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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1966

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

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

September 1966

DATE ISSUED

October 6, 1966

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 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 conducted in the ORR poolside facility to support the development of the General Atomic fast gas-cooled reactor design. Postirradiation examination of the two fuel elements from the fourth capsule in this series (P4B-4) has revealed no evidence of impending failure. However, we found a small local indentation in the cladding of the prepressurized fuel-supported element in the vicinity of one of the cladding thermocouples, and the gamma scan of the "flexcan" fuel element showed an unusual peaking of fission product activity at locations corresponding to pellet interfaces. This capsule was irradiated for two ORR cycles; the maximum cladding temperature was nominally 760°C but reached 860°C briefly during a reactor excursion.

Irradiation of the fifth capsule in this series (04-P5) continued. After one ORR cycle, during which the capsule was subjected to 36 thermal cycles and two cold pressure cycles, a periodic leak test gave no indication of cladding failure. The capsule contains two fuel elements of the fuel-support type and is being irradiated at 18 kw/ft heat generation, 815°C maximum cladding temperature, and 1000 psia external pressure.

High-Burnup Coated-Particle Irradiation Test. — The F9 high-burnup coated-particle irradiation test assembly contained two batches of pyrolytic-carbon-coated fuel particles in separate fuel holders. In one batch the fuel was sol-gel-produced UO_2 microspheres, and in the other the fuel was UC_2. Irradiation extended over a one-year period at a temperature of 1100 to 1400°C and to burnups in excess of 50% of the initial metal atoms. A common sweep gas was sampled periodically to monitor fission-gas release. At about 10% burnup, the fission-gas release rate (expressed as R/B, the ratio of release rate to birth rate) increased by about two orders of magnitude from $\sim 10^{-6}$ to $\sim 10^{-4}$ and remained roughly constant thereafter.

During postirradiation examination we have attempted to determine whether one of the fuel batches was the sole source of the observed increase in fission-gas release and to identify the cause. The results to date are inconclusive. After the fuel particles were removed from the graphite fuel holders, the holders were gamma scanned. We found that the holder that had contained the UC_2 particles showed ten times as much residual fission product activity as did the holder that had contained UO_2 particles. However, when we examined both batches of particles (approximately 10,000 particles per batch) under the stereomicroscope, we were unable to locate any failed particles in either batch. Similarly, no failures could be identified in metallographic mounts of samples of particles from each batch, although we did observe a small number of damaged particles – five out of 170 UC_2 particles examined and one out of 85 UO_2 particles examined.

Properties of Pyrolytic-Carbon Coatings Deposited from Propane. – We recently completed a systematic study of the effects of deposition conditions on the properties of coatings derived from propane. The coatings were deposited on a 55-g charge of 460-μ-diam ThO_2 microspheres in a 1-in.-diam fluidized bed in which there was a stationary axial probe. We studied deposition temperatures between 1200 and 1900°C and varied the propane supply rate from 0.05 to 3.2 cm^3/min per square centimeter of particle surface (0.07 to 4.3 gram-atoms of carbon per minute per square meter).

Results show that the density, anisotropy, and crystallite size of the deposits have the same general relation to temperature and gas supply rate as with methane. However, deposition rates are much higher at all temperatures. Of particular interest is the coating applied at 1200°C at a propane supply rate of 3.2 $cm^3\ min^{-1}\ cm^{-2}$. Such deposits are completely random in orientation, have a density of about 2.0 g/cm^3, and are deposited at a rate of about 9 μ/min. To achieve comparable coatings from methane requires a deposition temperature in excess of 2000°C and a gas flow that limits the deposition rate to about 1 μ/min. We are currently evaluating the irradiation behavior of these high-density low-temperature coatings.

Effect of Pipe Bends on Wall Deposition of Radioactive Iodine. – The effect a pipe bend may have on pipe wall deposition in a flowing gas stream containing a trace amount of radioactive iodine was investigated for 45 and 90° pipe elbows. The depositing surface was maintained isothermal at 250°C, and the helium flow was in the turbulent flow regime. In both bends the peak deposition per unit area was greater by approximately a factor of 8 than the deposition in the straight pipe downstream of the bend.

FUEL-CYCLE STUDIES

Thoria-Urania Sol Preparation. – Laboratory studies continue on the process for making mixed sols of thorium and uranium oxides directly from a solution of thorium and uranium nitrates by a two-stage solvent extraction procedure. In this procedure, organic solutions of long-chain

aliphatic amines contact the aqueous nitrate solutions of thorium and uranium and extract the nitrate ions, causing the metal ions to hydrolyze. A digestion of the aqueous solution between two extraction steps is necessary to produce a degree of metal-ion hydrolysis suitable for good sol formation. Our most recent studies show that a minimum digestion period of about 8 min at 98 to 101°C is required to achieve the desired nitrate:metal mole ratio of about 0.2. Longer digestion periods have little effect.

Microsphere forming studies with sols prepared by this solvent extraction procedure are also continuing. Microspheres were prepared from a sol that was 1.1 M in thorium, 0.32 M in uranium, and 0.30 M in nitrate, and that contained a small amount (0.01 mole of carbon per liter) of carbon-containing material. The sol was injected into 2-ethyl-1-hexanol containing 0.5 vol % H_2O and surfactants. A variety of surfactants and a range of concentrations were evaluated. After drying and firing at 1150°C, most of the spheres had a porosity of less than 1% and a density that was more than 95% of theoretical. Resistances to crushing forces ranged from 940 to 2000 g for spheres ranging in size from 150 to 350 μ in diameter. Spheres with these values are more than adequate for use in HTGR fuels. An exception occurred when Ethomeen S-15 plus bis-2-ethoxyethyl phthalate was used as a surfactant. In this case, the resulting microspheres had a density that was only 92% of theoretical.

Thoria-urania sols are also being prepared by mixing sols of urania and thoria. An effort was made to determine the accuracy of this method for producing sols of a desired composition. Calibrated glassware was used for mixing the analyzed sols. Since uranium enrichments as well as thorium:uranium ratios were specified, it was necessary, first, to mix an analyzed urania sol of natural uranium with one of 93.18% ^{235}U enrichment, and then to mix the resultant urania sol with a thoria sol. The percentages of uranium enrichment and the thorium:uranium ratios in microspheres prepared from these sols are in excellent agreement with those desired, as may be seen from the table below.

Thorium:Uranium Ratio		Percentage Uranium Enrichment	
Desired	Analyzed	Desired	Analyzed
0.3333	0.3337	43.5	43.3
1.0000	1.005	65.3	65.04
1.857	1.886	93.18[a]	93.18

[a]Undiluted enriched urania sol was used in this preparation.

Microsphere Forming Studies. — Current microsphere forming development studies are directed toward improving the methods for making uniform sol droplets. These droplets are dispersed through nozzles into the alcohol contained in the sphere-forming columns, where they are set to gel.

In our engineering development work, sol injection assemblies containing several two-fluid nozzles are being used to study the problems associated with the high sol throughput rates necessary for large-scale production plants. The slight bending of nozzle parts during the dismantling and the cleaning of the assembly was found to cause nonuniform droplet sizes and unsatisfactory column operation. An assembly containing 11 two-fluid nozzles, which will be easier to align and clean, is being built. We are confident that this approach will eliminate the mechanical problems. Alignment problems notwithstanding, we are able, by using care in cleaning and aligning our present nozzles, to achieve completely satisfactory sphere-forming column operation. We have recently produced about 50 kg of 210- to 250-μ-diam ThO_2 spheres for use in coating studies; we have also produced 2-kg lots of ThO_2–4% UO_2 and ThO_2–9% UO_2 for use in HTGR particle evaluation studies by General Atomic. It was noted in these preparations, as in the past, that variable sphere-forming column behavior may be experienced in consecutive runs under conditions that are, presumably, unchanged.

Fueled-Graphite Fabrication Development: Fuel Particle Bonding. – One of the requirements for mockup fuel segments or elements to be tested in the Peach Bottom Reactor is that the fuel particles (pyrolytic-carbon-coated microspheres) must be bonded together in channels in the graphite matrix so they cannot spill freely into the reactor in the event of element failure. In our studies of bonding methods, we hope to achieve a strong bond by using a graphite-base material, with some excess carbon to provide for fission product adsorption. So far, we have not been very successful in our attempts to add the excess carbon and still maintain adequately high fuel loading in the channels.

Another problem we are attempting to solve is that of finding a method of mixing, with each other and with bonding material, the two or more different sizes of particles that will probably be used. So far, our efforts have not been successful; however, we hope to circumvent the problem by controlled feeding of the particles directly into the fuel channels.

Fueled-Graphite Fabrication Development: Particle Coating. – During the past month we have concentrated on fluidized-bed coater studies. We are trying to determine the cause of the variations in coating thickness when low-density (0.6 to 1.0 g/cm^3) coatings are applied by using acetylene. Variations, which are 8 to 12 μ for total coating thicknesses of 30 to 50 μ, appear to be larger for low-density than for high-density coatings. We believe that the variations are the result of (1) unequal times of contact for the particles and the coating gas and (2) inadequate temperature control in the bed. Temperature control problems stem from the exothermicity of the acetylene decomposition reaction, and from the short duration of the runs, which does not allow the bed to reach equilibrium. The addition of helium to the acetylene has been found to reduce coating thickness variations to about 5 to 6 μ, presumably by suppressing the rise in bed temperature.

We are also continuing the design of a prototype remote coater, in which propane or propylene will be used for preparing high-density coatings and acetylene will be used for low-density coatings. The design is

about 40% complete. An area of special concern is the disposal of unreacted hydrocarbon and hydrogen gases. Our approach is to burn the gases in a combustion chamber; disposal of the large amount of heat generated is the principal problem.

Fueled-Graphite Fabrication Development: Particle Inspection. – It will be necessary to take samples from the microsphere coater and from the various handling and classification systems on a routine basis. These samples will be transferred to an analytical glove box, where various inspections necessary for quality control, such as those of coating densities, size, and shape, will be made. The number of particles in a given weight of product will have to be counted for inventory and process control. The incoming sol-gel particles, the low-density coated particles, the duplex-coated particles from the furnace, and the product after shape and size classification will require this particle-number determination for a known weight of particles. An instrument that operates on the principle of light-scattering photometry appears to be capable of performing this task.

We are also attempting to develop an in-line process monitor for determining the average carbon-coating thickness on a small batch of coated ThO_2 particles. We have chosen a method based on x-ray attenuation. A calibration curve has been established for coating thicknesses in the range 13 to 172 μ on 230-μ-diam ThO_2 microspheres. Trial determinations of coating thickness on 12 batches of ThO_2 particles gave excellent results in some cases and very poor results in others. The error appears to result from the variations in coating thickness among particles in a batch, combined with the statistically small number of particles that were measured. Better control of coating thickness, combined with a larger representative sample, should make this method quite adequate.

Urania Sol Preparation by Solvent Extraction. – The preparation of aqueous urania sols by solvent extraction is being studied. In this technique, aqueous solutions containing U^{4+}, nitrate, and formate are contacted with an organic solution of a primary or secondary amine, which extracts the anions. As this extraction proceeds, progressive hydrolysis of the U^{4+} occurs, leading to sol formation.

Last month the formation of 350- to 500-μ-diam gel microspheres from a 1.5 M sol that had been prepared by solvent extraction was reported. These microspheres have now been dried by heating in argon at 110°C overnight and then at 180°C for 5 hr, followed by cooling in argon to room temperature. They were subsequently fired by heating in helium and then in hydrogen at 1150°C, and by cooling to 450°C in hydrogen and to room temperature in helium. The microspheres were subjected to microscopic examination and to tests designed to determine their resistance to crushing.

Three different surfactant combinations were used in the alcohol contained in the sphere-forming column to prevent the spheres from clustering and sticking to the column wall. All were fairly effective; with two of the combinations, however, some of the larger microspheres (larger than 375 μ in diameter) were cracked after firing. These results are preliminary and should not be interpreted as a comparison of

the different surfactants. At present, the size of the microspheres continues to be the single, most important influence on the extent of cracking; spheres over 400 μ in diameter tend to crack excessively, while those under about 300 μ appear to be relatively crack free.

Thorium-Uranium Recycle Facility. – The Thorium-Uranium Recycle Facility (TURF) is being built to provide adequate shielded space for development of equipment for remote processing and fabrication of Th-^{233}U fuels. Prototype HTGR fuel segments for use in irradiation tests will also be made in this facility. 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.

Construction is approximately 84% complete. Recent efforts have been devoted, primarily, to the installation of mechanical and electrical equipment and to the fireproofing of various structural steel support members. The stainless steel cell liners have been completed up to the cell ceilings. The majority of the seams in cell A, the glove maintenance room, and the air-lock opening into the cell have been completed, and the stainless steel floor is being installed. All equipment furnished under ORNL participation is progressing satisfactorily; shield door 1 has been delivered to the contractor, and doors 2 and 3 have been erected and tested.

Equipment for preparing urania sol by precipitation-peptization in kilogram batches has been installed and is being tested. Laboratory-scale equipment (for preparing 50- to 100-g batches) for use in studies of sol preparation by solvent extraction has been built and is essentially installed and ready for testing. Larger-scale equipment (for preparing kilogram batches) is being built for use in engineering studies of sol preparation by solvent extraction; it is about 60% complete. Microsphere drying equipment for use with batches of up to 20 kg of gel spheres has been designed, and its fabrication is about 80% complete.

We are continuing the detailed conceptual design of the processing equipment that will be installed in the TURF to demonstrate the technology for fabrication of an HTGR fuel element. Such fuels are composed of pyrocarbon-coated microspheres contained in a graphite matrix with holes for passage of the coolant gas. This month we have concentrated on a prototype remote coating system and on a fuel channel filling and bonding mechanism.

Formation of Carbides from Sol-Gel Microspheres. – Further attempts were made to prepare UC microspheres from sol-gel UO_2 microspheres by first converting the UO_2 to UC_2, then heating the UC_2 microspheres in hydrogen to form UC by removal of carbon. The conversion of UO_2 to UC_2 was accomplished by heating with lampblack at 2000°C for 5 hr.

Previous attempts to form UC by hydrogen treatment of UC_2 at 850°C for 24 hr were unsuccessful. Results from heating the UC_2 microspheres in hydrogen at 1900°C for 1 to 2 hr are encouraging. Metallographic examination showed the material treated under these conditions to be a UC matrix containing thin platelets of UC_2. Apparently the UC_2 concentration was low, since a Debye-Scherrer x-ray diffraction pattern showed that only UC was present.

Our results appear to disagree with the findings of Harder, Read, and Sowden (AERE-R4877), who reported 850°C to be the optimum temperature for reduction of hyperstoichiometric UC by hydrogen treatment, with very little reduction being obtained above 1100°C. To determine whether our results may have been influenced more by the gettering of carbon by the tungsten crucible (used as a container) than by hydrogen reduction, we heated UC_2 microspheres in a tungsten crucible in an argon atmosphere (otherwise, the conditions were identical) for comparison. Although some UC was formed under these conditions, conversion was not as complete or as uniform as that obtained by hydrogen treatment. To study the role of tungsten in the reduction, we heated UC_2 microspheres covered with tungsten powder in a tungsten crucible in a hydrogen atmosphere. The conversion to UC under these conditions was much inferior to that obtained by simply heating in a tungsten crucible in hydrogen, and unidentified phases not previously noted appeared in the microstructure. These results suggest that tungsten may contribute to the reduction but that the hydrogen treatment at high temperature is also important.

Thorium dicarbide microspheres can be prepared from ThO_2-C sols. This method leads to a low-density (90% of theoretical) product; and in the relatively few experiments that have been performed so far, small amounts of unconverted ThO_2 have been present in the product. Present studies are designed to determine what control can be achieved over density and residual oxide (or free-carbon) content. In addition to being converted to ThC_2, ThO_2-C microspheres are precursors in some methods for nitride and sulfide syntheses.

During the past month, a new firing technique was investigated for converting ThO_2-C gels to ThC_2. Results are very encouraging, in that higher-density ThC_2 spheres (90% of theoretical density) were obtained. The principal difference between the present approach and those used earlier is that the conversion reaction was carried out under argon at 1 atm instead of in vacuum. Vacuum was applied only near the end of the run. Temperatures from 1850 to 2050°C were studied.

The basic properties of ThO_2-C sols were investigated further. Measurements show that the viscosity of ThO_2-C sols increases very rapidly upon carbon addition after the ThO_2 surface sites are occupied by carbon, and that a quite well-defined "stoichiometry" exists between the ThO_2 surface area and the carbon crystallite concentration.

<u>^{233}U Storage and Distribution</u>. — ORNL serves as a national storage and distribution point for ^{233}U. The storage, dissolution, and purification equipment in Building 3019, coupled with the oxide and metal production facility at the Y-12 Plant, provides the necessary flexibility to reprocess ^{233}U as aged solution, metal, or oxide scrap, and to furnish ^{233}U in any form.

During the past month, seven shipments (74.6 kg of ^{233}U) were received, and 13 shipments (67.1 kg of ^{233}U) were made. All the material was recently purified and contained less than 6 ppm of ^{232}U. Most of the transfers involved Nuclear Fuels Services, Erwin, Tennessee, who is the subcontractor for fuel fabrication for the Light Water Breeder Program, which is under the direction of Bettis Atomic Power Laboratory.

Approximately 33 kg of ^{233}U was transferred to the Neutron Physics Division for criticality experiments. Smaller shipments were made to Argonne National Laboratory, Goodyear Atomic Corporation, Virginia Polytechnic Institute, and to the country of Israel.

In the large solvent extraction facility, 10 kg of ^{233}U containing 5 ppm of ^{232}U was purified. This product will be transferred to the Y-12 Plant in 500-g batches for conversion to dioxide for Battelle Memorial Institute, Pacific Northwest Laboratory, for use in the High Temperature Lattice Test Reactor.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — The program of graphite-sample replacement, maintenance, and minor modifications continued. The work is on schedule except for delivery of the main blowers, which may delay power operation until the middle of October.

Salt plugs which had formed in the helium lines when the fuel pump was flooded were thawed by attaching external heaters, but salt that melted in the sampler line refroze lower in the pump bowl. We plan to reheat this line externally when the fuel loop is hot and flush salt is circulating.

A new assembly of graphite and Hastelloy N specimens was fabricated and installed in the core.

The No. 3 control rod was removed, and both the rod and rod drive were replaced. Slight modification of the lower end of the rod eliminated the binding, which was apparently due to a projection in the rod thimble. The slow drop times were apparently caused by a bow in the cooling-air line that caused a drag on the rod.

The west reactor-cell space cooler was removed and found to have considerable fission product contamination. After decontamination, radiation was still 200 to 700 mr/hr. The leaking brazed joints were repaired, and the cooler was reinstalled.

Toward the end of operation, the filter trap in the fuel off-gas line showed signs of plugging. This became worse during shutdown, and we replaced it. The inlet to the auxiliary charcoal bed also became plugged during shutdown. Application of external heat relieved this plugging. The filter in the coolant-pump off-gas line became plugged and was replaced.

Two samples of off-gas, isolated on June 23 at 7.2 Mw, were removed and analyzed. Xenon isotopic ratios indicate an ^{135}Xe poisoning effect of 0.35% $\delta k/k$. This is good agreement with conclusions from the reactivity balance.

The radiator doors were modified to decrease the warping and provide better seals. Additional supports were installed on the radiator enclosure, and other repairs were made. Temperature distribution and visual inspection with the radiator heated indicated that the modifications had improved the seals.

Delivery of the replacement units for the main blowers was delayed when one unit was destroyed due to a failure of the manufacturer's test

equipment. Also, some delay has been encountered because of welding and casting defects in the new units.

To stop the leaks in the heat exchanger between the cooling-tower water and the treated water, 17 of the 360 tubes were plugged. We diluted out the sodium contamination in the treated-water system and added new treatment chemicals. The treated-water system was revised to allow deaeration of all of the water from the thermal shield.

The component coolant pump flows were increased from 600 to 750 scfm to get better mixing of the gas in the reactor and drain tank cells. The electrical penetration to the motors was replaced, modifications were made to provide more cooling to the drive belts, and a strainer was installed in the discharge piping. Equipment was installed to permit draining any water which accumulates during operation.

Twenty-six of the main electrical breakers were removed, recalibrated, and repaired. Modifications were made to the control circuits to enable transferring salt through the fill lines and to allow emergency bypassing of all control circuits, if needed, to start the coolant pump. A method of measuring the control rod drive temperature was installed.

The salt-circulating systems are being heated and equipment is being checked out, in preparation for startup.

Metallurgy. — As part of the materials surveillance program, reactor core specimens of graphite and Hastelloy N were removed from the MSRE after 7800 Mwhr of operation. The objective was to remove one stringer (one-third of the assembly) at this time and return the others to the reactor. However, approximately one-seventh of the assembly in a zone just above the reactor midplane was severely damaged. Graphite specimens were buckled and broken, and the tensile specimen rods of Hastelloy N were bent. Apparently, the assembly had been locked together by frozen salt during the reactor cooldown, and the large difference in the coefficients of thermal expansion of the metal and graphite created high stresses. The damage in this zone occurred to all three Hastelloy N and graphite stringers, requiring that all three be replaced. However, the graphite and Hastelloy N from other portions of the array were suitable for the intended mechanical-properties evaluation tests. The Reactor Chemistry Division was supplied with graphite samples for fission product analyses from the top, middle, and bottom of the assembly.

The visual appearance of the metal and graphite was good. The external surfaces of both were virtually free of salt. The metal was a dull gray, and the graphite appeared unchanged by the exposure. The graphite and Hastelloy N specimens are being examined.

A slightly modified replacement for the damaged reactor core specimens has been delivered to the MSRE for installation. The modifications were made to reduce mechanical stresses induced in the assembly by reactor heating and cooling cycles.

The Hastelloy N tensile specimen rods in the new set are made from reactor vessel wall and head materials plus two modified alloys of Hastelloy N, one containing 0.52 wt % Ti and the other 0.43 wt % Zr. These additions are for the purpose of improving the radiation stability of the alloy.

Chemical Development. – Further exposures of metal specimens to the gas and fuel phases in the MSRE pump bowl confirmed previously reported results on the volatilization and plating behavior of the noble-metal fission products. The small samples of silver, Hastelloy N, and stainless steel were found to be coated with several times the quantities of ^{99}Mo, ^{103}Ru, and ^{132}Te that were found in a gram of fuel salt. Correspondingly, the amounts of these isotopes remaining in the fuel salt were only 60, 30, and 30%, respectively, of the total amounts calculated to be produced by fission. The balance presumably either volatilized or deposited on the Hastelloy N and in the graphite surfaces of the MSRE.

Further information on fission product behavior in the MSRE was obtained from examinations of the long-term surveillance specimens of graphite and Hastelloy N which were removed from a central position in the MSRE core after 7800 Mwhr of operation. Visual, x-radiographic, and metallographic examinations of the graphite specimens showed no radiation damage to the graphite, no films of metal or carbide on the graphite surface, and no penetration of fuel salt into the graphite pores. Autoradiographs showed that the radioactivity was confined principally to a very thin surface layer, with diffuse irregular penetrations of small amounts of activity to interior regions. X-ray examination of the graphite surface yielded only graphite diffraction patterns.

Successive layers averaging about 10 mils in thickness were milled from the surfaces of three graphite surveillance specimens and analyzed radiochemically for a number of fission products. The noble-metal isotopes ^{99}Mo, ^{103}Ru, ^{132}Te, and ^{95}Nb comprised most of the activity in the first milled layer. The concentrations of these isotopes dropped by a factor of about 100 in the second layer, and continued to decrease more slowly in successive layers. The fission products with xenon or krypton precursors, such as ^{140}Ba, ^{89}Sr, and ^{141}Ce, exhibited much flatter concentration profiles, particularly for the isotopes with longer-lived gaseous precursors. Isotopes with stable nonvolatile fluorides, such as ^{95}Zr, were present at very low concentrations in the graphite.

The deposition of noble-metal fission products which have moderate neutron cross sections on the graphite core is of some concern from the standpoint of neutron economy. The present results indicate that 12 to 18% of the total ^{99}Mo produced, 12 to 14% of the ^{132}Te, 6 to 12% of the ^{103}Ru, and 12 to 60% of the ^{95}Nb were deposited on the 2×10^6 cm^2 of graphite surface in the MSRE. Analyses of Hastelloy samples adjacent to the analyzed graphite specimens in the surveillance assembly indicated that more of the noble-metal fission products deposited on metal than on graphite. The observed ratios in terms of dis min^{-1} cm^{-2} on metal compared with graphite were 5 to 6 for ^{99}Mo, 10 to 15 for ^{132}Te, and 3 to 5 for ^{103}Ru.

A sample of MSRE cover gas was isolated on June 23, 1966, during 7.2-Mw operation. The sample was recently concentrated by a factor of 75 using a molecular-sieve adsorption technique and was analyzed by mass spectrometer. The results on the isotopic ratio of ^{136}Xe to ^{134}Xe indicated that only 7.9% of the ^{135}Xe produced in the MSRE was burned to ^{136}Xe before it reached the off-gas system. This low burnup is in agreement with indications from reactivity analysis.

Radiation Effects. — Enriched uranium was added as $^{7}LiF-UF_4$ to the molten-salt loop on July 27, 1966, after 1100 hr of operation in the ORR with unfueled solvent salt. Based on the salt inventory of 179.9 g, the concentration of uranium (93% enriched) in the fuel salt was 1.36 mole % (12.55 g of ^{235}U). For the total fuel inventory of 71.6 cm^3 at 600°C, the average fission power density was 81 w/cm^3 with the loop in the fully inserted position of maximum neutron flux. The corresponding average power density of the fuel in the core of MSRE-grade graphite was 108 w/cm^3.

Successful operation of the major heating, cooling, temperature control, and sampling systems was demonstrated. Operation with molten-salt fuel containing enriched uranium continued until August 8, 1966, when the experiment was terminated due to detection of radioactivity in the off-gas from the loop secondary containment, indicating a leak in the primary loop assembly. At that time the $^{7}LiF-BeF_4-ZrF_4-UF_4$ (65.16-28.57-4.90-1.36 mole %) fuel had undergone 1.2×10^{18} fissions/cm^3 (0.27% ^{235}U burnup).

The experiment was removed from the ORR and transferred to hot cells for disassembly and examination, which is now in progress.

A second in-pile molten-salt convection loop is being constructed.

MSR Processing. — We are developing equipment for the continuous removal of UF_4 from the fuel stream of an MSBR by contacting the salt with F_2 in a salt-phase-continuous system. Study of the countercurrent contact of molten salt with F_2 has continued in a 1-in.-diam nickel fluorinator 72 in. long. Three additional experiments were carried out at 600°C using an $NaF-LiF-ZrF_4$ mixture containing 0.37 to 1.16 wt % uranium as UF_4 and having a melting point of ~475°C. Salt feed rates of 9.8 to 30 cm^3/min and F_2 rates of 235 to 335 cm^3/min (STP) have been used with molten salt depths of ~48 in. Uranium removal during one pass through the fluorinator varied from 99.36 to 99.89% as determined from salt samples.

During the best run to date (CF-12), a molten-salt feed rate of 10.2 cm^3/min and an F_2 feed rate of 235 cm^3/min (STP) were maintained for a 2.5-hr period. At steady state, 99.89% of the uranium was removed by the fluorinator, as determined from inlet and exit UF_4 concentrations in the salt. Later in the run, a molten-salt feed rate of 22.5 cm^3/min and an F_2 feed rate of 270 cm^3/min (STP) were maintained for a 2.25-hr period. With these conditions the uranium removal at steady state was 99.62%. The uranium concentration in the feed salt was 1.16 wt %, and the exit uranium concentration was determined from salt samples taken at 15-min intervals. The equipment operated smoothly during the run, salt and gas feed rates were constant, and the system was operated at steady state for 2 hr at each of the above conditions.

Continuous fluorination of the fuel stream of an MSBR with equipment of this type appears feasible and will be studied further.

Analysis of Radioactive MSRE Fuels. — Four samples of the MSRE fuel, taken at full-power level, have been analyzed for oxide. The first two samples, which were analyzed as they were received at the High-Level Radiation Laboratory, contained 67 and 66 ppm oxide. The third sample was removed from the transport container and placed in the storage cell for 24 hr prior to analysis. No significant effect due to fluorine

evolution during standby was noticed, since the subsequent analysis of the sample showed the presence of 59 ppm oxide. The last sample, which was analyzed in the normal manner, contained 56 ppm oxide.

A special technique for the regeneration of the Beckman water electrolysis cells was developed to provide an accurate and dependable replacement component for the oxide apparatus in the hot cell. Previous studies had indicated that the standard regeneration technique resulted in a negative bias of about 5% in the oxide analyses, which was due to gas flow interruptions in the capillary cell. The new technique provides a desiccant coating sufficient to absorb the water in the gas stream at low flow rates and gives a minimal amount of flow interruptions during electrolysis. The cells which have been successfully regenerated have yielded oxide recoveries of 99.6 ± 1.3% from the hydrogenation of standard SnO_2 samples. Cells regenerated by the new method will also be used for the development of the in-line oxide instrumentation.

REACTOR FUELS AND MATERIALS

Zirconium Metallurgy. — The anisotropy in the mechanical properties of zirconium alloys is important for maximizing in-service performance as well as selection of optimum fabrication processes. However, three yield strengths in tension, three yield strengths in compression, and twelve strain anisotropy constants are required to characterize the three-dimensional anisotropy of mechanical properties in any material. To reduce the required mechanical testing, we developed an empirical correlation that relates the anisotropies of yield strengths and strain constants in tension and compression in Zircaloy-2. With this empirical correlation, five yield strengths and ten strain anisotropy constants can be calculated to within 5% accuracy from the experimental determination, on one specimen, of one yield strength and two strain anisotropy constants. Thus, only one tensile or compression test need be made for any Zircaloy-2 material, whatever the preferred orientation existing in it, to characterize its three-dimensional anisotropy of stress and strain properties for purposes of design, quality control in the mill, or purchase specifications.

Mechanical Properties Research. — For our continuing study of the irradiation embrittlement of structural metals, an apparatus has been designed and built to inject helium into 0.020-in.-thick type 304 stainless steel samples at the ORIC. A uniform distribution of helium in the samples is obtained by driving the samples at a constant speed past the cyclotron beam port, where the beam of alpha particles, moderated by a graphite wedge of appropriate dimensions, deposits a planar trace of helium in the samples. By using an 80-Mev beam and motors of different speeds to drive the samples, we obtained sets of samples containing 0.83×10^{-8}, 1.4×10^{-8}, and 8.3×10^{-8} atom fraction of helium. The dose rate was approximately 10^{-7} atom fraction of helium introduced per hour.

The decreases in ductility sustained by these samples in elevated-temperature tensile tests were similar to the decreases in ductility of

type 304 stainless steel irradiated in the ORR to the same helium concentration, where the helium is produced by (n,α) reactions. By electron microscopy, we are studying the properties of helium bubbles in the cyclotron-irradiated material.

Further cyclotron irradiation experiments are under way to determine whether the properties of stainless steel containing cyclotron-injected helium are similar in all respects to those of neutron-irradiated stainless steels at elevated temperatures. If this proves to be the case, irradiation in the ORIC facility may provide a rapid screening test for reactor materials and a useful tool for studying the more fundamental aspects of helium-induced brittle failures. More refined experimental techniques may allow the injection of 10 ppm or more of helium into materials in times relatively short compared with the reactor irradiation times required to produce the same amount of helium.

Fuel Element Fabrication. — Irradiation specimens of both solid and powder UO_2 produced by direct hydrolysis reduction of UF_6 are being prepared. Since the apparatus for deposition had been built to demonstrate chemical feasibility and not necessarily to produce material, we had to increase the efficiency of material recovery. In the case of powders, previously 40% of the feed material had been recovered for further experiments. Without significant changes to the apparatus, we increased UF_6 feed rate by 50% and UO_2 powder recovery to above 75%. Since sufficient material for preparation of pressed and sintered pellets may be produced at this efficiency, we have proceeded with the processing of the enriched material.

Irradiation specimens of solid uranium dioxide are also being prepared as coatings of highly enriched material on thoria pellets. If a fertile pellet is coated with a thin layer of fissile material that may be readily removed, reprocessing should be simplified. Uniform coatings of depleted UO_2 up to 0.020 in. thick have been deposited. On metallographic examination, good bonding was found between the UO_2 and the thoria; however, cracks were found originating in the thoria and penetrating the UO_2. On further examination, areas of variable density were found in the thoria pellets, which could have contributed to the cracking. We have ordered another batch of thoria pellets for evaluation prior to coating with highly enriched UO_2.

SAP Materials Development. — Sintered aluminum products material is the prime candidate for construction material for the fuel rods and pressure tubes of the Heavy-Water Organic-Cooled Reactor. Previously available SAP materials exhibit erratic behavior, in the forms of poor process yield, structural inhomogeneity, gas fissuring and blistering, anisotropic properties, premature fracture, and low creep ductility. Since most of the erratic behavior is either process or composition related, a thorough study is being conducted to determine the material and processing parameters necessary to produce a desired end product that is consistent.

Several powder-metallurgy routes are being investigated. In general, material characterization, dispersion preparation, powder consolidation, and primary fabrication are the operations being studied. Currently we have characterized our powder feed material and have selected ball milling

as the only dispersion preparation method that is now feasible in industry. Powder can be consolidated by either cold or hot pressing; we are now evaluating both. These materials produced on a laboratory scale by either method are stronger than the available commercial material having the same oxide content. The consistency of our product relative to commercial material has yet to be properly evaluated.

POWER REACTOR FUEL PROCESSING

Aqueous Process Development: General. — Development is continuing on mechanical, nonaqueous, and aqueous steps for head-end processes designed to convert power reactor fuels into aqueous solutions suitable for fuel decontamination and recovery by solvent extraction. 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 and grind-leach processes, also for graphite-base HTGR fuel; and (3) solvent extraction studies with grind-leach fuel solutions.

Aqueous Process Development: Burn-Leach Process. — Hot-cell evaluation of the burn-leach process for graphite-base fuels was continued. In this process, HTGR fuel [pyrolytic-carbon-coated $(Th,U)C_2$ particles dispersed in a graphite matrix] is 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 uranium and thorium can be recovered by conventional solvent extraction.

In the most recent hot-cell experiments, 90- to 180-g batches of coarsely crushed (−4 mesh) Peach Bottom GAIL III-B fuel, which had been irradiated to an average burnup of 40,000 Mwd/metric ton and cooled for two years, were successfully burned in 60 to 120 min at 750°C, using a nitrogen-air-oxygen mixture to control the burning rate. Control of burning was more difficult to maintain at 750°C with this particular fuel than with unirradiated prototype HTGR fuel or GAIL III-A fuel irradiated to 8000 Mwd/metric ton. Other experiments are planned to determine whether the highly irradiated GAIL III-B fuel has a lower ignition temperature and can be burned efficiently at temperatures below 750°C.

Of major concern are the degree of volatilization and entrainment of fission product oxides during burning and their satisfactory removal from the burner off-gas, which is passed, first, through a 20-μ, carbon-precoated micrometallic filter inside the top of the burner, and then through main and secondary absolute filter packs. The carbon-precoated micrometallic filter performed as a nearly absolute filter, yielding excellent separation factors for the principal isotopes released. Typical separation factors achieved are: gross fission products (gamma), 7.5×10^5; ^{106}Ru, 1.6×10^6; ^{125}Sb, 1.5×10^4; ^{137}Cs, 9.5×10^5; and ^{144}Ce, 1.1×10^6. It was noted, however, that if the carbon coating is dislodged and burned, as was done intentionally in one run, the separation factors are decreased by about a factor of 20. Final removal of submicron-size fission product particulates from the burner off-gas

downstream from the carbon-precoated filter was achieved by (1) reducing the superficial gas velocity from 20 fpm to 5 fpm, and (2) passing the off-gas through an absolute filter packed with 12 layers of type 25-FG Filterdown, a fiber-glass filter medium; only background activity was detected on a secondary absolute filter used to monitor the twice-filtered off-gas.

Aqueous Process Development: Grind-Leach Process. – Fine grinding followed by acid leaching is also being evaluated as a method for processing graphite-base HTGR fuels that contain coated fuel particles. Engineering- and laboratory-scale tests and hot-cell experiments are being made with various carbon-coated ThC_2-UC_2 and ThO_2 fuel particles in a graphite matrix. The best method found for grinding HTGR fuels is rough crushing to –6 mesh in a hammer mill followed by roll-crushing to reduce the particle size to –140 mesh. Grinding to this size ensures rupture of nearly all the particle coatings. In continuing engineering tests, kilogram-scale leaching and washing experiments with unirradiated Peach Bottom reactor fuel that had been crushed in a double-roll crusher set at a 2-mil spacing confirmed the 99.85% thorium and uranium recoveries obtained earlier in runs with 50-g batches. However, high fuel recoveries from the larger batches required concentrated HNO_3 instead of water for washing the graphite residue. Thorough washing of thorium and uranium from the 2-ft-deep beds of graphite residue required 5 to 6 hr and about 8 liters of wash per kilogram of residue. The acid percolation rate was about 15 gal hr^{-1} ft^{-2} at a pressure gradient of 10 psi per foot of bed depth. At the conclusion of these runs, the 12- by 4-in. double-roll crusher was disassembled for inspection and minor modifications. Total cumulative throughput of the machine, at roll spacings of 0.002 to 0.004 in., was about 35 kg of HTGR fuel (mostly Peach Bottom). Roll wear amounted to less than 0.0005 in.; the surface finish appeared to be unchanged (about 12 to 15 μin.).

In hot-cell studies, eight experiments with Peach Bottom GAIL III-B compact No. 7, which had been irradiated to a burnup of 36,400 Mwd/metric ton and cooled for two years, were completed. These experiments were designed to determine how completely UC_2, ThC_2, and fission product carbides can be leached and washed from this fuel. The fuel compact was first crushed to –4 mesh and then pulverized in a modified Waring Blendor. One batch of the crushed fuel was reduced in size to –100 mesh and another to –140 mesh; each batch was then sieved to make sure that all the coated particles were broken to expose the $(Th,U)C_2$ fuel kernels. Four 10-g samples of each fraction were then leached two to three times (up to 9 hr, total) with refluxing 13 M HNO_3 or 13 M HNO_3–0.05 M HF and washed five times with fresh leachant (or water) at 25°C; the graphite residue was dried with acetone.

In spite of extensive leaching and washing, all graphite residues retained excessive amounts of fuel (average of 1.14% uranium and 2.02% thorium) in a form that appeared inert to the leachant, as compared with losses of 0.02% for unirradiated fuel. Analysis of the washes indicates that the high losses are the result of incomplete leaching rather than inefficient washing. About 10% of the total fission product gamma emitters also were retained by the graphite residue, presumably because

of sorption on the large available surface area and because of limited dissolution by the leachant. The major contaminants are ^{106}Ru and the rare earths. Results obtained with other coated-particle fuels suggest that the nonleachable loss of heavy metals may be the result of fuel migration from the $(Th,U)C_2$ particle into the pyrolytic carbon coating and the graphite matrix, possibly by fission recoil. Carbides of uranium, thorium, and fission products, if bound up in the crystal structure of pyrolytic carbon and graphite, would be exceedingly difficult to leach.

Laboratory-scale leaching experiments were made on unirradiated UHTREX fuel that had been roll-crushed to −100 mesh. The UHTREX fuel contained about 10% uranium, in the form of 147- to 208-μ-diam triplex-coated UC_2 particles. The three particle coatings were as follows: (1) a 27-μ-thick inner buffer layer of porous carbon; (2) a 35-μ-thick intermediate layer of isotropic pyrocarbon; and (3) a 40-μ-thick outer layer of granular pyrocarbon. Samples of the roll-crushed fuel were leached for 5 hr with sufficient boiling HNO_3 to produce solutions 0.2 M in uranium (assuming that all the uranium dissolved). The residues were washed with three volumes of water. Uranium losses to the residue decreased from about 29 to 0.9% as the acid concentration was increased from 2 to 13 M. These preliminary results suggest that nearly quantitative recovery of uranium from unirradiated fuel can be achieved in two leaches with concentrated HNO_3.

Preliminary laboratory leaching studies were also made on two batches of unirradiated carbon-coated sol-gel ThO_2 microspheres, crushed to −100 mesh to ensure rupture of all particle coatings. One (batch OR-520) was composed of 275-μ-diam ThO_2 kernels coated with a single 45-μ-thick layer of dense pyrocarbon; the carbon content of these particles was about 23%. The other (batch OR-521) contained 275-μ-diam ThO_2 kernels with a duplex coating consisting of a 40-μ-thick inner layer of porous carbon and a 40-μ-thick outer layer of pyrocarbon; the carbon content of these particles was about 32%. Samples of each batch of crushed particles were leached for 5 hr with sufficient boiling 13 M HNO_3–0.05 M HF to produce a solution about 0.5 M in thorium (assuming that all the ThO_2 dissolved). In both cases, leaching of the ThO_2 was practically quantitative; the thorium losses to the carbon residues were less than 0.01%. The solution resulting from the leaching of batch OR-520 (single carbon coating) was faint amber in color and contained only a trace of soluble organic compounds (0.1% of the original carbon). The solution obtained by leaching batch OR-521 (duplex coating) was red-brown in color, but analysis showed that only 0.3% of the original carbon was present. However, the carbon concentration in the solution (about 0.2 mg/ml) was four times that in the solution obtained by leaching batch OR-520. On the basis of these very preliminary experiments, one can expect to find potentially troublesome quantities of soluble organic compounds in the acid leachate from fuels containing oxide particles that have a porous carbon layer in the coating.

Aqueous Process Development: Solvent Extraction Studies. – Solvent extraction studies were made on raw solutions obtained from the grind-leach processing of graphite fuels that contained $(Th,U)C_2$ particles.

This work was done to determine whether it is possible to develop a process that does not require the destruction of soluble organic species with strong oxidants and the separation of insoluble fission products prior to extraction. Previous attempts to recover the thorium and uranium from an untreated fuel solution in a conventional Thorex process, which normally uses acid-deficient feed, resulted in the formation of heavy emulsions.

In the new process the aqueous feed was made 2 to 5 M in HNO_3 and was fed directly to a column using 30% tributyl phosphate in dodecane. The corresponding thorium-plus-uranium concentration in the feed was approximately 0.05 to 0.1 M. No emulsion formed, and the product was free of soluble organic materials. The acid in the diluted grind-leach solution decreases the solubility of the thorium nitrate—TBP complex in aliphatic diluents. With normal paraffins, the solubility decreases with chain length. The solubility is slightly increased in highly branched solvents.

Fluidized-Bed Volatility Pilot Plant (FBVPP). — The FBVPP will be installed in cells 2 and 3 of Building 3019 to study the processing of spent UO_2 power reactor fuel clad in either Zircaloy or stainless steel. Equipment fabrication and cell preparation progressed during the period, but design was hindered by flowsheet uncertainties.

Thus far, development of the process flowsheet and design and fabrication of equipment have been done only for the head end of the process, that is, the primary fluidized-bed reactor, the pyrohydrolyzer, the primary trapping systems, the gas supply systems, and mechanical equipment for handling waste solids from the two main reactors. In the original ANL reference flowsheet, fluorine was to be used for volatilizing both UF_6 and PuF_6 from the primary reactor. Recently, a formal proposal was made by ANL, and concurred in by ORNL and ORGDP, to change to the use of a bromine fluoride for forming the volatile UF_6 without the evolution of the plutonium. The latter would be volatilized from the bed, as PuF_6, after treatment with fluorine in a second step. Such a procedure results in the separation of uranium from plutonium very early in the process, eliminating problems of plutonium deposition in much of the rest of the system. Agreement to change to the BrF_5-fluorine flowsheet was reached during a meeting at AEC Headquarters, Germantown, Maryland, on September 22, 1966.

The two main alternatives for uranium purification are: (1) distillation to separate UF_6 from BrF_5 and volatile fission product fluorides; and (2) use of NaF at 400°C for removing most of the ^{106}Ru, ^{95}Nb, ^{95}Zr, and ^{125}Sb, plus a stirred fluidized-bed of NaF powder followed by a fixed bed of NaF pellets. Cold traps and NaF and MgF_2 sorbents will be required with either alternative. A decision to use distillation was also made at the meeting mentioned above.

More laboratory work is required before the PuF_6 purification method can be made definite. Present plans call for deciding upon a method in February 1967.

Additional equipment items completed are the primary reactor, the reactor off-gas cooler, two NaF traps, and the NaF trap outlet filter. Other completion percentages are: pyrohydrolyzer and cold traps, 95; primary filter, 80.

Preparation of cell 3 was completed. Three floor sumps were drilled, and the trap system for the floor drains was revised. Valves were installed on the chemical waste drains to prevent the backup of contaminated wastes into the cell. Installation of shield support curbing was completed.

Construction was begun by the cost-plus-fixed-fee contractor. Shield support curbs were covered with metal plates, which were welded to the curbs, and guides were installed for the first-level (lower-level) shielding. The scrubber tank (FB-135) was set in position on the first level. Second-level floor supports are being installed.

Preparation of cell 2 was started. Valves for the chemical waste drains are being installed. New stainless steel floor drains from the penthouse and sample gallery were installed. Equipment and piping removal is 90% complete.

Fluoride Volatility Processing: Treatment of Zircaloy-Clad Fuel. — A method is needed for obtaining representative samples of solids existing in the fluidized-bed reactor at different stages of processing. These solids should be sampled twice: after the decladding and oxidizing step to determine the uranium:plutonium:fission product ratio, and after the fluorination step to detect the presence of uranium and plutonium residue large enough to constitute unacceptable losses (e.g., greater than 1% of the plutonium originally charged).

A recirculating fluidized-bed sampler was tested that utilizes a gas jet to continuously pull a small side stream from the fluidized bed near the bottom of the reactor and to lift it to the top of the reactor, where it is returned to the system. In tests with this equipment, where the sample exit pipe sloped downward at 45° for a distance of 6 ft to the air jet (0.093-in.-diam throat), the recirculation rate of solids increased with increasing velocity in the sample loop, becoming essentially constant (about 1200 g/min) at gas velocities above 10 fps. When the sample exit line was sloped upward at 45°, the recirculation rate of solids reached a maximum (about 1000 g/min) at a loop velocity of 15 fps. Samples were obtained by isolating a section of the sample loop and collecting the trapped solids. Screen analyses of samples taken at superficial bed fluidizing velocities of 0.5, 0.75, 1.00, and 1.25 fps showed rather good agreement with the alumina charged. Alcoa T-60 alumina, nominally 48 mesh (90% −48 +120 mesh, about 10% −120 mesh, and less than 1/2% −325 mesh), was used in the test.

Fluoride Volatility Processing: Use of BrF_3 to Remove U_3O_8 from Lines and Filters. — Photographic studies have shown that most of the U_3O_8 particles formed by oxidizing UO_2 in the fluidized bed are much less than 10 μ in diameter. This leads to the conclusion that some U_3O_8 will pass through the proposed 10-μ filters and will deposit in the process gas lines. In addition, some U_3O_8 always deposits in the disengaging section and on the gas filters. In the 0.9-in.-ID fluidized-bed reactor with 10-μ filters, approximately 0.9% of the uranium charge deposited in lines downstream from the filters; about 3% deposited in the filters. This is about ten times the amount of U_3O_8 required to reduce the PuF_6 expected to be present. A promising method for removing the excess U_3O_8 involves contact with bromine fluorides.

Small-scale tests have shown that BrF_3 will rapidly volatilize U_3O_8 at temperatures of 100°C or greater, while BrF_5 requires about 300°C for rapid reaction. In the small fluidized bed, BrF_3 was found to leave no uranium residue when the filters and disengaging section were heated to 160°C. Since heating the lines and filters to 300°C is not desirable, we recommend that, following the BrF_5 treatment to volatilize the bulk of the uranium, a BrF_3 treatment be used as a cleanup step.

Fluoride Volatility Processing: Disposal of Bromine Fluorides and Fluorine. — An aqueous system that appears to be excellent for disposing of both bromine fluorides and fluorine has been developed. The method consists in contacting the gases with a 1 to 10 N KOH solution containing 20 to 30 g of KI per liter, which reacts much more rapidly with fluorine, and is also more effective for BrF_5, than a solution of pure KOH. (Apparently, the initial reaction with KI is much more rapid than that with KOH.) The dark-brown color that is characteristic of iodine appears in the solution when the KOH is depleted, giving a simple end point. This end-point determination was used previously in BrF_5 flow calibrations. Such a system is applicable to the disposal of both bromine fluorides and fluorine in the present spray disposal equipment in the pilot plant.

Fluoride Volatility Processing: PuF_6 Process Tests. — Studies began at the plutonium facility in Building 3019 in support of the proposed fluidized-bed process for stainless-steel-clad and Zircaloy-2-clad power reactor fuels. Initial tests were made, primarily, to determine material balances and deposition profiles for plutonium in the disengagement section of the 0.9-in.-ID fluidized-bed reactor. Subsequent tests have been concerned with the evaluation of PuF_6 sorption by LiF. In the first two tests, using 40-g beds of alumina (Norton RR, 90 mesh), 14.0-g charges of PuO_2-UO_2 (5.4 wt % PuO_2) were fluorinated for 4-hr periods at 550°C, with a fluidizing gas velocity of 0.55 fps. Plutonium deposits on the blowback filters and on the walls of the disengagement section were about 2 and 1% respectively. Material balances were 100.2% in one test and 84.5% in the other.

In the first test to study the sorption of PuF_6 by LiF, 7 g of 5.4 wt % PuO_2—UO_2 with type 304 stainless steel was oxidized and fluorinated (fluorination conditions as described above). The trapping system consisted of two 25-g beds of LiF (−12 +20 mesh) in series at 300°C, followed by a 10-g bed of NaF. Of the 331 mg of plutonium volatilized as PuF_6, 13 mg was retained by one LiF bed and 9 mg by the other, to give a total sorption of 7%. In subsequent sorption tests the fluorination time was reduced to 3 hr, the use of stainless steel and the oxidation cycle was omitted, and operation of the LiF bed was changed. The following tabulation summarizes the results from these tests, along with those from a special run that used a subfluidization gas velocity of 0.1 fps to give an average F_2:PuF_6 mole ratio of 1270:1. (The sorption values are based on only the plutonium found in the LiF and NaF traps, that is, the material that was actually in the form of PuF_6 on leaving the fluidized-bed reactor.) These data indicated that some PuF_6 may have been thermally decomposed to PuF_4, especially in the subfluidization run.

Average Mole Ratio (F_2:PuF_6)	Weight of LiF (g)	Bed Temperature (°C)	Sorption (%)	Material Balance (%)
5700	50	300	7	106
3800	50	300	34	93
5700	50	250	56	107
5700	100	350	81	92
1270	50	300	99.6	58

In a duplicate of the subfluidization run, a neutron counter near the LiF bed showed that essentially all the PuF_6 was sorbed by the LiF bed in the first 0.5 hr of the 3-hr total. The F_2:PuF_6 mole ratio in this initial period was, therefore, actually about 200:1 instead of 1270:1. The actual value was well below the ratio of 396:1 that is required to prevent thermal decomposition at 300°C.

In the other tests, with higher F_2:PuF_6 ratios, sorption results indicated unfavorable kinetics. The poor overall material balance in the subfluidization run and the high material balances in the other tests were also consistent with only a small thermal decomposition effect in the latter.

We tentatively conclude that recycle of fluorine gas from the sorption unit back to the fluidized-bed reactor is the only way of using the LiF sorption process with a reasonably small sorption bed.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Reactor Analysis. — On September 9, 1966, the HFIR achieved full design power of 100 Mw for the first time and is continuing to operate at that power with no difficulties.

The central target used in this initial 100-Mw run contained enough ^{235}U to provide the maximum expected heat generation rate for a 300-g plutonium target. There is no indication that the target overheated.

Previous to operation at 100 Mw, one fuel loading was consumed without difficulty at 20 to 50 Mw, one at 75 Mw, and one at 90 Mw, the burnup at each power level being about 2300 Mwd. The anticipated shorter life for the 100-Mw core is the result of a more poisonous target.

After seven years of design and development it finally appears that the highest-performance research reactor in the world is a success. Some of the more impressive characteristics are as follows:

Power, Mw	100
Average power density, Mw/liter	2.0
Core volume, liters	50
Peak thermal flux, neutrons cm^{-2} sec^{-1}	5×10^{15}
Peak total nonthermal flux, neutrons cm^{-2} sec^{-1}	4×10^{15}

Metallurgy. — Work at Metals and Controls, Incorporated, the fuel element fabricator, is picking up and is again approaching the scheduled rate of two elements per month. Because of vacations and the temporary transfer of men to other jobs, the production schedule had slipped considerably during the last few months. Technically, the job is proceeding smoothly, although minor waivers still are being granted on each element. The fuel plate rejection rate seems to be stabilizing at just below 10% rejection, although occasionally higher values are still being encountered. To date, 12 complete fuel assemblies have been received from Metals and Controls.

The HFIR fuel element specifications have been revised and brought up to date in preparation for placing a new order. A considerable effort has been spent in discussion with the AEC on what type of contract should be used for the next order and how it is to be handled. The Laboratory has recommended that Metals and Controls receive an extension of their contract but with an increase in fee.

Reactor Operations. — The reactor was brought to its maximum design power level of 100 Mw on September 9, 1966, and has operated smoothly since then. The total energy accumulated to date is about 9000 Mwd. During the approach to the 100-Mw power level, several flow-coastdown tests were made and the reactor was brought back to power successfully each time without a scram.

During the 90-Mw run the reactor was scrammed two times as a result of power outages. However, the reactor was brought back to power each time within minutes without difficulty.

The shielding, cooling systems, and other auxiliaries appear quite adequate for 100-Mw operation, and no significant design deficiencies have been detected.

PHYSICAL RESEARCH

Reactor Operations. – During the months of July and August, the ORR and the LITR were operated 85 and 91% of the time respectively. Revisions of the ORR and LITR safety reports are being prepared to update the descriptions and other information.

The BSR was operated for approximately 225 hr during this period. In August the pool was drained and the reactor dismantled in preparation for modification of the reactor for the capability of continuous 2-Mw operation.

By the end of August, 70% of the fuel in the ORNL Graphite Reactor had been unloaded and shipped to Savannah River for processing.

Waste Disposal. – To increase the reliability of the steam turbines and the other components which must function to preserve cell ventilation and off-gas disposal in the event of electric power failure, the pressure-sensing devices which start the turbines were duplicated, as was each component that is actuated by the sensing devices, including the steam control valves. Back-draft preventers were installed in series with each pneumatically operated damper in the cell ventilation ducts to ensure positive closure if for some reason a damper should fail in the open position. Pneumatically operated valves in the smaller off-gas lines were replaced with check valves, which are considered more reliable.

The operating procedures have been revised to reflect the changes made, and testing of the entire system is now carried out on a routine basis.

Hot-Cell Operations. – The surveillance test from the MSRE was disassembled. Several graphite specimens were sampled by removing the surface in approximately 5-mil steps, and the samples were submitted for chemical analysis. The stringers of INOR-8 tensile specimens were sectioned into the individual specimens.

The molten-salt loop from HN-1 in the ORR was sectioned, and the failures were located. Samples were selected for metallographic and analytical examination.

One in-pile UO_2 meltdown experiment was examined; NSPP meltdown No. 15 was disassembled, photographed, and sampled for analysis. Examination of TRU target rod No. 4 was completed. A thorium-utilization capsule, three UO_2 capsules, and several coated-particle capsules were examined in detail. Several isotope source capsules (empty or containing alpha sources) and several PuO_2 pellet or sphere samples were examined metallographically. Two SM1-A absorber boxes were received from an Army reactor in Alaska; these were stored and will be examined at a later date. Specimens were recovered from 20 experiments in the ORR. Full operation of the eight creep-test machines in the cells continued.

PHYSICS AND MATHEMATICS

Charge Spectrometry. – The probability of ejecting two or more electrons by the interaction of an x ray with the outermost shell of He, Ne,

and Ar has been investigated. Energy spectra for electrons ejected in photoionization were measured with an electron spectrometer. In addition to characteristic photolines, continuum spectra were found, which are related to double electron ejection. The probabilities for double electron ejection as determined from these spectra are in agreement with those obtained by charge spectrometry. The shapes of the continuum spectra are qualitatively similar to those expected for electron shakeoff. However, the probabilities for electron shakeoff as calculated from single-electron wave functions are much smaller than those found experimentally. It is suggested that a many-body solution is necessary, which explicitly includes electron correlation. For example, such a calculation by Salpeter and Zaidi for the case where one electron goes to the continuum while the other goes to the 2s state in the photoionization of helium is shown to be in good agreement with experiment.

Theoretical Physics. — It is well known that the assumption of strong variations at small distances (hard core) in the interaction between two nucleons in free space leads to a density dependence of the effective interaction in nuclear matter. We have investigated a particularly simple density-dependent effective two-nucleon force appropriate for use with the Hartree-Fock approximation. Our motivation comes from Bethe's recent work in which a density-dependent interaction occurs as one of the intermediate steps toward a Thomas-Fermi theory of finite nuclei. Skyrme pointed out long ago that such density dependence is required for a consistent fit of nuclear matter and of light nuclei. We have made Hartree-Fock calculations of the nuclei ^{12}C, ^{16}O, ^{28}Si, and ^{40}Ca and have obtained binding energies, root-mean-square radii, densities, and single-particle levels. We have also calculated the overlaps of the single-particle wave functions with pure harmonic oscillator functions whose b is chosen to give the same root-mean-square nuclear radius. Our conclusion is that harmonic oscillator functions are excellent for the occupied states of light nuclei.

High-Voltage Experimental Program: $^{39}K(^{3}He,p)^{41}Ca$ Reaction and 2p-1h States. — The $^{39}K(^{3}He,p)^{41}Ca$ and $^{39}K(^{3}He,d)^{40}Ca$ reactions have been studied at an incident ^{3}He energy of 14 Mev with the expectation that states in ^{41}Ca and ^{40}Ca which are formed on the $(d_{3/2})^{-1}$ ground state of ^{39}K will be preferentially populated. The negative-parity states in ^{40}Ca are strongly excited, indicating their 1p-1h nature. This is in agreement with the observations of Erskine that in ^{41}Ca the well-known single-particle states are excited very weakly, the yield being less than 10% of the strong 2p-1h states identified near 5 Mev.

High-Voltage Experimental Program: Neutron Total Cross Section of $(^{204}Pb + n)$ for $5 < E_n$ (kev) < 80 and of $(^{208}Pb + n)$ for $26 < E_n$ (kev) < 84. — The neutron total cross section for $(^{204}Pb + n)$ was studied from 5 to 80 kev using fast time-of-flight techniques. At least 20 s-wave resonances were observed in this interval in addition to a number of probable p-wave resonances. The average observed s-wave level spacing is 2.7 ± 0.5 kev, and the s-wave strength function is $(0.55 \pm 0.2) \times 10^{-4}$. The total cross section for $(^{208}Pb + n)$ was measured from 26 to

84 kev. Resonances were observed at about 70 and 77 kev, both apparently due to $\ell = 1$ neutrons. The effective nuclear radius for ($^{208}Pb + n$) was found to be 8.4 ± 0.3 fermis.

High-Voltage Experimental Program: Total Neutron Cross Section of Nitrogen from 2.0 to 4.2 Mev with Good Energy Resolution. — An interpretation of differential scattering [Bull. Am. Phys. Soc. 11, 653 (1966)] and reaction cross sections [Nucl. Phys. 14, 277 (1959)] of neutrons on nitrogen requires more detailed information on the total cross section than appears in the literature. Accordingly, we have measured the neutron total cross section of nitrogen from 2.0 to 4.2 Mev with ~5 kev energy spread. Protons from the ORNL 5.5-Mv Van de Graaff incident upon ZrT targets furnished the source of T(p,n) neutrons. We measured the transmission of a 7.35-g/cm^2 sample of LiN_3 and made the correction for the lithium by use of lithium cross sections taken from the literature (BNL-325, 2d ed., suppl. 2, 1964). We assign J values and widths for four resonances in this energy region as given below, where the resonant energy in kev is followed by the J value and widths, the latter also in kev: (2230, 3/2$^-$, 65), (2950, 5/2, 14), (3210, 3/2, 85), and (3570, 3/2, 30). The parities of some of the resonances can be assigned on the basis of differential cross-section measurements [Bull. Am. Phys. Soc. 11, 653 (1966)].

Mathematics Research: Products and Linear Combinations of Involutory Matrices. — A nonsingular matrix A with elements in any field is involutory if $A = A^{-1}$ in the field. An nth order involutory matrix A with elements in a field F is of signature s if and only if $A = I + cQP$, where each of Q and P^T is $n \times s$ of rank s, where $c = -2$ and $PQ = I_s$ for $0 \leq s \leq n$ if F has characteristic $\neq 2$, and where $c = 1$ and $PQ = 0$ for $0 \leq 2s \leq n$ if F has characteristic $= 2$ [Am. Math. Monthly 69, 267 (1962)]. Two involutory matrices $I + cQP$ and $I + cQ_oP_o$ are said to be in the same P class (same Q class) if there exists a nonsingular matrix B such that $P = BP_o$ ($Q = Q_oB$).

Let $A_i = I - 2\,Q_iP_i$, $i = 1, \ldots, r$, be $n \times n$ involutory matrices of signature s in the P class [P] with elements in a field F of characteristic $\neq 2$. Then $\left(\sum_{i=1}^{r} \ell_i\right)^{-1} \sum_{i=1}^{r} \ell_i A_i$ is an involutory matrix in the P class [P] for all ℓ_i in F such that $\sum \ell_i \neq 0$, and $\prod_{i=1}^{r} A_i$ is an involutory matrix in the P class [P] if r is odd. It should be noted here that for r odd, $A_1 A_2 \ldots A_{2k+1} = A_{2k+1} \ldots A_2 A_1$, while no pair of distinct matrices A_i, A_j commute. Moreover, if at least two of the A_i are in the same P class, a necessary and sufficient condition that every product of an odd number of the A_i be involutory of signature s is that all the A_i be in the same P class.

If the $A_i = I + Q_iP$, $i = 1, \ldots, r$, are $n \times n$ involutory matrices of signature s in the same P class [P] with elements in a field F of characteristic 2, then the matrix $\prod_{i=1}^{r} A_i$ is involutory of signature the

rank of $\sum_{i=1}^{r} Q_i$, and $\sum_{i=1}^{r} A_i$ is involutory of signature the rank of $\sum_{i=1}^{r}$ if r is odd.

Mathematics Research: Solutions of Transcendental Equations. – An elegant method that appears to be new has been developed and tested for locating the zeros of an arbitrary transcendental function f(z) within a simply connected region of analyticity. It is based upon the fact that the integral of $z^m f'(z)/f(z)$ taken over the closed boundary curve is equal to $2\pi i$ times the sum of the mth powers of the zeros within that region, provided there is no zero on the boundary itself. In particular, for m = 0 the integral yields the number of such zeros.

Programming Research: Tungsten Deposition Studies. – As part of a program to investigate the deposition of tungsten on cylindrical mandrels, an experimental design consisting of 81 experiments has been completed. The primary purpose of the design is to estimate the effects and interactions of four process variables in a full cubic model on tungsten efficiency and deposition rate. Although statistical analysis of the data is incomplete, contour graphs have been obtained which illustrate the physical shape of the response surface.

Programming Research: Likelihood Ratio Test for Multinomial Distributions. – In some statistical applications, n observations from a hyopthetical population could be classified into k mutually exclusive categories. There will be some theory or hypothesis which gives the probability π_i that an observation falls into the ith category, which one would like to test by experimentation. There are at least two statistics for this purpose, referred to as the likelihood ratio test and Pearson's X^2 statistic. It is well known that if we have samples of extremely large sizes, these statistics behave alike. But in practice we will have to deal with small sample sizes. We have initiated a study on the exact distribution of these statistics in the case of the trinomial distribution, and have developed computer algorithms to study the validity of the large sample theory.

Programming Research: Exponential Approximation. – An iterative procedure has been developed and programmed to fit data by a function of the form $f(x) = \sum_{i=1}^{n} a_i e^{b_i x}$. The parameters $a_1, a_2, \ldots, a_n, b_1, b_2, \ldots, b_n$ are approximated by the solutions of sets of linear equations. These approximations are then used to obtain new sets of linear equations and the process is repeated.

The procedure requires estimates only for $b_1, b_2, \ldots, b_n$.

CHEMISTRY

Isotope Chemistry. – Studies were made of the physical and chemical properties of molecular addition compounds formed by BF_3 and group VI-B donors. The dimethyl telluride complex was too unstable to exist at

temperatures even as low as -30°C. The freezing point of the 1:1 dimethyl selenide·BF_3 compound was -43°C. From room temperature to its freezing point, the saturation pressure of the 1:1 dimethyl selenide complex was given by $\log_{10} P = 9.945 - (1824/T)$. The 1:1 butyl ether and ethyl formate complexes formed at 25°C but deteriorated slowly with the formation of noncondensable gases. The freezing points of these complexes were -30 and -8°C respectively. Between room temperature and their freezing points, the saturation pressures of freshly prepared $Bu_2O \cdot BF_3$ and HCOOEt·BF_3 were given by $\log P = 5.65 - (1010/T)$ and $\log P = 5.70 - (1330/T)$ respectively. For the same temperature range, the equilibrium constant for the isotopic exchange reaction

$$^{10}BF_3(g) + A \cdot {}^{11}BF_3(\ell) = {}^{11}BF_3(g) + A \cdot {}^{10}BF_3(\ell)$$

was given by $\log K_{eq} = (8.10/T) - 0.0130$ when A was dimethyl selenide, and by $\log K_{eq} = (1.6/T) + 0.0059$ when A was dibutyl ether. Previously reported equilibrium constants for the Me_2O and Me_2S exchange reactions were redetermined: for Me_2O, $\log K_{eq} = (8.76/T) - 0.0179$, and for Me_2S, $\log K_{eq} = (10.7/T) - 0.020$.

Based upon saturation pressures, the stabilities of the chalcogen complexes of BF_3 decreased in the order $Me_2O \cdot BF_3 > Me_2S \cdot BF_3 > Me_2Se \cdot BF_3 >$ HCOOEt·$BF_3 \geq Bu_2O \cdot BF_3$. The order of the dimethyl-substituted donors was explicable in terms of donor radii; the positions of the HCOOEt and Bu_2O complexes seemed to be determined by steric and inductive effects.

The equilibrium constants for the isotopic exchange of boron between a molecular addition compound and BF_3 varied with donor in the following manner: $Me_2S > Me_2Se > Me_2O \geq Bu_2O$. Except for the Me_2Se donor, this order follows that predicted on the basis of our previously stated rule that the equilibrium constant varies inversely with the strength of the boron-donor bond. The exceptional position of the Me_2Se donor was explained in terms of two opposing effects whose contributions to the isotopic equilibrium constant vary with increasing donor size: the electron-pair donating powers of the donor atoms, and the polarizabilities of the donors.

Effects of Radiation on Analytical Methods. - The study being carried out on the reaction mechanism of the zirconium-alizarin red S (ARS) complex has been completed. It has been determined that in acid medium (<1 N), zirconium and ARS form a 1:1 complex in which the zirconium is bound to the ARS via the 1,2-hydroxy functional groups. The quinoid oxygen is not directly involved in the Zr-ARS complex. The rate of the reaction has been found to be a function of the rate of hydrolysis of the zirconium ion to presumably the $Zr(OH)_2^{2+}$. This complex ion then reacts with the ARS to give the Zr-ARS complex. The effects of other parameters on the reaction such as temperature, zirconium concentration, etc., have also been studied.

The following conclusions have been drawn from the study on the rate of dissolution of some inert metals in HCl while undergoing alpha irradiation.

1. Gold, zirconium, tungsten, and platinum dissolve in irradiated HCl at measurable rates. The rate at which niobium dissolves is slower but still measurable. Tantalum shows the greatest resistance to dissolution in this medium.

2. The rate of dissolution is dependent on the amount of free chlorine being produced, which, in turn, is a function of acid and chloride concencentrations and dose.

3. The rate of dissolution is relatively unaffected by the surface area of the metal.

4. The G value for metal dissolution at best is one order of magnitude below that of chlorine formation.

The following rates were measured when each metal was placed in a comparatively large volume of HCl and irradiated:

Metal	Rate of Dissolution	
	(mg/10^6 rads)	(micromoles/10^6 rads)
Au	2.50	12.7
Zr	1.12	12.4
W	1.10	6.02
Pt	1.09	5.58
Nb	0.03	0.03
Ta	Below detection limits	

A redetermination of the rate of acid production from the radiolysis of chloral hydrate solutions shows that the relation between the concentration and the rate is much less than that reported by other workers. There is roughly a 50% increase in rate as the concentration is increased from 0.01 M to 1 M. The acid production at each concentration level is a linear function of the radiation dose, but the curves do not pass through the origin and have a common positive intercept.

Physical Chemistry of Molten Salts: Computer Programs for Crystal Structure Determination. — The Fourier summation and electron density mapping program for crystal structure determination, FORDAPER, has been brought up to date and made compatible with the IBM 360/75. The original program was written by Allan Zalkin of the Lawrence Radiation Laboratory and modified by Ibers and Hamilton of Brookhaven National Laboratory and by Levy and Ellison of ORNL. The new FORTRAN/360 version contains all of the previous modifications plus the addition of a new map-plotting subroutine for the CDC Dataplotter and a subroutine for the interpolation of both positive and negative electron density peaks in three dimensions. Source decks for this program may be obtained from G. D. Brunton, Reactor Chemistry Division.

Physical Chemistry of Molten Salts: Physical Properties of Molten Fluorides. — Densities and viscosities of BeF_2 and BeF_2-LiF mixtures were measured to determine if their temperature coefficients of viscosity are correlated with thermal expansion coefficients. For BeF_2, the viscosity, which varied between 10 and 10^6 poises over the temperature interval 573 to 985°C, exhibited Arrhenius behavior. The density of BeF_2

appeared to change only very slightly with temperature. For the BeF_2-LiF mixtures, the viscosity measurements confirmed earlier results which showed marked decreases of viscosity and its temperature coefficient with LiF concentration. From the density measurements, it was found that between 11 and 50 mole % LiF, thermal expansion coefficients decreased with decreasing LiF concentration. Tentatively, these results indicate that the temperature coefficient of viscosity decreases with increasing thermal expansion coefficient, and in particular (1) the Arrhenius behavior of pure BeF_2, like that of SiO_2, is associated with the temperature independence of free volume; (2) LiF, when dissolved in BeF_2, not only breaks up the network of linkages between beryllium and fluorine, but also increases the temperature dependence of the free volume, thereby decreasing the activation energy of viscous flow.

Chemical Engineering Research. — Scale-up studies of the stacked-clone solvent extraction contactor were made in machines that were 0.5 and 1.4 times the size of the standard contactor. The stage efficiency, using uranyl nitrate as the transferable solute in the system 1 M $NaNO_3$—18% tributyl phosphate in Amsco, averaged 75% and was independent of contactor size.

The flow capacity of the 0.5-scale machine was 40% of the standard machine; that of the 1.4-scale machine was 119%. Assuming that the pumps and lines were scaled proportionally, the relative residence times per physical stage for the 0.5-, 1.0-, and 1.4-scale contactors are 0.3, 1.0, and 2.3 respectively.

Separations Chemistry Research. — The well-known separation factor of 2.5 between adjacent lanthanides, usually observed in extractions from HNO_3 solutions with di(2-ethylhexyl)phosphoric acid, does not apply to some of the lighter members of the group. Whereas such a formula approximately applies to the overall range of elements from lanthanum to europium, we found that cerium is considerably more extractable and that praseodymium is slightly more extractable than predicted. Neodymium, on the other hand, is slightly less extractable than expected under various conditions. These anomalies probably explain the difficulty, reported by some workers, in separating these elements by extraction chromatography.

Volatility Studies. — A desirable step in the volatility process would be the use of a solid sorbent to selectively and reversibly sorb PuF_6, in much the same way that NaF is used in the UF_6-NaF system to recover and decontaminate UF_6. The PuF_6-LiF system, while not an exact analog of the UF_6-NaF system, may be adequate; therefore it is being more completely evaluated.

The equilibrium between PuF_6, fluorine, and Li_4PuF_8 is being studied by a fluorine flow method analogous to the transpiration method used in vapor-pressure work. For these measurements, about 300 mg of plutonium (as PuF_6) was first sorbed onto an 8-g bed of LiF with sufficient excess fluorine to avoid deposition of PuF_4 as a result of thermal decomposition of PuF_6. A total of 15 "good" equilibrium points were taken. For each point, with the LiF at a controlled temperature, a regulated flow of fluorine (50 ml/min, except for one test at 100 ml/min) was passed through the bed for periods of 60 to nearly 1000 min; PuF_6 in equilibrium with the bed and the fluorine was then collected in a backup NaF trap. The 15

points fit the equation

$$\log K = 2047/T + 0.817 ,$$

where $K = F_2/PuF_6$. From this straight-line relationship, values of K at the following temperatures (in °C) of interest were found to be: 2.01×10^6 at 100, 1.40×10^5 at 200, 2.45×10^4 at 300, 7.25×10^3 at 400, 2.92×10^3 at 500, and 1.45×10^3 at 600. The free-energy change for the reaction

$$LiF_4PuF_8 + F_2 \rightleftharpoons PuF_6 + 4LiF$$

is $\Delta F = 9360 + 3.74\ T(K)$ cal/mole.

TRANSURANIUM-ELEMENT PROCESSING

TRU Operations. — Dissolution of the first of four TRU-SRL irradiation elements (HFIR prototypes) was completed, representing the first full-scale processing in the facility. Approximately 70% of the actinides predicted to be present in the target were dissolved in uncatalyzed 15 M HNO_3; most of the remainder were dissolved in a subsequent step using fluoride as a catalyst. The two dissolver solutions were held separately. After adjustment of the acid concentration to 7.5 M, and oxidation of the plutonium to the tetravalent state, the fluoride-free solution was passed through the ion exchange system (Permutit SK resin), where high-purity plutonium was recovered (separation factor, approximately 600) with low (about 1%) losses. The fluoride-catalyzed dissolver solution was adjusted with H_3BO_3 to complex the excess fluoride and to permit redissolution of any precipitated actinide fluorides, and the actinides were precipitated with NaOH. This alkaline mixture is being stored temporarily until the material from the first dissolution has been processed.

The americium-curium product recovered from the plutonium ion exchange run was evaporated; the actinides were precipitated and centrifuged, and the resulting solid cake was washed several times. Measured actinide losses were 0.3%. The actinide cake was redissolved in 3 M HCl in the centrifuge bowl with good (greater than 99%) recovery. A cleanout dissolution indicated that only traces of actinide remained in the centrifuge bowl, which confirms the effectiveness of this technique.

Before this americium-curium product was purified by the Tramex solvent extraction process, a tracer-level run was made with a sample of the real feed as a "spike." During this run, which lasted 21 hr, the system operated quite satisfactorily and without interruption. Solvent extraction losses totaled 0.3%; 0.1% occurred in the raffinate, and 0.2% occurred in the stripped extractant. Decontamination from fission products improved throughout the run, with the decontamination factor (DF) increasing from about 15, initially, to greater than 35 at the end.

The actual processing of the dissolved target material was delayed by various mechanical difficulties and by an unexplained loss of acid from

the originally adjusted feed. After readjustment of the feed and after a period of smooth operation with "cold" feed, operation with radioactive feed was begun. This run continued for 17 hr, at which time a plugged air-bleed orifice assembly formed an air lock in the extraction column. Before the solvent extraction system could be restored to operation, a power failure occurred, forcing a complete shutdown. Cleanout and recovery operations were begun.

During the 17-hr period of operation, approximately 35% of the feed was pumped to the system. Extraction losses steadily increased, approaching about 3% when the feed was stopped, indicating that chemical equilibrium had not been reached at that time and that salting strength in the feed was not sufficiently high for good extraction. Stripping losses, measuring 0.05%, were satisfactory. Decontamination of the recovered product from fission products was satisfactory (DF = 35); a high background that was attributed to spontaneous fission products obscured any peaks on the product in-line gamma analyzer. Based on radiochemical analyses, DF's for ^{106}Ru, ^{95}Zr, and ^{144}Ce were 160, 6, and 560 respectively. Although lower than desirable, the decontamination from ^{106}Ru and ^{144}Ce approached satisfactory levels; that from ^{95}Zr did not. An organic backscrub will be added to improve decontamination from zirconium in future runs. The californium content of the first target rod now being processed was determined to be about 35 μg, based on analyses of the dissolver solution.

Inspection of the 18 HFIR elements irradiated at Savannah River Laboratory has been completed. Seventeen elements were returned to the HFIR for additional irradiation; nine of these required refurbishment with new hex can sheaths. The fin damage was similar on all elements, but the integrity of the welds and tubing was excellent. All the elements were helium leak-tested and found to be leak-tight. One element is to be retained for dissolution and for chemical separation of the actinides to obtain an estimate of the composition of the other elements at the start of the irradiation in the HFIR.

Process Development. — In continued studies of the cation exchange separation of actinide elements, four experiments were completed with up to 250-mg quantities of ^{241}Am and ^{242}Cm in cell 3 of the Curium Recovery Facility. Columns 1 in. in diameter and 28 in. long, packed with 274 ml of ammonium-form Dowex W-X12 resin, were loaded with equal amounts of ^{242}Cm and ^{241}Am (125 to 250 mg of each). Chromium and iron (total of about 2 g), several rare earths (trace quantities), and ^{106}Ru, ^{95}Zr, and ^{238}Pu were present as impurities.

In the first and fourth runs, about 125 mg of ^{242}Cm was used. Resin degradation at these activity levels (about 1200 w/liter) was not severe, and physical performance of the column throughout the runs was generally satisfactory. Both the stable and radioactive impurities were eluted with ammonium α-hydroxyisobutyrate from the column ahead of the americium-curium fraction. The best separation of ^{241}Am from ^{242}Cm was accomplished in the final run, in which a product fraction containing about 80% of the ^{242}Cm and 10% of the ^{241}Am was obtained. A center cut of this fraction contained 50% of the ^{242}Cm and only 1% of the ^{241}Am. In this run, flow rate characteristics were improved considerably after the glass frit (at the top of the column), used to pressurize the resin bed, was replaced

with a platinum screen. This prevented a gradual loss in flow rate, which was evidently a result of plugging and disintegration of the glass frit.

In the third run, severe resin degradation occurred with a ^{242}Cm loading of 250 mg. Approximately 1 in. of the resin bed was charred, and about 16% of the curium was irreversibly retained in the charred resin. Since ^{238}Pu (produced by ^{242}Cm decay) was not found in any of the fractions eluted from the column, the top 3 to 4 in. of resin was removed from the column after the fourth run and was digested in strong acid. Analysis of this solution showed that it contained about 0.1 mg of ^{238}Pu. These results indicate that the plutonium in the feed solutions was probably polymerized and retained irreversibly on the resin.

As a result of this work, we now feel confident that 100 to 150 mg of ^{252}Cf can be processed satisfactorily on columns of this size.

METALLURGY AND MATERIALS

Direct Observation of Lattice Defects. — The sudden change from ductile to brittle behavior of most body-centered cubic structural metals can seriously limit their application. Tungsten, which has attractive properties for use in high-temperature reactors, also has a ductile-to-brittle transition temperature above room temperature. This creates handling and fabrication problems. We recently found strong evidence that impurities segregated at the grain boundaries raise the ductile-to-brittle transition temperature of recrystallized tungsten. The results indicate that the impurities are probably segregated in solid solution at the boundaries. This interferes with the initiation of dislocations from grain-boundary sources, makes transmission of slip from grain to grain more difficult, and weakens the cohesive bonding across the boundary. Thus, dislocations are not generated until some high local stress level is reached. The developing slip band runs into a stiff barrier at the boundary, and the stress concentration at the point of contact is relaxed by formation of a brittle crack in the grain boundary.

While impurities are obviously important in raising the ductile-to-brittle transition temperature, we believe that the basic cause of the transition lies in the nature of dislocation activity. Brittleness may be associated with straight very narrow slip bands, which are efficient stress concentrators, and ductility with diffuse wavy slip bands. Currently we are using electron microscopy and etch-pitting techniques to examine the effects of temperature and stress level on the nature of slip bands in tungsten single crystals and their relationship to the ductile-to-brittle transition.

Physical Ceramics. — Continuing our studies of the sintering of ThO_2 and other materials with the same fluorite crystal structure, we found that partially sintered powder compacts sintered further on very short reheating if they had been isostatically pressed before the second heat treatment. A possible explanation rests on the premise that stresses present during the room-temperature isostatic pressing create dislocations. Subsequently, at elevated temperature these dislocations move and effect material transport, which would not have occurred without

the isostatic pressing between heat treatments. These results are compatible with our previous demonstration that the kinetics of the initial densification are not compatible with a diffusion-controlled process. Both results suggest, in a negative sense, that dislocation motion contributes to material transport.

Metallurgy of Superconducting Materials. – We have developed new chemical polishing and anodizing procedures for the room-temperature metallographic preparation of specimens of lanthanum and cerium. These methods now allow the study of the morphology of the phase transformation, face-centered cubic to double-close-packed hexagonal, which occurs in these elements near room temperature. Previously, the polished and etched surfaces of the specimens were so reactive to laboratory air that there was not sufficient time to examine and photograph the microstructures before the surface deteriorated to uselessness. The anodized surface of the specimen is stable for several days, each phase can be identified by its characteristic color, and the specimen can be examined and photographed in incident polarized or unpolarized white and monochromatic light. These phase transformations were studied previously only by x-ray diffraction techniques, which reveal nothing of the morphology of the transformation. A study of the morphology of the phase transformation in both lanthanum and cerium was started, and the results will be used to prepare specimens of known microstructural condition for the determination of the superconducting properties as functions of the amounts of the different phases present in the specimen.

CONTROLLED THERMONUCLEAR RESEARCH

Single-Pass Injection-Accumulation Experiments (DCX-1, DCX-3). – During this report period, work has continued on the coil configuration design for DCX-3, and certain experiments have been carried out which indicate the beam and vacuum performance for DCX-3. Work on the design of the magnetic bottle for DCX-3 has continued along two paths:

1. Ribbon coils. The results of hydraulic tests and of stress analyses of the proposed mechanical structure have in the main been encouraging. Some detailing of construction drawings is in progress. The principal problem continues to be the unavailability of epoxy-coated copper ribbon in sufficient quantities to permit construction and test of prototype coils of the quadrupole set.

2. Superconducting coils. Specifications for a magnet system using superconducting coils are almost complete. These specifications include calculations of magnetic-field intensities within the coils themselves and of electromagnetic forces generated by the coil system.

A choice between these two alternatives is expected soon.

Preliminary experiments utilizing the 10-kilogauss DCX-1 mirror field and injecting 15-kev H^0 with intensities up to 44 ma have been undertaken. With confinement times near 5 msec a strong microinstability was observed at the gyrofrequency and its second harmonic. We are now in process of utilizing our energy analyzer and calibrating our secondary

electron emission detectors in order to determine energy spreads and densities of this plasma as well as determining the polarization of this emission at the gyrofrequency.

Deliberate energy spread of the injected 15-kev H^0 beam has been possible to 25% full width at half maximum and 44 ma in the present configuration, and the transformer is being put in series with the extraction voltage in order to allow such spreads to be made at a rate of 360 per second. An apparatus is being assembled which will allow studies of arc excitation of an H^0 beam and its subsequent trapping.

Ion Source and Ion Beam Technology. — Satisfactory injection systems have been developed for DCX-3 and INTEREM. A current equivalent of 250 ma of 20-kev H^0 particles can be passed through the INTEREM plasma. Differential calorimeter probes developed previously have been improved and have been used to obtain the radial current density distribution in the neutral beam test system. The program of development leading to the design of a new, shorter, and more stable 600-kev accelerating system is continuing.

Properties of Plasmas Created by Electron-Cyclotron Heating. — The ELMO Facility is being operated with positive-gradient open-ended magnetic geometries. A magnetic well with a zero minimum is obtained with the folded cusp configuration, whereas a finite-minimum magnetic well is created with the addition of a current-carrying "stuffing bar" on the axis. Up to 5 kw continuous wave of 10.6-Gc microwave power is being used to create an electron-cyclotron plasma in the facility.

Detectors placed in the point and line cusp regions measure both the slow and fast particle currents. Total-energy measurements, microwave emission measurements, and bremsstrahlung measurements have contributed to the study of instabilities. The instabilities seen are related to (1) the strength of the external field, (2) the gradient of the field, and (3) the amount of cold plasma. The detailed nature of the instabilities is receiving major attention.

The INTEREM Facility has been shut down for modifications. A new ion beam source, accelerator, and neutralizer are being added to increase the neutral beam current into the facility. Currents equivalent to about 100 to 200 ma are expected from the new sources.

Properties of Plasmas Created by Beam-Plasma Interaction. — The modified Burnout V, with its larger midplane diameter and longer axial length, has been operated at about the same steady-state beam power input and plasma density as before modification. The intensity of impurity spectra was reduced, as expected. The beam power input appears to be limited by the temperature of the carbon mirror orifices, the anodes. The pressure gradient was not steepened as expected. Steeper pressure gradients have been achieved by adding additional bursts of gas to the midplane region. The beam current increases as the gas is added, and the beam voltage is held up by a capacitor bank that dumps its energy into the discharge. A neutron burst occurs at the time of the gas pulse. The technique provides a way of increasing the pressure gradient and suggests a method for measuring plasma lifetimes.

In separate work carried out in the beam-plasma facility, our attention has been directed toward increasing the electron density in the hot-electron blanket. Two arcs are being used, the first located 6 in. off

axis as before, and the second located 4 in. off axis. The second arc also has an iron shield concentric with the anode channel in order to compensate for the higher magnetic field present in the second anode. The operation of the 6-in.-off-axis arc has been improved with no increase in operating pressure. Both the saturation current streaming out the mirrors and the power delivered to the power probe indicate that the density of the intermediate-energy electron distribution has been increased by a factor of 2 to 4; the density is 5×10^{11} cm^{-3} or more.

Alignment of the second arc in the new 4-in. position is now being undertaken.

Theoretical Plasma Physics. - It is well known that a plasma is unstable for some velocity distributions, $f(\vec{v})$. These are the familiar "velocity-space" instabilities. Linear theory predicts an exponential growth of the amplitude of the wave. Quasi-linear theories attempt to discover how the fluctuating fields can modify the velocity distribution, $f(\vec{v})$, in such a way as to restore stability. When stability is restored, there remains a certain spectrum, $N(\vec{k})$, of waves in the plasma ($\vec{k}$ is the wave vector). The usual quasi-linear equations do not in general predict an approach to thermal equilibrium. We have derived modified quasi-linear equations which predict that the particle velocity distribution approaches the Maxwell-Boltzmann distribution and that the energy distribution of plasma oscillations approaches the Rayleigh-Jeans distribution. These equations differ from the usual quasi-linear equations by terms due to spontaneous emission of plasma oscillations by particles.

Atomic Cross Sections. - An existing charge spectrometer has been supplemented with gas cells and an electrostatic energy analyzer to allow investigation of charge-exchange reactions. Emphasis is laid upon measurements of multiple electron transfers to highly charged rare-gas ions. Preliminary results show a large probability of transferring two and three electrons from neutral Ar to Ar^{4+} and Ar^{5+} ions. Ratios σ_{42}/σ_{43} = 0.4 and σ_{41}/σ_{43} = 0.08 were obtained for Ar^{4+} ions of 20 to 40 kev energy.

Measurements have been made of the electron stripping cross section (σ_{01}) and the statistical charge equilibrium fractions ($F_{1\infty}$ and $F_{0\infty}$) for hydrogen particles in target gases of H_2, He, O_2, N_2, Ar, H_2O, CH_4, and C_2H_6 in the energy range 100 to 550 kev. Excellent agreement exists between the present results and previous results for gases of H_2, He, N_2, and Ar. The energy range is being extended to 3 Mev, along with the additional measurement of the electron capture cross section (σ_{10}).

Development has continued on the secondary electron detector as a quantitative detector of low-energy neutral particles escaping from a plasma. Detailed investigation of the statistics of the secondary electron distribution revealed that previous measurements failed to include all the secondaries emitted. Increasing the area of the silicon barrier detector to 200 mm^2 apparently results in 100% collection. Placing a high-resolution surface-barrier detector in the target position permits the determination of the probability of zero electron emission for incident H^0 particles in the energy range 10 to 50 kev. This probability is 0 ± 2% throughout this energy range. The electron distribution of an AgMg target approximately fits a Poisson distribution with departure occurring for groups of electrons greater than eight.

BIOLOGY AND MEDICINE

Body Fluid Analysis. — An automatic high-resolution analyzer capable of quantitatively determining ultraviolet-absorbing molecular constituents of urine is being developed for use as a clinical tool and for biochemical research. This analyzer, which is a modification of a previously developed nucleotide analyzer, uses a separations system based on anion exchange; the detection device is a recording spectrophotometer operating at four wavelengths. It has separated up to 107 ultraviolet-absorbing urine constituents, of which 11 have been tentatively identified. The results are reproducible.

The resolution of the anion exchange separations column has been increased by using 5- to 10-μ-diam resin, and the time required for a single urine analysis has been decreased to about 40 hr by using a high-pressure flow system ($>$1500 psi).

The system has shown differences between normal urine and urine from individuals having acute leukemia. Other abnormal urine specimens are also being analyzed.

SOMATIC EFFECTS OF RADIATION

Statistical Services and Research. — The fifth in a series of experiments designed to investigate the mortality from secondary disease in irradiated mice treated with foreign bone marrow is now being performed. The experimental design being used is a central composite design, the purpose of which is to approximate the point in the factor space which will result in the lowest mortality of mice treated with rat bone marrow. Designs for further exploration of the response surface are now being considered.

Pathology and Physiology. — The estrous cycle in RF and (101 × C3H)F_1 mice was studied by cytological, histological, histochemical, and autoradiographic methods. Mucous cells were seen to differentiate in the vaginal epithelium throughout the stage of diestrus, suggesting that this stage should properly be considered a part of proestrus. Hence a new histological classification was proposed, in which the estrous cycle was divided into three stages: proestrus (stage of mucification), estrus (stage of cornification), and metestrus (stage of sloughing). The use of these terms to classify the stages of the estrous cycle enabled the cyclic changes in vaginal cytology to be correlated more closely with the corresponding histological appearance of the vagina, uterus, and ovary and the functional relationships among these organs.

The mitotic index and uptake of tritiated thymidine in the vaginal epithelium were highest in basal cells, and varied with the stage of the estrous cycle, being maximal late in proestrus and early estrus. The appearance of tracer in Malpighian cells occurred later, consistent with the interpretation that these cells are derived from basal cells. Mitosis and uptake of tritiated thymidine were observed in a small percentage of vaginal mucous cells, indicating that differentiation in these cells does not necessarily involve loss of the ability to synthesize DNA.

Medium(Low)-Level Long-Term Effects of Radiation. — A data processing system that has met our needs for accurate, rapid, and efficient methods of handling data from our mouse breeder colony has been developed to replace our handwritten cards and ledgers. The system includes electromechanical recording equipment (IBM 357 Data Collection System) and several computer programs. It offers the following advantages: (1) recording of data in their final form at the site of observation by the original observer, (2) automatic preparation of data in a form acceptable for computer input, (3) enforcement of a standardized routine for recording, (4) elimination of the need to read poor handwriting or to decipher abbreviations, (5) minimizing the lag between recording and use of data. The computer programs translate data from punched cards to magnetic tape; file data under the appropriate mouse or cage record; print weekly lists of mice to be weaned, mated, retired, or prepared for an experiment; list various recording errors; provide printed and punched cage or identification cards; and print various lists containing ordered information about the breeding colony, its production, and its operation. The system allows continuity of recording and analysis regardless of personnel changes. It processes data rapidly enough so that they aid in providing continuous supervisory control of breeding operations and help to detect changes in the health status of the colony.

Influence of Microbial Environment on Radiation Pathology. — The life-span and incidence of diseases have been studied in unirradiated and x-irradiated (300 r at five to six weeks of age) conventional and germfree RFM/Un and ICR mice. Preliminary data suggest that the mortality rate is lower in both irradiated and unirradiated germfree mice than in their conventional counterparts over the first two-thirds of the life-span and that such differences tend to diminish and may disappear in the last third of life. The germfree mice of both strains have resembled the conventional mice in the development of lymphatic leukemia, reticulum cell sarcoma, and solid tumors of various sites; however, they have failed to develop myeloid leukemia, which is a characteristic radiation response of the conventional RFM mouse. Both irradiated and unirradiated germfree mice have shown glomerulosclerosis, although the incidence and severity of the disease appear to be lower than in the conventional mice. Miscellaneous lesions (arteriosclerosis, amyloidosis, auricular thrombosis, pyelonephritis, and polyarteritis) have been seen in the germfree, as well as the conventional, mice of both strains, but inflammatory lesions associated with infectious microorganisms (such as otitis media, abscesses, and ulcerative dermatitis) have been seen only in the conventional mice. Cecal volvulus, apparently associated with the large cecum of germfree mice, has been noted only in the germfree ICR mice. The general similarity of widely diverse pathological lesions between germfree and conventional mice suggests that the absence of a detectable microbial flora and fauna alters aging processes and late somatic effects of radiation relatively little. However, a few notable exceptions have been observed, one being the absence of myeloid leukemia in germfree mice. These exceptions warrant further study.

Physiology of Blood Platelets. — The distribution of ^{35}S-labeled platelets transfused into irradiated and control rats is being studied by autoradiography of sections of tissues taken from the experimental

animals. Preliminary results indicate that a large amount of the radioactivity is distributed within the tissue of the spleen, largely the red pulp, within 30 min after transfusion of the labeled platelets. Although quantitative measurements have not yet been made, it appears that the radioactivity is actually greater at 1 hr after platelet transfusion in the red pulp of the spleen than in the large blood vessels that course through the spleen. Liver and lung, on the other hand, were almost devoid of activity as ascertained in autoradiographs. By 24 hr the radioactivity level in the spleen had declined somewhat. These results will be useful in helping to answer questions about pooling of platelets in the spleen after transfusion. There is some indication that transfused platelets tend to accumulate in the spleen immediately after transfusion and then are released again into the circulation some hours later. The results described here are consistent with this concept, and further study of our material should help to clarify the nature and extent of the pooling and release, as well as the nature of any differences in these parameters that may occur between untreated and irradiated subjects.

Virology of Radiation Oncogenesis. — Several studies on the biological activity and the morphology of the Rauscher murine leukemia virus have been reported. To date, quantitative studies have been relatively unsuccessful because of the lack of stable, infected, and control tissue culture cell lines. Recently, however, we have propagated Rauscher virus in cells in two different media; in one (NCTC109), the infected cells appeared unaltered; in the second (4XBME), the infected cells became altered, exhibiting a cytopathogenic effect. Observations with the electron microscope confirm the vacuolization of the cytoplasm and the presence of large inclusion bodies in the cells grown in 4XBME medium. Budding of the virus from the periphery of the cells has been observed in both media. Intracytoplasmic inclusion bodies in these same cells frequently appear to contain mature viral particles, some of which look degenerate. Studies with dense tracer materials suggest that these bodies are the results of phagocytosis of cell debris and virus particles. It is suggested that a dynamic equilibrium with respect to virus titer may be set up in these cultures; that is, the same cells which are infected and proliferate virus also phagocytose the virus from the exterior environment. Biological assay of tissue culture fluid from these preparations might lead to false conclusions regarding quantitation of virus synthesis.

Clinical Microbiology and Biochemistry. — Separation procedures employing vertical flat-bed discontinuous electrophoresis in acrylamide gel were used to separate sheep erythropoietin purified to stage III by the method of Goldwasser and to separate mouse plasma erythropoietin. A two-stage process was employed. Following electrophoretic separation, protein zones were located by one of three techniques: (1) Schlieren optics were used to locate zones by their refractive index. (2) A strip was removed from the acrylamide slab and stained; the bands in the unstained portion of the slab were then located by Rf measurements. (3) The nonspecific esterases were located by using α-naphthol butyrate as substrate and fast blue RR as the diazonium salt. The protein zones were then excised from the gel slab, and the proteins were eluted therefrom electrophoretically into a Tris-glycine buffer at pH 8.9. Erythropoietic activity was found in only one protein zone of the stage III

purified sheep material. This zone migrated similarly to an alpha-1-globulin and was shown to be a glycoprotein by virtue of the Schiff reaction. This electrophoretically pure material had an $S20_1$ W coefficient of 3.45×10^{-13} and an approximate molecular weight of 40,000 to 50,000. Plasma from C57 Bl/6J mice treated with phenylhydrazine, containing 2.4 units of erythropoietin per milliliter, was processed similarly, and its erythropoietic activity was found to be associated with the band 6 esterase. The concentration of this esterase has previously been shown to be testosterone dependent, and the material has been found to be a glycoprotein. The eluted protein of band 6 contained 0.4 unit of activity for 51 μg of protein, or a specific activity of 7.9 units of erythropoietin per milligram of protein. A 68.4% recovery of the initial activity of the phenylhydrazine-treated mouse plasma was obtained in the band 6 eluate.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — In collaborative work with the National Cancer Institute it was observed that the Rauscher virus causes massive changes in spleen white pulp of the mouse before leukemia develops. It is suggested from these studies that this agent (or agents) produces malignant germinal center changes that become leukemic.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation. — The energy resolution of several silicon diode detectors operating in the avalanche mode was measured for low-energy x rays and for light input from scintillation crystals. The energy resolution using one of the best diodes to detect 5.9-kev ^{55}Fe x rays was 3.6 kev full width at half maximum when the diode temperature was +25°C and 1.8 kev fwhm when the diode temperature was −50°C. The avalanche gain of the diode was about 4 for the 1.8-kev resolution. When the diode was used in conjunction with a CsI(Tl) scintillation crystal, the room-temperature resolution for 5.3-Mev alpha particles from ^{241}Am was 11% at an avalanche gain of 12. The resolution and avalanche gain for a temperature of −50°C were 9% and 11 respectively.

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