 0.2% was lost to reactor residues. No severe temperature excursions occurred during either decladding or fluorination. The evolution of UF_6 during the fluorination period was monitored by the ultraviolet spectrophotometer. The indications were consistent with those derived from temperature elevations in the NaF traps resulting from the NaF-UF_6 complexing reaction.

Fluoride Volatility Processing: Corrosion Problems Related to the FBVPP. — Data on corrosion expected in the FBVPP (Fluidized-Bed Volatility Pilot Plant) was compiled at the Columbus Laboratories of Battelle Memorial Institute (under subcontract). The summary report (BMI-X-362) was received late in November 1965. Data from studies at ANL, BNL, ORGDP, ORNL, and the open literature confirmed earlier choices of nickel for the primary reactor and the porous nickel filters, Inconel for the pyrohydrolyzer, and Duranickel alloy 301 for certain high-temperature flanges.

Metallographic studies (made in part at the originating sites, but mostly at BMI) revealed intergranular modifications in specimens of nickel that had been exposed to process conditions. Subsequent studies at BMI showed that similar modifications were produced in nickel 200 and 201 by exposing coupons to several types of alumina that might be used as the fluidized media. The nickel specimens were sealed in a Vycor capsule in a vacuum and exposed at 520°C. Results for nickel 200 and 201 were almost the same. With Alcoa T-61 alumina, intergranular modifications ranged from a depth of about 0.5 mil in 25 hr to 1.6 mils after 200 hr. Coupons exposed to Norton RR Alundum were modified to a depth of about 0.5 mil in 100 hr; Norton 38 Alundum caused modifications ranging from 4.0 mils after 25 hr to 9.0 mils after 200 hr; and specimens exposed to Norton regular Alundum were modified to depths of 7.0 and 7.5 mils during exposures of 25, 50, 100, and 200 hr. With no alumina present, coupons exposed in vacuum and in an atmosphere of water showed no modifications; those exposed in an atmosphere of nitrogen showed traces of modifications. When the various types of alumina were analyzed by combustion with oxygen and titration of the evolved SO_2, the following concentrations of sulfur

were measured (ppm): Norton regular, 190; Norton 38, < 30 (probably about 10); Norton RR, < 50 (probably about 30); and Alcoa T-61, < 50 (probably about 20). Sulfur is thought to be responsible for the intergranular modifications in view of the above results and the sensitivity of nickel to sulfur. Also, sulfur was identified in the affected areas of nickel coupons and in portions of nickel from process equipment used at BNL by both a chemical spot test [Materials Protection 1(9), 100 (1962)] and microprobe analyses. Other tests are in progress to measure changes in tensile properties in specimens with intergranular modifications.

We plan to install equipment in the FBVPP for leaching waste solids from the primary reactor. The leachant will probably be 13 M HNO_3 with as much as 0.1 M HF present. Screening tests were started at BMI to find an alloy from which leaching equipment can be fabricated. Eighteen alloys or forms of alloys were tested. The exposures were conducted in the above solution under refluxing conditions in an all-Teflon container equipped with Teflon support rods. The alloys with most favorable corrosion rates based on either 24- or 48-hr exposures were Corronel 230 and Hanford alloys 17, 20, and 21. Corronel 230 is nominally 36% Cr, 58% Ni, 1% Mn, and 5% Fe. The Hanford alloys all contain 25% Cr, 50% Ni, and 0.6% Mn. In addition, alloy 17 contains 21.5% Fe and 1.5% Mo; alloy 20 contains 16.0% Fe, 6.0% Mo, and 1.0% Cu; and alloy 21 contains 18.5% Fe, 1.5% Mo, and 3.0% Cu. Corrosion rates of welded specimens submerged in liquid were: after a 48-hr exposure, about 27 mils/month for Corronel 230; after 24-hr exposures, 24 mils/month for Hanford alloy 17, 17 and 27 mils/month for Hanford 20, and 24 mils/month for Hanford 21. Pure chromium corroded at 4 mils/month; therefore nickel alloys high in chromium will be tested next.

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.

Equipment for the FBVPP will be designed so that BrF_3 or BrF_5, as well as fluorine, may be used to convert uranium compounds to volatile UF_6. When using the interhalogens, nonvolatile PuF_4 will remain in the alumina bed and subsequently will be removed by fluorine. An attempt will be made to design a still suitable for either the interhalogen process or the present method, using fluorine to remove both uranium and plutonium. Thus far, all features of the fluorine flowsheet are being retained.

Supplementing the last report of progress in project engineering (ORNL-3830, p. 6), design of all phase 1 process vessels is complete, except for the new equipment required in the interhalogen process. Design of the cooler for the off-gas from the primary reactor and design of the transition zone between the cooler and the primary filter will be modified slightly to minimize the possibility of fines retention. Piping drawings are being prepared.

The surge tank and scrub tower for disposing of HF, HCl, and fluorine are finished. The pyrohydrolyzer, cold traps, reactor backup filter, and pyrohydrolyzer backup filter are all about 75% complete. Work began on the primary reactor, and it is about one-fourth finished.

In Building 3019, the decontamination of cell 3 was completed, and the cell was painted. It has now been released from masking requirements. Cleanup in cells 1 and 2 continued. The cost-plus-fixed-fee contractor began work in cell 3 on April 24, 1966. The first task is drilling the 18-in. holes through the 5 ft of concrete between cells 2 and 3.

FUEL-CYCLE STUDIES

Urania Sol Preparation Studies. — The preparation of aqueous sols of urania by a solvent extraction technique is being studied. Here, aqueous solutions of $U(NO_3)_4$ and formic acid are contacted with an organic solution of a long-chain amine that extracts the nitrate and formate ions. As these anions are extracted, hydrolysis of the U^{4+} ion occurs, leading to formation of a urania sol. In laboratory studies, anion extraction is done in two separate batch extractions, with a 0.5-hr digestion period at about 50°C between the extractions. Sol prepared this way is about 0.2 M. A sol of this type was concentrated, by evaporation of the water, to about 1.3 M, where it set to a solid. Usually we are able to concentrate urania sols to higher concentrations than this. We think that the NO_3^-/U mole ratio may have been too low because of accidental extraction of too much of the nitrate ion, and that this may have led to solidification on evaporation.

Firing Studies on Sol-Gel UO_2. — Firing studies are being made on sol-gel-derived UO_2-ThO_2, UO_2, and UO_2-PuO_2 to establish conditions that yield products of low carbon content (less than 100 ppm), near-theoretical density, and good resistance to crushing forces. The major problem in firing UO_2 is removal of the carbon introduced during the microsphere-forming step as sorbed 2-ethyl-1-hexanol and surfactant. Previous work showed that the difficulty of removing carbon from microspheres increases as the particle diameter increases. Low-temperature drying methods, such as steam stripping to 200°C or washing with methanol or acetone, were unsuccessful. We have removed carbon from 200-μ-diam UO_2 microspheres to acceptable levels (less than 100 ppm) by firing in CO_2. This approach was unsuccessful with 400-μ-diam spheres; however, when 20 wt % of either ThO_2 or PuO_2 is present in UO_2, carbon contents of 10 to 40 ppm are readily attained. The ThO_2 or PuO_2 appears to prevent pore closure and densification at temperatures where the carbon reacts with CO_2.

^{233}U Storage and Distribution. — Oak Ridge National Laboratory serves as a national storage and distribution point for ^{233}U. A processing facility is installed in the pilot plant (Building 3019) to permit cleanup and packaging of ^{233}U in various forms as requested by users.

During the past month a number of cans of $^{233}UO_2$ that had been stored for as much as 14 months were monitored to compare the radiation readings with those made previously.

Radiation readings of the cans (2-1/2 in. in diameter and 8 in. long), each containing 450 g of ^{233}U and 42 ppm of ^{232}U, had increased from 300 mr/hr to 10,000 mr/hr over a 14-month period. Readings of identical cans

containing 200 ppm of ^{232}U had increased from 340 mr/hr to 65,000 mr/hr over the same period. The original readings were taken four to eight weeks after solvent extraction.

Two processing runs, 200 g of ^{233}U each, were made with the glove-box solvent extraction facility. The results are being evaluated to determine the "extractability" of ^{220}Rn and subsequent scrubbing of ^{212}Pb.

One shipment, 8 g of ^{233}U, was made during the month.

Gas Evolution from Sol-Gel Oxide Fuels. — In a continuing study to improve procedures for degassing sol-gel microspheres, new techniques have been explored. Results showed that heating the wet (as-received) microspheres in steam causes a pronounced reduction in the amount of gas evolved during subsequent heating. Temperatures in the range 500 to 600°C proved most effective and avoided sintering effects. In no case, however, could the degassed microspheres be considered to be completely degassed, since grinding of microspheres that had been "cleaned up" gave a powder that evolved copious amounts of gas.

In tests of ThO_2, a flowing 1/1 mixture of oxygen and water was passed over the microspheres at temperatures ranging up to 1000°C. The gas was changed to pure oxygen at 1000°C and held there for 9 hr. Subsequent heating in vacuum yielded 0.06 cm^3 of gas per gram of ThO_2. The gas consisted of the following: 59% CO_2, 19.4% CO, 17.2% H_2, and 4.8% N_2. The spheres were then ground to −270 mesh and reheated to 1000°C in steam. Gas (1.06 cm^3/g) composed of 96.0% H_2, 1.3% CO, 2.2% N_2, and 0.55% CO_2 was released.

In tests of UO_2, a flowing 1/1 mixture of CO_2 and water was passed over the microspheres. It was observed that both the CO_2-sorptive and water-sorptive capacities of the UO_2 decreased with increasing temperature up to 550°C. At 550°C, 0.18 cm^3 of H_2O and 0.05 cm^3 of CO_2 per gram of UO_2 were sorbed reversibly.

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

Building construction is approximately 61% complete. Finishing of interior building walls with an application of a protective coating is progressing satisfactorily. Cell construction has progressed, with the walls of cell E up to ceiling level and the remaining cell bank walls up to approximately 4 ft from the operating-area floor. All equipment furnished under ORNL participation is progressing satisfactorily except the radiation shield doors. Shield door 2 has been tested and accepted, and is in transit to Oak Ridge. Shield doors 1 and 3 are slightly behind schedule.

As to the design of sol-gel equipment, the uranyl nitrate storage-system drawings have now been completed. Design of the small-scale precipitation-peptization process development unit is nearly 85% complete, and construction is expected to be completed in an additional month. A set of bench-scale equipment to assist the chemical development groups in the development of the amine extraction process for producing urania and urania-thoria sols is being designed.

Preparation of ThC_2 Microspheres. — Two approaches are being studied to make ThC_2 microspheres from ThO_2 microspheres prepared by the sol-gel process. The first involves heating 1150°C-fired ThO_2 microspheres in a rotating bed of lampblack at temperatures in the range 2150 to 2250°C in an argon atmosphere. This approach shows promise of producing theoretically dense ThC_2. The second includes incorporating carbon in the ThO_2 sol, forming gel microspheres of the ThO_2-C sol, and converting to ThC_2 by firing to about 2000°C. This approach yields a porous product.

When the rotating bed of lampblack is used, it appears that carbon introduced in the sphere-forming operations, as sorbed alcohol or surfactant for example, decreases the rate of conversion to dicarbide. Complete conversion was obtained only with spheres from which the extraneous carbon had been removed by firing in air prior to conversion. Also, this extraneous carbon appears to cause porosity in the dicarbide product. The gas evolution studies, discussed elsewhere, are intended to find ways of removing the extraneous carbon.

Coated-Particle Development Laboratory. — In the Coated-Particle Development Laboratory (CPDL), methods are being developed for remotely making and coating microspheres. During the past report period, 50 kg of ThO_2 spheres was prepared for pyrolytic-carbon coating studies. In this and other preparations the porous metal and glass frits used to collect the spheres tended to blind after repeated use. This problem has been solved by using a 30- by 250-mesh stainless steel "Twilled Dutch Weave" screen.

In the coating studies, 15 fluidized-bed experiments were conducted last month to help establish conditions for applying coats of a specified nature. The major problem was that of achieving a high-density isotropic coating. High flow rates were used to reduce pyrocarbon buildup on the nozzle; this led to lowered temperatures in the coating zone and consequently to decreased coating density. Decreasing flow rates should increase coating density but also increase faceting and maintenance problems. Four entrained-bed coating runs in which a gas-cooled nozzle was used showed that water cooling of the nozzle is not necessary to the operation of the entrained-bed coater.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — Operation at 5 Mw was interrupted when, for some reason that cannot be determined, the control rods dropped, setting off a chain of events that led to draining of the fuel and coolant and freezing of the salt in part of the radiator tubes. The frozen tubes were thawed without damage, the salt loops were refilled, and operation was resumed after three days. A faulty control relay interfered with action to prevent the coolant freezing, but the incident showed that the heat losses to the radiator doors were also higher than anticipated. As a consequence we made some revisions in interlocks to improve the freeze protection.

When we resumed 5-Mw operation, we encountered difficulty in withdrawing fuel samples, because of dragging of the sample latch at a bend

in the sample tube. We installed a revised latch. While we were testing it, an electrical failure stopped the drive with the latch down near the pump bowl. It was necessary to drain the fuel and pull up the cable with the manipulator before the isolation valves could be closed. The drive mechanism was then removed to a maintenance area in the building, where a shorted connector was replaced and the cable was straightened. Since reinstallation, the sampler has worked perfectly. The reactor was down ten days for this job.

After a total of 164 hr at 5 Mw, we raised the power to 7.5 Mw, at which we operated for 103 hr. Operation at the higher power level revealed that the coolant radiator capability would limit the heat extraction to less than the design 10 Mw. We increased the pitch of the main-blower blades to the limit of the drive motor capability, then raised the power to the maximum attainable. This was 8.8 Mw, as indicated by the nuclear instruments. The deficiency is due to low air-side heat transfer, substantially poorer than predicted. The overall heat transfer coefficient in the fuel-to-coolant heat exchanger was also below predictions. The reactor was operated continuously at 8.5 to 8.8 Mw for two days to complete the program of bringing the reactor to full power.

Operation at 5 and 7.5 Mw confirmed that the ^{135}Xe poisoning was lower than expected, only 0.4% $\delta k/k$ at 7.5 Mw, compared with 1.0% predicted and 2.0% if no xenon were removed. Radiochemical analysis of the salt indicated no significant loss of ^{135}I, ruling out this explanation for low ^{135}Xe. The most likely reason appears to be a small amount of bubbles circulating with the salt, which enhances the stripping of xenon.

When the reactor operated at high power, radiolytic decomposition of cooling water in the thermal shield produced gases which accumulated, probably in the slides. Water flow through the slides was increased by repiping the supply to a higher pressure point in the system. Other measures are being designed to cope with the gas evolution and accumulation.

The pressure drop through the particle trap in the fuel off-gas line began to rise noticeably after four weeks of operation. By the seventh week, the pressure drop was as high as 10 psig. A replacement trap, like the first, is being fabricated for use while the first is examined to determine future measures for the off-gas system.

Metallurgy. — A major problem in the MSBR is the necessity of joining graphite to the structural material, Hastelloy N. The more promising techniques use molybdenum as a transition material. A new brazing alloy (35 Ni–60 Pd–5 Cr, wt %) has been developed for use in this system. Joints of graphite to molybdenum have been brazed and survived a 700°C corrosion test ($LiF-BeF_2-ZrF_4-ThF_4-UF_4$) of 1000 hr with only a slight surface roughening (< 1/2 mil). Good wetting of both materials had been achieved.

Six new grades of graphite are being evaluated for physical properties that are related to the MSBR. Four are isotropic and two are anisotropic needle-coke graphite. One isotropic grade and one needle-coke grade were in the form of pipe.

Specimens from the six grades were subjected to the MSRE standard salt impregnation, screening test of evacuation, and subsequent exposure

for 100 hr to molten fluoride salt at 700°C and under a pressure of 150 psig. The average percentages of bulk volume of graphite penetrated by salt were 0.3, 0.5, 4.0, 7.1, 13.5, and 14.0. The two lowest values were obtained on specimens from the two anisotropic needle-coke graphites. The 7.1 value was obtained on specimens from the pipe of isotropic graphite. Since the value for the MSRE core graphite was 0.2 in such tests, it appears that none of the six new grades of graphite is of a quality suitable for the MSBR.

Pore entrance diameter spectra, gas permeabilities, and strength measurements are being made on the six grades of graphite to determine what potential they may have for additional development to make them suitable for MSBR requirements.

Chemical Development. — Fission product analyses of fuel salt sampled from the MSRE during 5- and 6-Mw operation indicated that 85 to 95% of the ^{135}I remained in the salt phase at equilibrium. The observed low ^{135}Xe poison fractions cannot therefore be ascribed to volatilization of xenon precursors. Values of fission power calculated from the equilibrium concentrations of 9.7-hr ^{91}Sr and 2.6-hr ^{92}Sr were about 20% lower than the nominal total power values of 5 and 6 Mw. Radiochemical analyses of metal specimens exposed to the MSRE pump-bowl gas phase showed the presence of several tellurium isotopes and ^{99}Mo in the gas phase. Only those iodine isotopes with long-lived tellurium precursors were observed on these specimens. In particular, 6.7-hr ^{135}I, formed from the decay of 0.4-min ^{135}Te, was not detected on the gas-phase metal specimens. Although ^{99}Mo would be expected to plate out on the INOR-8 walls of the reactor container vessel, it was in fact found in approximately normal yield in the salt phase. Further indications of nonzero valence of some of the molybdenum were the plating of ^{99}Mo on both the gas-exposed and liquid-exposed portions of the stainless steel cable on the salt sampling container.

Radiation Effects. — Modifications to beam hole HN-1 in the ORR, required for the molten-salt irradiation experiment, were begun during the April 10 reactor shutdown. A new aluminum beam hole liner, the equipment chamber, and the retraction mechanism were installed in beam hole HN-1 during the scheduled shutdown period. Subsequently, instrument and control panels were installed, and wiring between the control panels and equipment chamber is under way.

Fabrication of the thermal loop irradiation assembly has been completed, and it has been installed in a mockup facility in Building 9204-1 at Y-12 for out-of-pile operation. On completion of satisfactory out-of-pile test operation, the assembly will be transported to the ORR for installation and operation in the irradiation facility.

Analysis of Radioactive MSRE Fuels: Analyses of MSRE Samples. — Fuel and coolant samples removed from the MSRE during the recent experiments were received and analyzed routinely at the High-Radiation-Level Analytical Laboratory with relatively few problems other than the contamination of the transport containers. The inner walls of the MSRE transport containers have become highly contaminated during the recent power level experiments. Readings from point sources have exceeded

250 r/hr. Gross contamination has been reduced by using thin wall liners to contain the sample ladles during shipment. Nevertheless, minute particles of salt have succeeded in contaminating the inner walls. Upon receipt of a sample, the empty transport container is transferred to the decontamination cell. Versene, oxalate, 1-1 nitric acid, and formula 50 are used to reduce the level of contamination to < 0.5 r/hr. The transport container is then removed from the decontamination cell. At this point the contamination is further reduced until the container measures < 20 mr/hr, < 500 dis/min beta, and < 30 dis/min alpha. After the maximum power level is attained, elaborate decontamination methods may become necessary in order to decontaminate the containers to the above levels.

Analysis of Radioactive MSRE Fuels: Determination of Oxide in MSRE Fuel. – Since the installation of the apparatus for the determination of oxide in MSRE fuel in the HRLAL, two samples have been analyzed. These samples contained 50 and 55 ppm oxide respectively. Using the in-cell radiation monitor, the first sample read 30 r at 1 ft and the second sample read 50 r at 1 ft. Sufficient equipment, which is disposed of after each analysis, is being made for 24 samples, approximately a three months' supply. The ORNL Master Analytical Manual method for the determination of oxide is being written.

Throughout the period of development of the method for the determination of oxide in fused fluoride salts, difficulty was often encountered with the water electrolysis cell. The gas flow through the cell was often erratic as a result of successive momentary pluggings of the helical capillary in the cell. Since the possibility exists that the electrolysis cell in the remote oxide apparatus may eventually have to be replaced, it was deemed necessary to find a method of regenerating the cell which would permit a steady gas flow at relatively low flow rates. The results to date indicate that it is not possible to acquire a coating of partially hydrated P_2O_5 compatible with low flow rates when an aqueous solution of phosphoric acid is used as the starting material. At present, acetone solutions of phosphoric acid are being investigated. We hope that the acetone will wet the plastic capillary in the electrolysis cell and provide a more even coating of P_2O_5 on the capillary walls.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. – The fuel element producer is now in routine production at a rate of two elements per month. Minor problems, particularly in maintaining concentricity during handling, continually occur. All elements still require minor waivers, but none has contained a defect that might jeopardize full-power operation. Planning for the next fuel element contract has now begun.

The feasibility of attaching two full-length wire spacers to an HFIR fuel plate by ultrasonic welding has been demonstrated. The spacers do not appear to interfere with forming, although data on the forming reproducibility of plates with spacers have not yet been obtained. By machining after attachment, it has been possible to control the spacer

height within 1 mil. It will now be necessary to show that plates containing spacers with such a tolerance may be assembled into an element.

Component Development and Testing. — The installation of a poison injection system has been completed, and the system has been put into service. This system consists of a storage tank containing 95 gal of $Cd(NO_3)_2$, a 2-in. (IPS) gravity feed line to the pressurizer pump suction manifold, a 1/2-in. (IPS) thermal convection loop from pressurizer pump to storage tank, and a pneumatically actuated valve system that can be remotely operated from the control room. Functional tests of the system indicate that the poison can be injected into the primary piping system at 60 gpm within 5 sec after the valves are placed in the "inject" position.

A redesigned outer shroud flange has been fabricated. This shroud flange incorporated a strengthened skirt for distributing the water entering the control region and ball socket attachments for target tower snubbers.

Tests on the outer-element fuel plate buckling apparatus were continued. This facility utilizes the differential axial thermal expansion between the fuel plate and a carbon steel base to simulate the differential encountered in the fuel element between plate and sidewalls. Early tests indicated a plate deflection, in the convex direction, of about 0.015-in. maximum. The deflection was uniform along the central 16 in. of plate length, and tapered off to essentially zero deflection at each end.

Radial deformation was found to have a pronounced effect on deflection. The test apparatus was altered to simulate the relative rotation of the fuel element side plates, which relieves the radial expansion. This reduced the amount of deflection in the convex direction, but the ends moved in opposition to the central portion. The total runout and overall profile remained the same as before.

Operations. — The reactor operated quite well during this period, considering that it is still in the shakedown and testing phase. The problems encountered have been of a minor, but sometimes aggravating, nature. The total energy accumulated to date is about 1250 Mwd, all on the same assembly. The average expected life of the fuel assembly is 1400 Mwd.

Flow coastdown and power outage tests were conducted. It was demonstrated that the entire HFIR electrical load could be transferred satisfactorily from the preferred to the alternate feeder.

The first bank of new 1-1/8-in.-thick charcoal filters for the special building hot exhaust system has been installed and tested. The decontamination factors of these filters for elemental iodine and methyl iodide were measured as 20,000 and 4000 respectively.

Two flux mapping runs were made at ~1 Mw to measure the neutron flux in the vertical experiment holes. Eighteen ^{242}Pu target rods were received from Savannah River, and a cursory inspection indicates that some of the fins which position the rods in the can may have corroded or otherwise worn away. A detailed inspection is scheduled in one of the TRU cells to determine whether the targets are satisfactory for irradiation in the HFIR.

The Advisory Committee on Reactor Safeguards completed its review of the HFIR on May 6, 1966, and granted permission for operation at full power, 100 Mw. Written approval is in the mail; on receipt of the approval the reactor will begin an escalation in power beyond 20 Mw.

Instrumentation and Controls. — Debugging of the process and nuclear instrumentation is continuing as the operation encompasses more of the systems. The improved system performance is indicated by the decreasing number of false shutdowns recorded in the past two weeks.

A xenon study has been made on the analog computer to establish an operating procedure to be used during recovery from a scram. The procedure was determined on the basis of allowing the operator sufficient time to inject liquid poison into the system to prevent the reactor power from reaching the nucleate boiling level, should the rods become immobile at any time while burning out the excess xenon.

PHYSICAL RESEARCH

Reactor Operations. — The ORR and the LITR both operated approximately 87% of the time during March and April. At the ORR the last of 18 capsules of ^{241}Am was removed from the reactor core.

Orientation of a 24-in. valve in the ORR inlet water line was changed from a vertical to a horizontal position so that a failure of the support mechanism of the gate could not suddenly stop the water flow. A new pneumatic tube was installed in hole HN-2 at the ORR which will send samples to Building 3001 in approximately 4 sec after they leave the reactor.

The Bulk Shielding Reactor operated approximately 238 hr during this period, and revision of the reactor for 2-Mw operation was 43% complete at the end of April.

Unloading of the ORNL Graphite Reactor fuel was begun. Fuel slugs containing about 500 g of plutonium with the highest ^{239}Pu purity are being separated so that they may be processed separately and the plutonium used for special research purposes.

Waste Disposal. — Operation of the intermediate-level waste evaporator was begun on March 29. Since then, only 95,000 gal, or 15% of the total waste generated, has been disposed of in the soil seepage trenches. The trenches will probably not be used in the future unless an equipment failure occurs and the waste fills all available storage space before repairs can be made.

Some difficulties have been experienced in the operation of the evaporator, but all were of a type to be expected during initial operation. The worst difficulty has been a foaming problem which, more than any other factor, has limited the output of the plant to a point that little progress has been made in reducing the backlog of waste in the storage tanks.

The normal decontamination factor for the operation has been approximately 10^5. The concentration factor has varied over a wide range, depending upon the type of waste discharged from the operating building, and has averaged approximately 20.

Hot-Cell Operations. — The third ORR-AGCR capsule (P4B-3) was disassembled and examined in detail. A UO_2-graphite compatibility test was disassembled in a dry box (in-cell) maintained at a dew point of less than −40°F. Five fueled experiments were examined in detail for General Electric. One UO_2 in-pile meltdown experiment and one irradiated fuel meltdown from the NSPP were disassembled, photographed, and sampled. Disassembly operations were started on a failed SNAP generator, and examination of several uranium nitride capsules was begun. The nondestructive part of the examination of 8 fuel tubes and 24 coupons from an Army PM-fuel experiment was completed.

An MSRE valve was disassembled, and deposits of material were examined. The cladding from several heat source capsules was examined metallographically. About 275 tensile specimens and 200 impact specimens were tested; the eight creep-test machines continued in full operation. In the ORR cells, specimens and experimental capsules were recovered from 22 experiments; also, dimensional measurements were made on 12 beryllium pieces.

PHYSICS AND MATHEMATICS

Theoretical Physics: Elastic and Inelastic Scattering of Heavy Ions. — The elastic and the inelastic scattering of heavy ions from nuclei were considered in terms of a generalized optical model. Amplitudes for the single excitation of target or projectile were evaluated in a distorted-wave approximation, while amplitudes for more complicated excitations, such as double excitation or mutual excitation of both nuclei, were computed using the derivative relations of Austern and Blair. For the special case of double excitation, the results of the Austern and Blair theory were compared with the predictions of a coupled Schrodinger equation calculation. Spectroscopic information derived from these analyses is in good agreement with previous analyses.

Theoretical Physics: Optical Model for 30-Mev Proton Scattering. — The differential cross sections, polarizations, and absorption cross sections for the elastic scattering of 30-Mev protons from the nuclei Si, Ca, Fe, Ni, Co, Cu, Sn, and Pb have been analyzed in terms of the optical model. We find a smaller radius and a larger surface diffuseness than have previously been inferred for the real potential. A volume absorption strength of about 3 Mev is required, while the surface absorption term increases in strength as the atomic mass increases. The diffuseness of the imaginary potential is also larger for the heavier nuclei. Fits to the polarization reveal evidence for a spin-orbit coupling with radius and diffuseness different from those of the real central well, and the trend is for the spin-orbit diffuseness to be smaller, rather than the radius. The symmetry or $(N - Z)/A$ dependence of the potential may be ascribed either to the real well depth or to its radius; an average potential of both types is found which gives a good overall account of the data.

Theoretical Physics: Comparison of the Effective Two-Nucleon Interaction in 2 $s_{1/2}$, 1 $d_{3/2}$ Shell Nuclei with a Surface Delta Interaction. — An effort has been made to find an effective interaction for shell-model calculations of nuclei in the region A = 29 to A = 40. It is assumed that these nuclei can be described as having an inert 28-nucleon core and that all "active" particles are in the 1 $d_{3/2}$, 2 $s_{1/2}$ shell. The pertinent single-particle energies and two-body matrix elements were first assumed to be free parameters, and a search was made to find a set of these parameters which minimized the deviation between experiment and the calculated energy levels. Searches were made to fit the total binding energies of all states, to fit ground-state binding energies plus level splittings, and to fit only energy level differences. It was found that satisfactory fits could be obtained in each case. In this way an effective interaction is drawn from experiment. Moskowski has suggested a surface delta interaction (S.D.I.) as a simple approximation to the shell-model effective interaction. It can be argued that for such an interaction, the only parameters that enter are an interaction strength for T = 0 state, and an interaction strength for T = 1 states. Thus, where there are 15 two-body matrix elements to vary in searching for an effective interaction, there are only two numbers to vary if one assumes that the S.D.I. is the effective interaction. It is found that the fits to the experimental energies

with the two-parameter S.D.I. are quite comparable to the fits obtained with 15 two-body matrix elements for energy level differences. The predicted ground-state binding energy with the S.D.I. is not in as good agreement as the 15-parameter predictions. Similar results are found for the calcium isotopes. This work is a collaborative effort with P. W. M. Glaudemans and B. N. Wildenthal of Rice University, Houston, Texas.

Low-Energy Neutron Time-of-Flight Spectroscopy. – The 1.25-m scintillation detector installed in the RPI electron linac has been limited to neutron measurements below ~30 kev energy. The intense gamma flash from electron linacs usually overloads this type of detector so severely that it cannot recover for many microseconds after the electron pulse. Recent improvements in this detector have completely eliminated this problem, and measurements have just been obtained on a sample of iron from 25 Mev to 5 ev. In the energy range 25 to 3 Mev the detector responds to inelastic scattering in iron, and the inelastic scattering counting rate showed 18 strong resonances. These resonances are attributed to elastic scattering of neutrons from carbon in the polyethylene moderator. After correcting for recoil energy losses from the carbon scatterer, these resonances occurred at neutron energies of 3.5, 4.9, 5.4, 5.8, 6.2, 6.5, 7.2, 7.9, 8.6, 9.5, 11, 13, 14, 16, 21, 25, 30, and 35 Mev. In the energy range below 2 Mev the detector was responding to radiative capture in iron, and over 30 resonances were resolved in iron below 200 kev. Capture measurements were also made on samples of F, Na, S, Al, and Cr below 200 kev, and several resonances were observed in all samples. An ^{6}Li glass detector was combined with the large scintillator to measure scattering from the low-energy resonance in tungsten. The neutron widths of tungsten resonances below 27 ev determined by this measurement were in agreement with the results reported in BNL-325, 2d edition.

Low-Temperature, Nuclear and Solid-State Physics. – The $Fe_{0.48}Rh_{0.52}$ alloy has the interesting property of being antiferromagnetic below 330°K and ferromagnetic in the temperature region above 330°K to the Curie temperature at 665°K. The Mössbauer method provides a unique method of obtaining information about this alloy, in that it enables one to measure the temperature dependence of the thermal average of the electric spin projection of the iron in both magnetic phases, using the same apparatus and the same interpretative concepts. It is found that the Néel temperature for the antiferromagnetic phase is about 50°K less than the Curie temperature. A theoretical model has been developed which shows the relation between the Néel temperature, the Curie temperature, and the temperature of transition between the two magnetic phases. The model also shows the reason for the sensitivity of the transition temperature to the addition of small concentrations of the transition elements.

High-Voltage Experimental Program: Charge-State Distribution of Channeled 40-Mev ^{127}I Ions in Gold Single Crystals. – The energy loss of ions in the multi-Mev region is primarily due to inelastic electronic stopping. The decreased energy loss associated with channeling here therefore reflects the lower average electron density on the trajectory. Moreover, because of the reduced probability of small-impact-parameter collisions, channeling may lead to lower charge states, which also act to decrease the energy loss of channeled particles. To investigate this

question more thoroughly, a 40-Mev ^{127}I beam from the Oak Ridge tandem accelerator was passed through a thin gold single-crystal target (about 3500 A thick); the emerging beam was analyzed in an electrostatic-deflection system. The charge distribution was measured for beam incidence (1) in a random lattice direction and (2) in the [110] channel direction. In case (1), a Gaussian distribution with a most probable charge Z = 14.3 was obtained; case (2) showed a skewed distribution with a most probable charge Z = 12.5. The width of both distributions was about 5 charges (full width at half maximum). Assuming a Z^2 dependence of (dE/dx) and a constant electron density, the corresponding relative difference in stopping power is 10%, averaged over the distributions, while the actual relative difference in energy loss was measured to 43%.

High-Voltage Experimental Program: The $^{14}C(^3He,p)^{16}N$, $^{14}C(^3He,d)^{15}N$, $^{14}C(^3He,^3He)^{14}C$, and $^{14}C(^3He,\alpha)^{13}C$ Reactions at 2.0, 4.0, and 5.0 Mev. — Angular distributions of protons to the ground state of ^{16}N, and to states at 0.120, 3.34, 3.51, and 3.96 Mev, were measured. The state at 3.34 Mev was determined to be $(0, 1)^+$. The other states were determined to be negative-parity states, $(0, 1, 2)^-$. The ground-state deuterons to ^{15}N are predominantly direct interaction and confirm the $1/2^-$ ground-state spin and parity assignments. The angular distribution of elastically scattered ^3He particles was measured at 5.0 Mev. Angular distributions of alpha particles leaving ^{13}C in its ground and first excited states were measured. Elastic-scattering angular distributions of alpha particles from ^{13}C were also measured at 15, 18, and 20 Mev, in hopes of being able to obtain consistent optical-model parameters for both entrance and exit channel elastic scattering and the $^{14}C(^3He,\alpha)^{13}C$ reaction. Analysis is in progress.

CHEMISTRY

Effects of Radiation on Analytical Methods. — The acidity of a 10 M LiCl—0.2 N HCl solution containing 10% formaldehyde increases with gamma irradiation. A differential titration in 94% acetone medium revealed that the HCl content had decreased, and two weaker acids had appeared. Since these latter acids cannot be titrated separately in water, they must have pK values of the order of 4. The stronger and more abundant (about 90%) of the two weak acids is believed to be formic acid.

The acidity functions of formic acid and HCl in water and in 10 M LiCl were determined. The activity of formic acid in 10 M LiCl is greater than that of HCl of the same concentration in water. It is assumed that this indicates that formic acid is at least as effective in repressing metal ion hydrolysis in 10 M LiCl as HCl is in water. This would mean that the radiolysis of formaldehyde to produce formic acid may be one way to counterbalance the loss of HCl in acid chloride solutions and prevent the hydrolysis of many cations.

Associated with the high activity coefficients of acids in 10 M LiCl is the high tendency of the acid to disappear from solution. Tests run over a period of two weeks showed that there was measurable loss of HCl and formic acid from 10 M LiCl medium, while the amount of acids in water remained unchanged when these solutions were kept open to the air. The

rate of loss of HCl is about 0.05 meq/ml when the original acidity is near 0.3 N. This is comparable with the order of acid loss encountered in some of the transuranium processing operations. The rate of loss of formic acid is about one-fourth to one-third the HCl rate.

The Alizarin Red-S (AlRS) reaction with zirconium is the basis for one of the more sensitive colorimetric methods for that element. The quinone structure of the complex, however, is extremely susceptible to radiation damage, and, therefore, the measurement of the absorbance of this complex under conditions of radiation may lead to erroneous results in the determination of zirconium. Work has been carried out to establish a method for the measurement of zirconium as the Zr-AlRS complex which will not be so susceptible to radiation as is the colorimetric measurement of the complex. It has been found that the electrometric oxidation of the hydroxy groups of the complex at the pyrolytic graphite electrode results in two anodic waves. The first at ~+0.7 v vs the S.C.E. is due to the oxidation of the free AlRS, while the second at ~+1.0 v vs the S.C.E. is due to the oxidation of the Zr-AlRS complex. Either the reduction in the wave at ~0.7 v or the increase in the wave at ~1.0 v may be correlated with the zirconium concentration. Further work is being carried out to determine optimum conditions and to study the mechanism of the observed phenomenon.

Separations Chemistry Research: Biochemical Separations. — Membrane osmometry is a direct method of determining molecular weights of transfer ribonucleic acids (tRNA's). Experiments were made at several tRNA concentrations and temperatures from 13 to 37°C. A plot of π/c vs c (where π is osmotic pressure and c is concentration) for crude tRNA solutions showed a slight positive slope, while the slope for dialyzed crude tRNA solutions was near zero. It appeared that dialysis reduced charge and ionic interaction effects; also, there appeared to be a slight trend toward decreasing molecular weight with increasing temperature. The apparent molecular weight of crude *E. coli* B tRNA varied from 33,200 to 36,500.

Gel permeation chromatography is an alternate comparative method of measuring the molecular weights. Columns of Bio-Gel P-100 (a polyacrylamide gel) were calibrated with serum albumin, egg albumin, and β-lactoglobulin. These proteins were chosen since they can be represented as ellipsoids of revolution with an axial ratio of 4:1, a value believed to be representative of tRNA. Excellent linear correlation was obtained when elution volume was plotted against the logarithm of the molecular weight. The value for crude *E. coli* B tRNA was 39,000, in good agreement with the values obtained previously by osmometry. A change from phosphate to sodium chloride as the supporting electrolyte revealed the presence of a higher-molecular-weight RNA contaminant. Thus the value of 39,000 represents an average molecular weight for the sample. The chromatographic peak representing mixed tRNA's free of contaminant RNA indicated an apparent molecular weight of 26,000.

Calibrated columns of Bio-Gel P-150 and P-300 were next used to determine the molecular weight of highly purified *E. coli* B $tRNA^{Phe}$ and $tRNA_4^{Leu}$. The respective values were 26,000 and 29,500. These values are in excellent agreement with reported values: 26,600 for yeast alanine tRNA, and 26,500 for mixed yeast tRNA's.

Separations Chemistry Research: Solvent Extraction. — In the expanding study of cation exchange extraction by organic acids, the only available acid stronger than the phosphoric acids has been dinonylnaphthalenesulfonic acid, which has been of marginal value because it is an indefinite mixture of isomers and because its alkali salts are excessively soluble in aqueous solutions. A homolog of higher molecular weight, 2,6-di-n-dodecylnaphthalene-4-sulfonic acid (HDNS), has now been prepared and shown to be negligibly soluble in aqueous solution: less than 2 ppm HDNS or NaDNS distributed to water from 0.1 M solution in benzene. Preliminary tests have confirmed sodium-hydrogen cation exchange at acidities up to at least 3 M.

Foam Separation Studies. — As part of a basic study of the mechanisms of foam separation, the effect of foam reflux on volume reduction and on decontamination factors for radioactive strontium is being investigated. The apparatus used in these experiments permits any chosen fraction of the condensed foam stream to be returned to the countercurrent (liquid vs air) foam separation column. Thus the effects of foam reflux ratio and gas/liquid volume ratio can be studied as process variables in the removal of strontium from aqueous dodecylbenzene sulfonate (DBS) solution.

The distilled-water feed stream, 10^{-6} M in Sr^{2+} carrier plus ^{85}Sr tracer (pure gamma emitter), was fed continuously into the top of the foam column at a flow rate equivalent to 41 gal ft^{-2} hr^{-1}. Part of the surfactant (i.e., enough 5000-ppm DBS solution to yield a feed concentration of 82 to 83 ppm) was introduced continuously just before the feed entered the column, while the remainder (equal to about another 13 ppm in the feed) was added to the liquid pool at the bottom of the column.

Strontium decontamination factors ranged from 6.3×10^3 to 1.7×10^4, respectively, when 42 to 97.5% of the foam was refluxed to the foam column, where the gas/liquid volume ratio was maintained practically constant at 12 to 15. The volume reduction factors achieved under these conditions were 27, 58, 90, 136, 538, and 805 for 42, 71, 82, 87, 95, and 97.5% foam reflux respectively.

When foam reflux was held constant at 95% while the gas/liquid volume ratio was decreased from 13.5 to 4, the ^{85}Sr decontamination factor remained constant at about 5×10^3 (within experimental error), whereas the corresponding volume reduction factors increased from about 500 to about 4500.

The conclusion reached was that by lowering the gas/liquid volume ratio and/or increasing the foam reflux ratio, both high decontamination and high volume reduction factors could be achieved simultaneously.

Volatility Studies. — A desirable step in volatility processing would be the capability of reversibly sorbing PuF_6 by a solid sorbent as in the UF_6-NaF system. In previous work we showed that some sorption of PuF_6 occurred on each of the fluorides of groups I and IIA metals, but that desorption could not be confirmed in 15-min tests. A fluorine sweep was used. Even for 10-hr tests, no significant desorption from NaF was found at 600°C in the presence of fluorine. In a continuation of the longer desorption tests, partial desorption from CaF_2 has now been achieved, and almost complete desorption from LiF. For these tests the apparatus was the same as that used previously, consisting of two traps (3/8-in. OD × 0.035-in. wall) in series. The first trap was filled with about 2 g of

test sorbent, and the second trap with 2 g of NaF, which at 200°C efficiently collects PuF_6.

With CaF_2, 7 and 15% of the previously sorbed plutonium (about 40 mg) was desorbed as PuF_6 in duplicate 10-hr tests with a fluorine flow of 60 ml/min through the 600°C sorbent bed. No significant desorption was noted at lower temperatures or with reverse flow.

Desorption of PuF_6 from LiF proceeded to 95 to 98% completion in about 9 hr at 400°C, in 6 hr at 450°, and in 4 hr at 500°. At 600°C the desorption rate was about the same as that at 400°.

Sorption of PuF_6 by LiF appears to have an optimum temperature range near 300°C. In 20-min sorption tests with fluorine flows of 30 ml/min, passing about 30 mg of plutonium (as PuF_6) into 1.7 g of porous LiF, 24.7% of the PuF_6 was sorbed at 200°C, 73.6% at 250°, and 98.3% at 300°. In three additional tests at 300°C, the retention was 98.9, 98.5, and 99.3%.

Separation of PuF_6 and UF_6 has been demonstrated in duplicate tests with 1.7 g of LiF at 300°C in series with 2 g of NaF at 150°. In 20 min with fluorine flow of 30 ml/min, about 40 mg of plutonium and 20 mg of uranium, as the hexafluorides, were passed into the first bed. The LiF bed retained 98 and 95% of the plutonium and less than 0.1% of the uranium, whereas the NaF bed retained the remaining plutonium and over 99.9% of the uranium.

Chemistry of Carbides and Nitrides. — The reactions of thorium monocarbide (ThC) with nitric acid solutions are being studied. Scouting experiments showed that ThC would react with dilute nitric acid solutions; this is in contrast to UC, which is practically inert in 0.005 to 0.5 M HNO_3. In the first of a series of quantitative experiments, arc-melted $ThC_{0.98}$ (carbon-deficient ThC containing about 5% ThC_2 as an impurity) was allowed to react with 0.3 M HNO_3 at 90°C. The initial HNO_3/ThC mole ratio was about 15. The reaction was practically complete in about 90 hr. The gas evolved, 79.5 ml (STP) per gram of carbide, had an average composition of 18.3% CH_4, 32.2% CO_2, 4.4% CO, 38.1% N_2O, and 6.9% NO. About 55% of the carbide carbon was found as gaseous products; the remainder formed species that were soluble in the final solution. The reaction with 0.3 M HNO_3 was much slower than the reaction with water, which required only 3 to 4 hr for completion. Reaction with 0.3 M HNO_3 also formed less gas: 79.5 ml per gram of carbide vs 91.2 ml/g with water.

Chemical Engineering Research: Stacked-Clone Contactor. — A new solvent extraction stacked-clone contactor, consisting of 18 stages and featuring an integral pump-clone design whereby all stage pumps are driven by a common shaft, has been fabricated, installed in a test rack, and operated with the system 1 M $NaNO_3$–18% TBP–Amsco to extract uranyl nitrate. Preliminary results verify that this contactor performs in a manner equivalent to the 11-stage experimental unit reported previously. Hydraulic, flooding, and mass transfer experiments have been performed. Each stage pump develops about 21 psig, and the pressure increase per stage in the direction of aqueous phase flow averages 1.6 psi. Solution throughputs at flooding are 3.0 liters/min (aqueous/organic ratio of 3, 40°C), and mass transfer efficiencies of 60% have been calculated. The rapid response to control, low solution holdup, and low residence time of the device are inherent in this plant prototype, which is suitable for eventual testing at full radioactivity levels.

Chemical Engineering Research: Macromolecular Biological Separations. — A process for separating and isolating several transfer ribonucleic acids (tRNA's) from a mixture of about 20 is being scaled up. This process consists in the selective elution of the desired tRNA from a diatomaceous earth column saturated with a quaternary amine. The eluting solution contains sodium chloride and is saturated with isoamyl acetate. The concentration of sodium chloride in this solution is steadily increased during an elution run. Some scale-up difficulties have been encountered: obtaining adequate separation in a column and the short operating life of column packing.

A series of runs under varying conditions on 1/2-, 1-, and 2-in.-diam columns 8 and 24 ft long failed to yield entirely adequate separation of the phenylalanine tRNA from the other tRNA, particularly from the leucine tRNA. Furthermore, the separation achieved under a given set of conditions was progressively lessening. Trace-metal contamination was suspected, but a wash of a column with a solution of HCl and Versene (to dissolve and complex the metal ions) did not improve the separation that could be achieved. Several lots of isoamyl acetate of differing degrees of purity were tested, and although good separation of the leucine from phenylalanine was achieved in several runs, no relation between the solvents used and the separation achieved was noted. Furthermore, only a few runs could be made before these columns, too, failed to give adequate separation. It was finally concluded that the quaternary amine on the column packing was being washed off by the gradient solution. Operation conditions were altered to reduce this amine loss, and the life of a batch of column packing was extended considerably. The entrainment of globules of isoamyl acetate floating on the surface of the sodium chloride eluting solution is suspected as a cause for the release of amine from the column.

A large batch of packing that had been used previously was dried and re-treated. A test of this packing showed that complete rejuvenation had been achieved. Apparently, packing can be prepared, run until the phenylalanine and leucine can no longer be adequately separated, dumped, rejuvenated, and reused. This will obviously greatly reduce the amount of packing required and make the problem of deterioration much less serious than it would otherwise be.

Since an answer to the problem of packing stability has apparently been found, the large batch of mixed tRNA's that has been prepared can now be processed to separate phenylalanine, leucine, and tyrosine from the others and from each other. Once significant quantities have been separated, the subsequent purification steps will be evaluated.

Chemical Physics: Microwave and Radio-Frequency Spectroscopy. — Studies of the paramagnetic resonance spectra of simple, short-lived, free radicals present in liquids during photolysis continued. Photolysis of hydrogen peroxide contained in alcohol solution gives rise to the OH radical, which abstracts hydrogen from the alcohol, giving the observed radical. Radicals that we most frequently observed in earlier work were those formed by abstracting an α-hydrogen from the alcohol. We have now studied the effects of water and acid on radical yields. The addition of water to t-butyl, isopropyl, and ethyl alcohols enhanced the formation of radicals resulting from abstraction of β-hydrogens. The principal effect of acidifying the solutions was the enhancement of the rate of exchange

of hydroxyl protons. Hyperfine couplings and g values were measured for $(CH_3)_2(\dot{C}H_2)COH$ and $CH_3(\dot{C}H_2)CHOH$. The primary product from ethylene glycol with or without water was $\dot{C}HOHCH_2OH$, but with small amounts of acid this radical was not obtained. In its place was $\dot{C}H_2CHO$. Hyperfine couplings and the g value were measured for this radical.

High-Temperature Aqueous Solution Chemistry: Solubility of Magnetite. — In the continuation of a previously reported study of the high-temperature equilibrium between magnetite (Fe_3O_4), hydrogen, and water (ORNL-3845),

$$Fe_3O_4(s) + 6H^+ + H_2(g) = 3Fe^{2+} + 4H_2O \ ,$$

measurements have been obtained in acid solutions in order to determine the dependence of the equilibrium on acidity as well as to prevent precipitation of dissolved iron which was found to occur on cooling some of the saturated solutions. From these data we hope to determine the equilibrium constant for the above reaction as well as the extent of Fe^{2+} hydrolysis. The method of measurement, as previously, is to pump the test solution slowly through a column packed with Fe_3O_4 and then to analyze the discharging equilibrated solution. We are now using nonradioactive Fe_3O_4 in order that a sufficiently large amount of it may be placed in the column to provide the necessary area of contact with the solution. The results have, in general, been found independent of flow rate over a wide range, indicating attainment of equilibrium. Typical results at 260 and 300°C are as follows:

	Concentration of Fe^{2+} (μM)	
	260°C	300°C
10 μM HCl	2.7	1.0
40 μM HCl	12.0	6.0

At lower temperatures the results thus far show only that Fe_3O_4 reacts nearly quantitatively with the acid initially present at concentrations greater than ~10 μM. At lower acidities, at these lower temperatures, precipitation of iron on cooling of the saturated solution remains a problem. The apparatus is now being modified to permit the injection of acid into the equilibrated solution before it is cooled as a means of overcoming this difficulty.

High-Temperature Aqueous Solution Chemistry: Isopiestic Studies. — The isopiestic study of mixed electrolyte systems containing multivalent ions has been continued with standard sodium chloride being compared with several salt pair systems, $NaCl-MgCl_2$, $NaCl-CaCl_2$, and $NaCl-Na_2SO_4$, in the

mole ratios 9:1. The data indicate that the isopiestic ratios

$$R \equiv (I \text{ standard})/(I \text{ salt pair})$$

show the trend $R_{NaCl\text{-}MgCl_2} > R_{NaCl\text{-}CaCl_2} > R_{NaCl\text{-}Na_2SO_4}$. Consequently, concentrations alone may not completely define the osmotic behavior of solutions of mixed ion types.

It has been tentatively noted that whereas the ionic strength principle more faithfully describes the concentration dependency of the osmotic coefficient of these mixtures in the dilute range (<1 m), and the "total ion" (or osmolality) concept describes that in the high concentration range (>2 to 3 m), the geometric mean of the two has proven useful in the systems investigated.

Physical Chemistry of Molten Salts. — Equilibrations of molten fluoride melts with alloys of thorium in lead are being carried out in a study of oxidation-reduction reactions in which thorium serves as a reductant and lead serves as a sink for reduced species. In the course of these experiments, which are directed toward the basic chemistry involved in processing breeder blankets, information on the solubility of thorium in lead was required. Accordingly, samples from saturated solutions of thorium in lead were obtained over the temperature interval 400 to 600°C. The results could be represented by $\log X_{Th} = -4175/T + 1.055$, where X_{Th} is in mole fraction, and T is the absolute temperature. This corresponds to a heat of solution of 19 kcal/mole and a solubility at 600°C of 1.85×10^{-4} mole fraction.

Isotope Chemistry. — Infrared and Raman spectra of the hexafluoride ions of silicon, germanium, and tin have been observed. The spectra were obtained from crystals grown by slow evaporation at room temperature. The infrared observations were made using the KBr pellet technique and a grating-type spectrometer. The three Raman-active and two infrared-active vibrational frequencies for each compound are listed below:

Observed Raman and Infrared Frequencies for Na_2SnF_6, K_2GeF_6, and Na_2SiF_6 Crystals (cm^{-1})

	ν_1 Raman	ν_2 Raman	ν_3 IR	ν_4 IR	ν_5 Raman
Na_2SnF_6	591	480	566	300	253
K_2GeF_6	623	468	604	339, 359	335
Na_2SiF_6	663	478	738	483	407

The data confirm the octahedral structure of these ions (point group O_h).

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TRANSURANIUM-ELEMENT PROCESSING

Curium Recovery Facility. — The third of four phases of the current ^{242}Cm production campaign is nearly complete. More than 20 g of ^{242}Cm is to be produced in the campaign. Seventeen irradiated ^{241}Am targets (5 Martin, 12 ORR) have been dissolved and processed through the mixer-settler system. Nitrate product solution (4.5 g ^{242}Cm, <100 ppm Cl^-) from the Martin targets was transferred to the Isotopes Division. Product solution (4.7 g ^{242}Cm) from the first set of six ORR targets was transferred to the product storage vessel in cell 3. Product from the remaining six ORR targets is being converted to the nitrate.

Experimental Engineering Studies. — Hydraulic testing of the 3/4-in.-ID glass pulse columns, using the Pharex flowsheet (separation of americium and curium from the heavy actinides), was continued. A serious problem with flooding of the columns was evident during checkout in TRU. Two problems are obvious: (1) impurities in the solvent tend to increase plate wetting by the solvent and subsequently cause phase inversion; (2) cleaning of the plates to remove the organic film deposited by prolonged contact with the solvent is not simple and straightforward.

Various treatments of the solvent, 0.5 M 2-ethylhexylphenylphosphonic acid in Amsco, included contact with activated alumina, silica gel, activated charcoal, finely divided tantalum, 6 M and 12 M HCl, 10% Na_2CO_3, and ethylene glycol. The most successful treatment consisted in contacting the Amsco with alumina, washing the 0.5 M solvent with five volumes of 6 M HCl, followed by treatment with 12 M HCl, in which a third phase was formed and discarded. This procedure permitted column operation of about 26 hr before plate wetting and flooding occurred.

Tested plate-cleaning procedures included washing with 10% Na_2CO_3, acidified ethyl alcohol (10% HCl), acetone, trichloroethylene, soap (Knox-60, 12 g/liter), dichromate cleaning solution, aqua regia, 10% NaOH, Versene, Haemo-sol (14 g/gal), RD 16F (descaler), and 1% HF–3% HNO_3. At first, treatment with acidified alcohol followed by a soap wash seemed successful, but cleanings following subsequent solvent contact were less effective. Flaming of the plates with an acetylene torch (deposition of an oxide layer) appeared to make them less susceptible to organic wetting, allowing an acid-treated batch of solvent to run for 30 hr. Testing of these plates is in progress.

Deposition of an oxide layer on the tantalum plates by anodizing in 1% phosphoric acid will be investigated.

TRU Operations. — Leak checking of all cubicles is complete, and leak testing of the cell bank was begun. Work has started on the shielded cave facility in room 111. Eight stainless steel ferrules had been improperly installed on Zircaloy-2 process lines and were replaced with Zircaloy-2 ferrules.

Checkout of process equipment continues, with additional runs being made in the dissolution, centrifugation, and first-cycle solvent extraction equipment. The second cycle was not operated, pending resolution by development groups of a problem with plate wetting and subsequent flooding.

The entire shipment of 18 irradiated HFIR targets has now been received from Savannah River and is being stored in the HFIR pool.

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METALLURGY AND MATERIALS

Physical Properties Studies. — Thermal conductivity was measured on a W–26% Re sample over the temperature range 20 to 140°C. The results were used with previous electrical resistivity data to express the thermal conductivity (w cm^{-1} deg^{-1}) in the form $2.695 \times 10^{-8}\ T/\rho + 9.11/T$, where ρ is the electrical resistivity in ohm-cm, and T is the absolute temperature in °K.

The thermal conductivity of Nb–10% W (an alloy similar to D-43) was determined to 1000°C in the radial heat flow apparatus. It increased steadily with increasing temperature from 0.46 w cm^{-1} deg^{-1} at room temperature to 0.63 w cm^{-1} deg^{-1} at 1000°C. Measurements on T-111 have been made to 450°C and will be extended to 1000°C. A special disk of Nb–10% W, machined and instrumented to allow electrical resistivity and Seebeck coefficient measurements to be made in the radial apparatus, has been installed with the T-111. This technique of measuring electrical resistivity and Seeback coefficient has worked satisfactorily up to 450°C and will be extended to higher temperatures with the T-111 thermal conductivity measurements.

Physical Ceramic Studies. — Continuing our investigation of material transport in sintering, we examined the initial densification kinetics of ZrO_2–14 mole % CaO compacts. The diffusion coefficient (7.4×10^{-14}) calculated from isothermal densification data at 1350°C agreed well with a value extrapolated from zirconium diffusion coefficients in similar ZrO_2-CaO determined by Rhodes and Carter using a tracer method. However, the diffusion coefficient calculated from densification data taken as the ZrO_2-CaO compacts were being heated from 1200 to 1350°C was 38 times higher, suggesting that contributions by other material-transport methods occurred.

Structure of Metals. — The control of preferred orientation (texture) is often extremely important to the successful fabrication of metals into usual structural components by deformation and heat treatment. Our aim is to gain a clearer understanding of the preferred orientations that develop in body-centered cubic materials, particularly the high-melting transition metals and alloys. We obtained x-ray diffraction pole figures on randomly oriented polycrystalline niobium samples that had been cold deformed 20, 40, 60, 80, and 95%. The resulting textures can be described approximately in terms of at least three "ideal" crystallographic orientations. The general trend is for the lattice reorientations to follow the pattern {544} <225> to {223} <110> to {001} <110> with increasing deformation. The last reorientation is approximately in agreement with that predicted by the latest theories, but the stability of the {544} <225> orientation at low reductions has not been predicted.

Solid Reaction Studies. — Self-diffusion of chromium, Cr–20% Ni, and Cr–44% Ni was measured over the temperature ranges 1000 to 1900°C, 1000 to 1525°C, and 1050 to 1340°C respectively. The penetration plots for experiments carried out within approximately 100° of the melting point were nonlinear and yielded two diffusion coefficients. By comparison with the results from single-crystal specimens, the higher diffusion coefficient was shown to be due to grain-boundary diffusion, possibly as a result

of local melting at grain boundaries. In addition, the Arrhenius plots were curved upward within this temperature range almost identically to that for self-diffusion in vanadium.

CONTROLLED THERMONUCLEAR RESEARCH

The DCX-1 Injection-Accumulation Experiment. — During this report period, experiments were performed by the Plasma Stabilization Group using a variety of end-wall configurations whose purpose was to suppress losses caused by the instability in DCX-1. Recent theoretical calculations show that with appropriate inductive configurations, the negative-mass instability may be cured. Experiments here reveal that, indeed, configurations can be found which profoundly alter the energy spread, rf emission, and density of the plasma. However, the coupling of the energetic plasma to the reactive end walls is only effective when the walls are in substantial contact with the energetic plasma (a detrimental contact); and it appears that the cold-plasma conductance alone is not sufficient for strong coupling. The plasma density is always greater when the walls are retracted to positions outside the fast ion plasma. It is hoped that these studies when compared with theory may reveal structures that can be more effective in stabilizing the plasma. The experiments have been completed.

Modifications to the apparatus are being made to accommodate a 20-kev neutral injection experiment which will extend the studies on microinstabilities.

Properties of Plasmas Created by Electron-Cyclotron Heating. — The transverse (Z axis) distribution of trapped 20-kev protons in INTEREM has been measured with a scanning detector. The trapped proton distribution is sharply peaked on the midplane; the peak has a full width at half maximum (FWHM) of about 1 cm. There is also a low broad "talus slope" distribution with a 2- to 3-cm FWHM. This is surprising, since the beam enters the facility through a hole about 5 cm in diameter. A large variation in the ion-source focus coil current does not cause large spreading of the observed distribution. The presence or absence of the electron-cyclotron plasma does not significantly broaden the peak width.

The charge-exchange decay time of the 20-kev protons after beam turn-off is not a function of the transverse position. The previously observed "long tails" on the decay are usually not seen now, due probably to the lower microwave power available.

The facility cannot be operated at high power at gage pressures below 2×10^{-6} torr, apparently due to plasma bombardment of contaminated surfaces. The contamination may be due to oil streaming from the 20-kev neutral-beam drift tube.

Properties of Plasmas Created by Beam-Plasma Interaction. — Two neutron counters have been placed inside the vacuum chamber of Burnout V. One is near an end electrode, and the other is on the midplane of the device, both being about 15 in. from the magnetic axis. In these positions, they are each partially shielded by the magnet coil and its cooling water from seeing the same source of neutrons. With this arrangement of counters, a test source was placed at the anticathode end which produced a

neutron flux at the end counter 13 times greater than at the counter on the midplane.

The response of these neutron counters with Burnout V in operation clearly indicates neutron sources other than at the end electrodes. The ratio of end-counter response to midplane-counter response is usually about 2/1 in operation and can be made to vary from 1.8/1 to 3.1/1 by adjustment of gas feed rate and power input. The neutron source strength of the midplane region appears to be about 10^5 neutrons/sec.

Ion Source and Ion Beam Technology. — The primary effort during this report period has been the evaluation of the components for 20-kev neutral beam injectors for DCX-3 and INTEREM. Two new all-epoxy, non-PIG (horizontal vacuum insulator surface), 0- to 100-kev accelerator columns have been designed and tested for these applications. These systems perform very satisfactorily and can be used either for a vertical or a horizontal injector. In conjunction with the DCX-1 group, a program has been started to use this equipment to evaluate various neutral forming cells with intense hydrogen ion beams (H^+ or H_2^+) in the energy range 20 to 60 kev. Preliminary results with two magnesium cells have been very encouraging.

Theoretical Plasma Physics. — A recent comparison between a theoretically predicted instability threshold and experimental observations has demonstrated the necessity of augmenting existing analysis with a much more detailed analysis of the propagation characteristics of unstable waves. The experiment, performed in the DCX-2 facility, showed that stable, highly anisotropic energetic plasmas can exist in circumstances where theory would have predicted the spontaneous emission of rf signals at the first few harmonics of the ion gyrofrequency. The "central peak" plasma in DCX-2 has a mean perpendicular ion energy of ~500 kev, a mean parallel ion temperature of ~500 ev, and an ion density $\approx 1 \times 10^8$ cm^{-3}, with at least as many cold ($T_e \lesssim 50$ ev) electrons. The plasma is predicted to be unstable with respect to the "Harris" modes associated with the large ion temperature anisotropy for densities in excess of $\approx 1 \times 10^7$ cm^{-3}. That is, the theoretically predicted threshold density for this instability is at least a factor of 10 too small when judged by experimental observations.

We are presently attempting to analyze the characteristics of the modes which are predicted to be unstable in this plasma. If the growing waves are convectively unstable (grow in space as e^{kz}) rather than absolutely unstable (grow in time as $e^{\gamma t}$), then it may well be that the spatial growth rate is too small to permit the waves to grow to observable amplitudes before they encounter strong damping in the axial density gradients at the ends of the plasma. For convective instabilities, it is necessary to know not only the density thresholds at which growth may occur, but in addition the critical length determined by spatial growth rates and reflection-transmission-damping rates in the density gradients.

Atomic Cross Sections. — Additional measurements have been made of the electric-field ionization of the excited electronic quantum states of H, H_2, and H_3. The experimentally determined magnitude of the H_2 and H_3 state population was less than that of H, due mainly to secondary ionization and dissociation processes which occurred in the electric-field region. Excited H state population formed by electron capture collisions of protons in H_2 gas was dependent on particle energy. For H energies greater

than 60 kev the population was approximately constant; whereas for lower energies the population decreased, indicating a steeper dependence than the predicted n^{-3}.

Silicon surface-barrier detectors are now used routinely with an energy resolution of 1.8 kev. A new neutral-particle detector has been developed which uses the high-resolution silicon-barrier detectors to measure secondary electrons from a 15-kv negatively biased copper target. Pulse spectra were composed of several resolved groups, with each group corresponding to the number of secondary electrons emitted per incident neutral. Comparison of the ratio of the magnitudes of each group indicated the average particle energy, while the total count rate was a quantitative measure of the incident-particle flux. The detector system was used also for a detailed study of secondary emission.

BIOLOGY AND MEDICINE

SOMATIC EFFECTS OF RADIATION

Medium(Low)-Level Long-Term Effects of Radiation. — To study the effect of the microbial environment on aging, germfree (GF) and conventional (CONV) irradiated and unirradiated mice were examined with respect to their life span and incidence of disease. A non-inbred albino (ICR) and an inbred albino (RFM/Unf) strain have been studied to date. Conventional mice were reared with "conventional" techniques such that the microbial environment was essentially uncontrolled. Germfree mice were raised in rigid isolation inside plastic film isolators free of detectable microorganisms. Irradiated mice were either survivors of $LD_{50/30}$ experiments receiving 600 to 900 r at 12 weeks of age (ICR) or were exposed to 300 r at 5 to 6 weeks of age (RFM).

Unirradiated GF RFM mice had an increased survival rate in the middle one-third of the life span when compared with their CONV counterparts, and a similar increase was seen in GF ICR mice when mice dying with cecal volvulus (a disease seen almost exclusively in GF mice) were omitted. This difference in survival appears to be associated with infectious diseases of the CONV mice and tends to diminish in the last third of life, so that survivorship is not greatly different in the last decile.

In irradiated RFM mice, the GF groups also had greater survival in the middle third of the life span, but the differences were largely obscured by the high incidence of leukemia and could be ascribed solely to the absence of myeloid leukemia in the GF mice. Irradiated GF ICR male mice, however, had considerable less survival in the last two-thirds of the life span than their CONV counterparts. Whether this difference is ascribable to a specific disease or to an unusually small life-shortening effect of radiation in the CONV group is unknown at this time.

Irradiation of GF mice resulted in life shortening in both strains. When all causes of death were considered, the longevity was shortened by approximately 125 days in ICR male mice, by approximately 300 days in RFM females, and by 200 days in RFM male mice. In the latter strain, early induction of lymphatic leukemia was the primary cause of life shortening,

since longevity was reduced by only about 85 days in mice not dying with that disease. The life shortening observed in ICR male mice, however, was not attributable to lymphatic leukemia, since mice not dying with the disease had as great (or greater) a life-span reduction as did those dying of all causes.

The data suggest that although the absence of detectable microbial flora and fauna may alter the form of the survival curve, and may increase the mean age of death, it may not increase the maximum age of death. It is also evident that senescent changes and the life-shortening effect of radiation occur in germfree mice as well as in conventional mice, suggesting that the microbial environment is not crucial to the development of such phenomena.

Gnotobiotics. — Polyarteritis of the aorta and major arteries has been noted with unexpectedly high frequency in aging male and female noninbred RF mice of the Oak Ridge subline. The incidence and age distribution of the lesions are not known precisely, but the disease has been observed in up to 20% of the mice older than nine months of age that have been examined histologically in various experiments. It has not been noted in younger mice, nor has its incidence been detectably affected by whole-body irradiation early in life. The lesion is characterized in its acute stages by infiltration of polymorphonuclear and mononuclear cells throughout the wall of the affected vessel, with fibrinoid necrosis of the media. Aneurismal dilatation, rupture, and fibrosis of the affected vessel have been noted in the later stages. Sites of predilection include the proximal aorta and the coronary, renal, uterine, ovarian, and splenic arteries. The pathogenesis of the disease is under study.

Physiology of Blood Platelets. — During their maturation, megakaryocytes become polyploid and their cytoplasm increases and becomes highly granular. Microspectrophotometric measurement of DNA of individual megakaryocytes of rats confirmed the presence of 4, 8, 16, and 32 N ploidy classes. However, the frequency distribution of these ploidy classes is unlike the distribution of age groups of megakaryocytes based on morphological classification. Since only "immature" megakaryocytes were initially labeled upon injection of tritiated thymidine (^{3}HTdr), although the development of polyploidy and hence the synthesis of DNA have been associated with maturation of megakaryocytes, it was postulated that a very active period of DNA synthesis produces polyploidy of various degrees (4 to 32 N) prior to differentiation of the cytoplasm of megakaryocytes. To investigate the hypothesis, both the amount of DNA and the labeling with ^{3}HTdr were measured in the same cells of rat marrow. The labeling index of 8, 16, and 32 N megakaryocytes was only 0 to 9% 1/2 hr after injection of ^{3}HTdr and gradually increased in each ploidy class, reaching 60 to 90% at 72 hr. The grain count of these cells appeared to remain relatively constant throughout this time period. The results suggest that chromosomes of unrecognized megakaryocyte precursors undergo repeated, almost continuous replication to various ploidy levels, whereupon the megakaryocytes pass into a recognized phase of cytoplasmic maturation in which replication is essentially absent. The relative constancy of the grain count argues against a major reutilization of ^{3}HTdr.

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RADIATION GENETICS

Human Cytogenetics. — Work is being continued on the induction of chromosomal lesions by chemical mutagens. The cytological experiments using cytosine arabinoside as the chromosome breaking agent are completed, and the data show that there is no apparent relationship between the inhibition of DNA synthesis and the induction of chromosomal aberrations. Cytosine arabinoside is capable of inducing aberrations at all times of the cell cycle of mammalian cells.

Preliminary experiments being conducted with Drs. Trosko and Zimmerman indicate that cytosine arabinoside stimulates a nuclease reaction in the cell that is degrading cellular DNA. It is hoped that further investigation will substantiate this observation and correlate this nuclease activity with the induction of chromosomal aberrations.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — As part of a program to study in vitro recovery of x-irradiated mouse bone marrow cells, we have tested the effect of temperature during irradiation on cell survival. The cells were irradiated at either 2, 25, or 37°C, then quantitatively tested for their reproductive capacity by the spleen ^{59}Fe method. Curves constructed from the data clearly show that radiation temperature does not appreciably influence the radiosensitivity of the marrow cells. The respective D_{37}'s and extrapolation numbers are 74 r and 1.5, 79 r and 1.8, and 70 r and 1.5. This observation implies that the radioprotective effect of hypothermia in mammals as reported by other investigators is probably related to something (decreased O_2 to tissues) other than temperature per se. Secondly, our data are in contrast with some of those obtained with mammalian tissue culture cells for which radiation temperature has been found to influence survival such that some protection is afforded at 2°C. This is an example in which extrapolation of data obtained from studies with altered cell lines in culture to normal mammalian cells may not be made without qualification.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation. — Work on the scintillation detector for neutron diffraction patterns is continuing. A single-stage image intensifier on loan from the AEC Radiation Instrument Branch is being used to obtain experience with such devices. Measurements of gain, pulsed-light response, and resolution were made. Alpha sources, collimators, and a ZnS phosphor were used to determine the resolution loss introduced by the phosphor and the glass faceplate of the intensifier.

A closed-circuit television system will be used to study the limitation of the vidicon camera tube.

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