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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JULY 1965

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JULY 1965

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

EGCR Charge Machine. — Tests are being run on the actuating cylinder of the EGCR charge machine in an effort to develop a piston ring that will operate unlubricated with low leakage in high-temperature helium. In the first series of tests, Viton B–carbon wool O-rings were subjected to 50 cycles, with each cycle consisting of 64 single strokes at room temperature and 22 single strokes at 350°F. The results indicated that the leakage was approximately three times greater than desired; however, the mechanical efficiency remained constant during the test period. Additional tests are planned in which two piston rings will be used in modified piston-ring grooves.

EGCR Rod Ejection Transient. — The safety studies for the EGCR have all been based on a space-independent approximation to the neutron kinetics equations. Techniques recently developed for analysis of pulsed-source transients have now been applied to the rod ejection transient as a check for the presence of space-dependent effects not allowed for in the previous studies. In the case studied the reactor was assumed to be made critical with the central control rod fully inserted. This control rod was then instantly ejected to make the reactor supercritical with reactivity equal to 0.012, and the resulting flux transient was studied at different positions in the core. With the exception of a region in the immediate vicinity of the ejected control rod, it was found that the transient was very well approximated by the space-independent calculation. The flux differences near the ejected rod are of minor importance and in such a direction that the space-independent calculation should slightly overestimate the maximum temperatures reached in the transient. Thus the space-independent approximation appears to be satisfactory for analysis of such transients in the EGCR.

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HTGR Analysis. — We studied the effect of the release of volatile fission products on the performance of a 1000-Mw (electrical) General Atomic TARGET reactor. This high-temperature gas-cooled reactor (HTGR) is moderated by a combination of graphite and BeO, cooled by helium, and operated on a thorium-uranium fuel cycle with recycle of ^{233}U. Three limiting cases were compared: (1) retention of all fission products in the fuel, (2) complete release and removal from the core of all isotopes of xenon, krypton, and iodine, and (3) complete release of these fission products and also of the ^{6}Li formed by the Be(n,α) reaction. Results were obtained for several fuel-to-moderator ratios. Minimum fuel doubling times and the corresponding breeding ratios are listed below for two different thorium concentrations, as indicated by the beryllium-to-thorium atomic ratios; the carbon-to-beryllium atomic ratio in all cases was about 2.4.

	Fission Products Retained	Xe, Kr, and I Released	Xe, Kr, I, and ^{6}Li Released
Be/Th = 32			
Maximum breeding ratio	1.06	1.10	1.11
Minimum doubling time, years	53	25	23
Be/Th = 24			
Maximum breeding ratio	1.07	1.11	
Minimum doubling time, years	63	35	

Fuel-cycle costs were also calculated on the basis of the following economic ground rules:

Interest at 10% per year

Reprocessing and shipping costs for a 15,000-Mw (electrical) industry

Fabrication costs of $50 and $100 per kg

Uranium ore costs of $8.00 and $20.00 per lb of U_3O_8

UF_6 conversion cost of $2.70 per kg of uranium

Separative work cost of $30.00 per kg of uranium

Holdup time for fabrication and reprocessing of 60 days

The low figures for fabrication cost and holdup time and the high figure for separative work cost were purposely chosen in an effort to determine whether any conditions could exist that would favor the operation

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of an HTGR as a breeder. The results are listed below for minimum fuel-cycle cost under each of the above assumptions:

	Fabrication: $100/kg		Fabrication: $50/kg	
	Ore: $8/lb	Ore: $20/lb	Ore: $8/lb	Ore: $20/lb
Release of Xe, Kr, and I				
Burnup, Mwd/ton	55,000	50,00	50,000	42,000
Conversion ratio	1.012	1.022	1.022	1.036
Fuel-cycle cost, mills/kwhr (electrical)	0.70	0.85	0.59	0.72
Release of Xe, Kr, I, and Li				
Burnup, Mwd/ton	75,000	71,000	67,000	54,000
Conversion ratio	1.020	1.027	1.033	1.050
Fuel-cycle cost, mills/kwhr (electrical)	0.62	0.75	0.53	0.65

Phase Relations in the Pseudobinary System UC_2-ThC_2. — We completed studies of the phase relations in the UC_2-ThC_2 pseudobinary system; drop-cast and machined specimens were used. The microstructural features of all as-cast alloys were similar, and all contained excess graphite. Only when specimens were annealed above 1500°C for extended periods of time were there significant changes in the microstructure of the carbide or the morphology of the graphite. Microhardness tended to increase slowly with annealing time at 1900°C but did not change significantly for anneals at lower temperatures.

Powder x-ray diffraction patterns were generally diffuse and did not improve appreciably with annealing for material that contained much excess graphite. Specimens of ThC_2 that contained only a trace of free graphite gave very sharp diffraction patterns after annealing at 1400°C and below. X-ray diffraction revealed that two distinctly different body-centered tetragonal structures coexisted for alloys in the composition range from 35 to 42 mole % ThC_2.

Dilatometry and thermal analysis clearly showed two phase transformations in ThC_2 (1500 ± 25°C and 1430 ± 10°C), and the results indicated that the higher-temperature transformation was diffusionless. Two similar transformations were observed for ThC_2-rich alloys containing up to 25 mole % UC_2. One phase transformation was observed for UC_2 at 1768 ± 25°C. One solid-state transformation occurred for the $(U,Th)C_2$ alloys

in the composition range from 25 to 75 mole % ThC_2. The transformation temperatures in this composition range passed through a minimum (~1100°C) at ~46 mole % ThC_2.

Mathematical Model for Predicting Coated-Particle Behavior. — We developed a mathematical model for predicting stresses and strains in pyrolytic-carbon coatings surrounding fuel microspheres under irradiation. These stresses arise from differential thermal expansion of the fuel particle against the coating, fuel swelling, fission-gas release, damage from recoils, and anisotropic dimensional changes in pyrolytic carbon resulting from thermal expansion and fast-neutron damage. With this model, we can evaluate the relative merits of low- or high-density fuel particles, single- or multiple-layer coatings, and low or high values for coating thickness, density, and strength.

We made calculations with this model to investigate the effects of configuration and environment on the expected service life of coated particles typical of those of interest in current reactor technology. Conditions investigated included fuel particle radius (100 μ), single- and two-layer coatings (total thicknesses from 100 to 200 μ), inner-coating porosity (15 to 50%), fuel material (UO_2 and UC_2 with varying enrichments), fuel density (90 and 100% of theoretical), irradiation temperature (250 to 1450°C), fast-neutron doses (0 to 4×10^{21} neutrons/cm^2), fuel burnup (0 to 50 at. %), and fission-gas release from fuel (1 and 100%). The results of these calculations led us to the following predictions.

1. Two distinct modes of coating failure may occur: (a) failure initiated at the inner surface from combined effects of fuel-particle swelling and fission-gas pressure and (b) failure initiated at the outer surface from anisotropic thermal expansion and fast-neutron damage in the pyrolytic carbon.

2. For enriched-uranium-based fuel particles operating to high burnup, failure will normally occur by the first mode given above. To ensure adequate coating life, a free volume consisting of 2 to 3% of the fuel volume should be provided per atom percent burnup expected. Free volume may be obtained by use of a low-density fuel, a porous inner coating that densifies rapidly when subjected to fission-recoil bombardment, manufacturing techniques that provide empty space between fuel and coating, or a combination of these.

3. For fuels with low uranium enrichment or high thorium content, failure may occur by the second mode given above, that is, from fast-neutron damage in the coating before fuel swelling or fission-gas pressure becomes significant. Hence, there is an incentive to use isotropic outer coatings in such cases; however, free volume must still be provided in accordance with the burnup expected.

4. If the inner surface of the outer coating is adequately protected against fission recoils, increasing thickness of the outer coating beyond the level at which the ratio of its inner to its outer diameter is 2/3 will not appreciably increase its life.

5. The release of gaseous fission products from the fuel has a minor effect on coating life so long as adequate free volume is provided (see 2 above) and the coating rupture stress is above about 20,000 psi.

Hence, the use of a low-density fuel particle to help provide free volume should have little adverse effect on coated-particle life, in spite of attendant higher fission-gas release rates.

GCR-ORR Loop 1 Irradiations. — Experimental fuel assembly 15 was installed in the test section of loop 1 on July 8, 1965. The fueled portion of the assembly is fully retracted into a region of low neutron flux during performance tests on the new coolant cleanup system and will be inserted into the high-flux region during the next ORR shutdown, which is scheduled for August 2, 1965.

Initial experience with the coolant cleanup system indicates that it is operating satisfactorily. After four days of loop operation at low temperatures, the total level of impurities in the helium coolant was less than 30 ppm; from previous experience several hundred ppm would have been expected with no cleanup system. Additional tests will be made with the loop operating at elevated temperature.

Experimental assembly 14 was removed from the loop May 2, 1965, and is being examined.

LITR Capsule Irradiations. — Capsule L-CP9 was removed from LITR core position C-42 on July 13, 1965. This capsule contained pyrolytic-carbon-coated UC_2 particles in each of four compartments and had operated at indicated central fuel temperatures of 1630 to 1970°F. Estimated burnups reached approximately 21 at. % of heavy metal. Capsule L-CP10, which is similar to L-CP9, continued to operate satisfactorily in the LITR C-46 position. The estimated burnup reached 9.8 at. % of heavy metal, and the central fuel temperatures are 1490 to 1750°F.

ORR Poolside Irradiations. — Capsule 04-8 containing EGCR-vendor fuel material reached an estimated burnup of 5345 Mwd per ton of UO_2 and is continuing to operate satisfactorily. The maximum measured cladding temperature is 1543°F.

BeO-Graphite Compatibility Experiment. — The Mark II assembly for the BeO-graphite compatibility irradiation experiment continued to operate satisfactorily at 1500°C. However, this temperature can now be maintained only with pure argon as the sweep gas, and we believe that the heat-reflective baffles are becoming less effective.

A carbon monoxide analyzer of the infrared type was installed for measuring the CO content of the effluent sweep gas. Operation of the analyzer has been somewhat erratic, but preliminary results indicate that the effluent contains less than 10 ppm CO.

The neutron flux exposure of this experiment is determined by monitor wires that are exposed with the experimental assembly from time to time — normally for about two weeks of each reactor cycle. Based on the average of neutron fluxes calculated from nickel wires exposed to date, we estimate that the Mark II capsule has now received 7×10^{20} neutrons/cm^2 at neutron energies above 0.18 Mev.

High-Burnup Coated-Particle Irradiation Experiment. — An irradiation experiment is being conducted to evaluate the performance of pyrolytic-carbon-coated fuel particles at high burnup under conditions of interest to the TARGET reactor development program. The irradiation capsule

contains loose UC_2 and sol-gel UO_2 particles in separate graphite compartments. It is desired that the fuel be maintained at temperatures between 1000 and 1400°C throughout the irradiation, that burnup exceed 50 at. % of heavy metal in one year, and that the burnup rate not exceed 10 at. % of heavy metal per month. The capsule is designed so that most of the heat generation is from gamma heating in order to minimize the effect of fuel burnup on temperature. The experimental assembly is being irradiated intermittently in the ORR F-9 facility when it is not required for fuel meltdown experiments.

Irradiation began on July 13. Initial operation with pure helium sweep gas indicated that the fuel temperature was slightly below that predicted by the design calculations. However, with a sweep gas of pure neon, the design fuel temperature of 1400°C was attained with the capsule retracted 2-1/2 in. from the position of maximum neutron flux. The burnup rate at this point is approximately 10 at. % heavy metal per month, and therefore the capsule will not be inserted further until some of the fuel has been consumed.

REACTOR FUELS AND MATERIALS

Nondestructive Test Development. — We have successfully developed and applied ultrasonic techniques for the detection of defects in tapered nuclear heat-exchanger tubing (i.e., tubing with a diameter constantly varying along its length). Ultrasonic inspection of tubing requires the sound beam to impinge upon the tubing surface at a constant angle of incidence. To obtain this condition in tapered tubing, we designed and built a spring-loaded system to adjust the tube to a constant point of tangency within large-diameter bushings. Thus the proper ultrasonic orientation was maintained despite the variation in diameter as a longitudinal scan was made. Associated circuitry provided an audible alarm and a "B" scan presentation to denote the presence of discontinuities. This system has been applied successfully to the inspection of both 0.009- and 0.050-in.-wall tapered tubing. Sensitivity to discontinuities 0.0005 in. in depth was demonstrated for the thin-wall tubing.

POWER REACTOR FUEL PROCESSING

Shear-Leach Process Development. — The principal investigations in the shear-leach program during the past two months were an evaluation of the feasibility of shearing intact second-generation fuel assemblies in the ORNL 250-ton prototype shear, with only the massive end hardware removed from the assemblies, and an evaluation of the safety aspects of handling and disposing of Zircaloy-2 particles produced by wet and dry abrasive-disk sawing of prototype zirconium fuel assemblies.

Three prototype fuel assemblies, with the end hardware removed, were successfully sheared into 1-in. lengths in the prototype shear. Dresden-II fuel (casing or sheath is normally removed at the reactor; about 4 × 4 in.; 36 Zircaloy tubes) and Elk River (no sheath is used; about 3.5 × 3.5

in.; 25 stainless steel tubes) prototypes were the most easily sheared. A Consolidated Edison core B prototype (sheath cannot be removed; 6-1/4 × 6-1/4 in.; 196 stainless steel tubes) was the most difficult to shear. The sheath section of the core B prototype was not completely severed in initial cuts but was successfully sheared after (1) the original 0.020-in. blade clearance was decreased to 0.002 in., (2) the blade was reversed to present a new cutting edge, and (3) the gagging pressure was increased five to six times. The shearing force varied from 73 to 114 tons. From these tests it appears that typical second-generation fuel assemblies can be sheared intact if the massive end hardware is removed beforehand. The maximum cross section of a fuel assembly that can be sheared intact by the ORNL prototype shear appears to be 6-1/4 × 6-1/4 in., such as that of the Consolidated Edison core B assembly.

Zircaloy-2 particles produced by dry and wet abrasive-disk sawing of solid rod stock were examined to determine whether such particles constitute a safety hazard when handled. Initial conclusions are: (1) Dry-cut particles in the range 150 μ and smaller were nearly all converted to the oxide and thus were not hazardous. This is somewhat less true for particles produced by wet cutting. (2) Dilution of the Zircaloy-2 particles by particles from the cutting wheel varied from 4:1 for wet cutting to as much as 7:1 for dry cutting. A ratio of 1:1 has been reported as sufficient to render the Zircaloy-2 particles safe to handle. (3) All Zircaloy-2 particles, regardless of size, appeared to be oxidized and to be readily attacked with a solution consisting of about 3 M HNO_3 and 0.2 M HF.

Fluidized-Bed Volatility Pilot Plant (FBVPP). — Design of the FBVPP is continuing. This pilot plant will be installed in Building 3019 and will be used to study the processing of zirconium- and stainless-steel-clad UO_2 power reactor fuel elements.

Fabrication drawings of the primary and hydrolyzer fluidized-bed reactors, and of their primary and backup filters, are almost complete. The interstage and recycle coolers for the fluorine recycle pump are being designed. Location of equipment in the head-end cell has been reasonably well established by means of a model. Concepts of a method for charging fuel elements to the primary reactor are being developed; drop tests indicated that fuel elements could be dropped onto an expanded bed of Al_2O_3, but present plans call for lowering the element. Work on design of instrumentation has begun.

A network diagram covering the design portion of the job is being prepared for use in scheduling by the critical-path method. Costs will be collected and reported by a related computer program. The cost of the entire project was reestimated on the basis of present, incomplete information.

A process flowsheet is being prepared for the plutonium-uranium separation and purification portion of the facility.

Personnel at Battelle Memorial Institute (working under a subcontract) have been compiling data on the corrosion expected in the fluidized-bed volatility process. A preliminary draft of their report was distributed for comments by those who had provided original data.

The equipment in cells 1 and 2, Building 3019, used for studying the molten-salt fluoride volatility process, is being decontaminated prior to

removal. Pulse-echo measurements of dissolver-wall thickness were completed. Metal loss during the aluminum program was about 5 mils. This represents total corrosion during ten dissolutions and the major portion of aqueous decontamination. The general radioactivity level in cell 1 has been reduced to about 10 r/hr. A survey showed an appreciable pocket of radioactivity remaining near the bottom of the dissolver. Hence decontamination of this portion of the system has been resumed.

Processes for HTGR Fuel. — The burn-leach process, which is applicable to graphite fuels containing carbon-coated thorium-uranium carbide and/or oxide particles, consists in burning the fuel with oxygen in a fluidized bed of inert alumina followed by leaching the resultant bed with fluoride-catalyzed nitric acid to recover the uranium and thorium. Purification and final recovery of the uranium and thorium are achieved by conventional solvent extraction methods. Treatment of stainless-steel- and zirconium-clad UO_2 fuels with $HF-O_2$ mixtures at 600 to 650°C in a fluidized bed of alumina results (1) in conversion of the cladding to a powdered oxide and (2) in conversion of the UO_2 to U_3O_8 powder. These powders are dispersed throughout the alumina. After the $HF-O_2$ step, the bed contains 5 to 10% fluorine, as UO_2F_2 and "stainless steel" or zirconium fluorides. At least 90% of the fluorine can be removed by pyrohydrolysis at 600°C. The bed, which is now nearly free of fluorine, is leached with nitric acid to recover the uranium in preparation for solvent extraction.

Fluoride Volatility Processing: Molten-Drop Fluorination Tests. — The fluorination of uranium and plutonium from falling droplets of molten salt was studied to determine the feasibility of using a spray tower as a contactor in the molten-salt fluoride volatility process. Use of a continuous-gas-phase contactor of this type results in more rapid volatilization of uranium (and plutonium with low-enrichment fuels) and lower corrosion rates than use of a molten-salt fluorinator operated batchwise. The uranium volatilization results for six salt blends were successfully correlated by an empirical equation, and the constants of this equation were found to be related only to the liquidus temperatures of the salt blends. These blends consisted of various mixtures of NaF, ZrF_4, LiF, BeF_2, and UF_4. The plutonium volatilization studies were made with mixtures of PuF_3 in 50-50 mole % $NaF-ZrF_4$. Results indicated that volatilization of plutonium is controlled by diffusion of the plutonium to the surface of the drop. Assuming a barren salt equimolar in NaF and ZrF_4, the necessary conditions were calculated for 99 and 99.9% plutonium removals. For example, use of a 20-ft-high tower would result in removal of much more than 99.9% of the plutonium from droplets 58 μ in diameter at fluorination temperatures of 600°C.

To explore the possibility of using a falling-drop tower in reprocessing molten-salt reactor fuel, falling-drop experiments were made with salt simulating the reactor fuel and with salt containing protactinium. Experiments confirmed the ability of the falling-drop method to remove uranium from simulated molten-salt reactor fuel. Starting with a fuel containing 0.942 wt % (0.395 mole %) uranium in $2LiF \cdot BeF_2$, the best experimental removal at 575°C with a fluorination column 52 in. long was 99.1% for droplets 105 to 125 μ in diameter. The fluorination time for

these droplets was 3.2 sec. The results for this salt fit the previously developed correlation equation, with the constants for the equation about the same as those for another salt (31.7-31.7-31.7-5 mole % $NaF-LiF-ZrF_4-UF_4$) with the same liquidus temperature (about 480°C). Uranium is volatilized more rapidly from these low-melting salts than from higher-melting ones.

In the protactinium experiments, a salt containing 0.3 mg of ^{231}Pa per gram of 50-50 mole % $NaF-ZrF_4$ was used. No volatilization at either 547 or 615°C was detected by gamma counting. Hence, the falling-drop method, used with MSR salt, would remove nearly all the uranium but would leave the protactinium.

Fluoride Volatility Processing: UF_6-NbF_5 Vapor-Liquid Equilibria. — In fluoride volatility processes, one proposed method for separating UF_6 from other volatile fluorides is fractional distillation. Knowledge of the vapor-liquid equilibria of mixtures of UF_6 and the other volatile fluorides is desirable to aid in design of the distillation equipment. The vapor-liquid equilibria of UF_6 and NbF_5 (bp 233°C) are being studied.

The dependence of total pressure of the liquid composition of UF_6-NbF_5 mixtures at 150°C was determined, and a positive deviation from Raoult's law was found. With the assumption that Dalton's law of additive partial pressure holds, the data were empirically fit with the Scatchard-Hamer equation [J. Am. Chem. Soc. 57, 1805 (1935)], and the activity coefficients of UF_6 and NbF_5 were determined for the entire composition range. The ratio of activity coefficients of UF_6 to those of NbF_5 varies from 3.6 at 1 mole % UF_6 in the liquid to 0.18 at 99 mole % UF_6.

An isobaric equilibrium still is now being operated, and the boiling points of various mixtures of UF_6 and NbF_5, as well as the composition of the vapor phase in equilibrium with the mixture, are being determined at 5000 mm Hg total pressure. Both the isothermal and the isobaric data obtained thus far indicate that separation of UF_6 from NbF_5 by fractional distillation is feasible. A minimum of five theoretical stages at total reflux will result in overhead and bottom concentrations of 10 ppm of heavy and light components respectively.

Fluoride Volatility Processing: Chromium Fluoride Studies. — Several volatile fluorides and oxyfluorides of chromium may be formed in processing stainless-steel-clad UO_2 fuel by the fluidized-bed volatility procedure. The studies being reported are part of an attempt to determine the as-yet unknown physical and chemical characteristics of some of these compounds.

In a preliminary investigation of the properties of the higher chromium fluorides, methods were sought for the preparation of laboratory-scale quantities of the materials. Estimates of the free energy of formation of the compounds indicated that of the reactions

$$AgF + CrF_3 \rightarrow CrF_4 + Ag\ , \qquad +17\ \text{kcal}\ ,$$

$$\frac{1}{2}\,AgF_2 + CrF_3 \rightarrow CrF_4 + \frac{1}{2}\,Ag\ , \qquad -7\ \text{kcal}\ ,$$

$$AgF_2 + CrF_3 \rightarrow CrF_4 + AgF\ , \qquad -29\ \text{kcal}\ ,$$

the last two might be used for synthesizing CrF_4. In initial laboratory tests, we found that CrF_4 could be formed above 400°C, the boiling point of the compound, when equimolar amounts of the reactants were present or when there was an excess of AgF_2. The formation of CrF_4 was easily detected by the evolution of dense, blue-black vapor.

Fluoride Volatility Processing: Fluidized-Bed Method for Stainless-Steel-Clad UO_2 Fuel. — Studies of the proposed fluidized-bed process for stainless-steel-clad UO_2 power reactor fuel containing plutonium were continued. The studies consisted of simulated flowsheet tests carried out in 1/2-in.-OD mini-bed reactors, each containing a 5-g bed of alumina (Norton RR, 120 mesh), 500 mg of 20% PuO_2–UO_2, and 0.3 g of stainless steel (type 304L). Oxidation of the fuel charge was carried out by passing a mixture of HF-O_2 through the bed at 650°C for 2 hr. Plutonium and uranium were then volatilized as the hexafluorides at 450°C in 1-hr fluorinations to determine relative differences in retentions on the bed. In one test, in which stainless steel was omitted from the oxidation cycle, 21.9% of the plutonium was retained on the bed. This was not significantly different from the 21.0% retention observed in a test where stainless steel was present, indicating that the decladding products do not contribute appreciably to the retention. In another test, changing the ratio of HF to O_2 in the decladding cycle from 40-60 vol % to 20-80 vol % HF-O_2 resulted in the lesser value of 16.7% retention. Pretreatment of the alumina with fluorine at 500°C before the oxidation step resulted in an appreciable decrease in plutonium retention. A 5-g bed was heated to 500°C and subjected to fluorine at 300 ml/min (STP) for 1 hr. The bed was cooled, charged with PuO_2-UO_2 and stainless steel, and then oxidized at 650°C for 2 hr. The 1-hr fluorination reduced the plutonium retention to 7.5%. The retention was not significantly different with more extensive pretreatment of the alumina. A 2-hr pretreatment at 600°C resulted in a plutonium retention of 9.6%.

THORIUM UTILIZATION PROGRAM

Fueled-Graphite Development. — Process development work is continuing on methods for preparing fueled graphite. The work includes preparation of microspheres of both oxides and carbides, and of coating them with pyrolytically deposited carbon.

The microsphere preparation equipment is being tested. All craft work on the particle-coating furnace is complete. Calibration of the system flowmeters is under way.

The draft of the hazards analysis for the Coated-Particle Development Laboratory is currently being reviewed by Radiation Safety and Control personnel. Approval to begin operations soon was requested.

Room-temperature experiments in the 3-1/2-in.- and 5-in.-ID entrained-bed Plexiglas models were continued, and the scope was increased to include spherical particles 300 to 1200 μ in diameter and of widely differing densities (2.5, 7.5, and 11.1 g/cm^3). Except for the higher gas flows required to achieve a pressure difference of zero across the gap between the insert and bottom cone, the high-density material can be

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conveyed as uniformly and smoothly as the material investigated before. Static-electricity effects seem to overshadow the results from low-density material (glass beads), and we are seeking means to minimize these effects.

To study the influence of the changes in gas density and viscosity with temperature on entrainment, some experiments were made on a bed of 14.5 g of 1000- to 1190-μ-diam glass beads in air, argon, and helium up to 500°C. The degree of particle entrainment was detected by a microphone-counter system which recorded the number of particles hitting a sensor placed 5 cm above the bed. It was found that this degree of entrainment is constant within a few percent for constant velocities of a given gas in the investigated temperature range, and that the gas velocities for helium and argon are, respectively, 1.7 and 0.8 times the air velocity producing the same degree of entrainment. These ratios are quite different from the ratios of velocities required for fluidization. These results, which confirm the results of our computer program on gas velocities, will facilitate the interpretation of our model studies for the operation of high-temperature entrained-bed columns.

Fabrication of the drum coater components is nearly complete. The components are being assembled in the fabrication shop, where they will be tested before delivery.

Drawings showing modifications to the vacuum chamber into which the drum coater is to be installed are complete. Also, piping and electrical design for the chamber installation is complete. We are awaiting a field-installation schedule for this equipment.

Detailed design of the rotary furnace components is 75% complete and should be finished in early August. Associated piping, electrical work, and instrumentation is about 40% complete. Completion of this portion of the design is also expected in early August.

Fabrication of the internal parts of the 5-in. vacuum coater is complete, and it is being assembled. These parts are to be used in the particle-coating furnace when coatings are to be applied at subatmospheric pressures.

Sintering and Grain Growth of Sol-Gel ThO_2 and $(Th,U)O_2$. — The differential thermal analysis apparatus was recalibrated, using the known heats of decomposition of calcium and magnesium nitrates. The results previously obtained, using the water-loss endotherm at 100°C, were found to be too high. This was probably due to the influence of combined water on the thermal conductivity of the powder. The calibration factors obtained from nitrate decomposition have been extrapolated to the temperature range associated with the surface-energy-release peak, using the known thermal conductivities of the materials in the cell. This procedure yielded a corrected average surface energy for thoria, over the temperature range 700 to 1200°C, of 730 ergs/cm^2. This value is the mean of three determinations, the largest deviation from the mean value being 40 ergs/cm^2. However, the absolute accuracy of the result is estimated to be about ±100 ergs/cm^2.

Edge and corner effects have been calculated to increase the apparent surface energy by about 3%. Allowance was made for this factor in the results reported here.

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Peak profile analysis of the water-loss endotherm supported the belief that reaction rate as a function of temperature might be extracted from the differential thermal analysis peaks. For the water-loss reaction, this procedure gave apparent activation energies in the range 11 to 12 kcal/mole, compared with the expected value of about 10.

Thorium-Uranium Fuel-Cycle Development Facility. — Field construction of this facility is almost 7% complete. ORNL procurement is proceeding, with all deliveries to the contractor expected by the scheduled date. Some difficulties are being encountered in fabrication of pipe fittings, and the shielding door design is proceeding more slowly than hoped. Bids for viewing windows and for master-slave manipulators were received which are within the available funding.

As to the design of process equipment, the in-cell equipment and piping design was continued for the uranium-solution handling equipment and the thorium-uranium sol-preparation equipment. Design of the uranium storage vessel and the sol-blending vessel, as well as their metering vessels, is in progress. Methods for thorium oxide addition to the blend tank and methods for sampling process solutions were tested. Preliminary work was started to develop methods and equipment suitable for oxide transfer and sampling. The tray drive and dumping portion of the sol-dryer design is nearly 30% complete.

Conversion of Sol-Gel Oxide Particles to Carbides. — Conversion studies were made on UO_2 microspheres 100 to 150 μ in diameter. The particles were heated with an equal part, by weight, of lampblack in a single-fin crucible rotating at 44 rpm in an argon flow of about 3 ft^3/hr. In all experiments the particles retained their sphericity, and little or no agglomeration or reaction bonding was observed.

Complete conversion was obtained (according to metallography) after 2 hr at 2000°C and after 4 hr at 1900°C. Conversion was not complete after 3 hr at 1800°C. Voids were observed in most of the particles, regardless of conversion temperature, and they were largest and most abundant in the 2000°C conversion. The number and size of the voids decreased with decreasing conversion temperature. The voids may be related to the initial carbon content, which was 0.6 wt % (about 12 mole %), since part of any carbon initially present in the oxide would be removed as CO during conversion to the carbide. In this connection, wide variations in density and porosity have been deliberately introduced in ThC_2 and $(Th,U)C_2$ prepared by a sol-gel process where the carbon for the reaction was incorporated in the particle before heating. The present study differs in that the carbon is diffused into oxide particles at high temperatures. The incompletely converted uranium oxide in the particles from the 1800°C conversion was located more or less in the center of the particle, but had an extremely ragged appearance, some spots being isolated from others, unlike the unconverted areas in the similar studies with ThO_2, where the unconverted oxide was generally well consolidated and in the center of the particle.

A repeat conversion run was made with $(Th\text{–}25\%\ U)O_2$ microspheres 177 to 250 μ in diameter, at 2150°C for 2 hr. Again, there were central voids in about half the particles; and numerous small inclusions or spots, presumably another phase, were present in the structure. A conversion run

of this material at 2000°C for 2 hr resulted in fewer voids but more numerous spots in the particles. These spots are too small to be identified metallographically, but x-ray diffraction analysis may reveal some identifying clue.

Uranium-233 Processing Facility. — The solvent extraction plant was operated for 28 hr to purify 7215 g of uranium (91.24% ^{233}U) for shipment. The uranium losses amounted to 0.3%, and the thorium separation factor was 3396. The product, concentrated to 181.5 g of uranium per liter, was withdrawn into 21 polyethylene bottles and packaged in bird cages and shipped. A portion of this material was shipped to the Babcock and Wilcox plant in Lynchburg, Virginia, for use in preparing sol-gel material.

The dissolution of 5950 g of ^{233}U present in the slag (MgO, U_3O_8, metal) received last month was started.

Installation of the solution storage system was completed during the month. Additional piping is 60% complete.

The intermediate-scale solvent extraction facility was moved to its permanent location, and "cold" testing was resumed.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — The preliminary nuclear experiments were concluded, and the reactor was shut down for modifications and preparation for power operation.

A total of 87 enriching capsules were added, bringing the ^{235}U inventory to 76.1 kg. Period measurements with the circulation stopped were analyzed to obtain rod sensitivity. Integration of the observed points gave a worth of 2.18% for one rod, coinciding with the predicted worth. The ^{235}U concentration coefficient of reactivity observed also coincided with the calculated number: 0.25 $(\delta k/k)/(\delta c/c)$. The total temperature coefficient of reactivity between 1150 and 1230°F was observed to be -7.6×10^{-5} $(\delta k/k)/°F$, compared with -7.0×10^{-5} and -9.6×10^{-5} $(\delta k/k)/°F$ predicted with two different coefficients of thermal expansion for the fuel salt. With normal salt level in the fuel pump bowl, a gamma-ray densitometer on a fuel line showed no circulating bubbles, and there was no measurable effect of pressure on reactivity. This was not true when the salt level was lowered to 1.2 in. below normal by reduction of the fuel temperature to 1050°F. When the pump bowl pressure was rapidly reduced from 15 psig to 4 psig, the densitometer and other evidence indicated about 2.5 vol % bubbles in the circulating salt at the higher pressure. A reactivity change of 11×10^{-5} $(\delta k/k)/psi$ was observed, consistent with 6×10^{-5} $(\delta k/k)/psi$ predicted for 1.2 vol % bubbles.

Experimental measurements being analyzed include period measurements with the fuel pump on and dynamics experiments involving reactivity perturbations with the rods and the fuel temperature.

The vapor condensing system was leak tested and connected to the cell exhaust. The original, heat-warped radiator doors were removed and are being replaced with doors of improved design. The anchor sleeves at

the coolant line penetrations of the reactor cell are being revised to reduce thermal stresses. Other maintenance and modifications of a minor nature are also in progress.

Metallurgy. — Studies have been started on the effects of irradiation on the creep properties of Hastelloy N used in building the MSRE. The purpose of the program is twofold: (1) to determine the ductilities of various irradiated heats of Hastelloy N for test conditions of low stresses and strain rates, and (2) to compare and correlate the creep and stress-rupture properties measured in in-reactor tests with those measured in postirradiation tests.

We have irradiated heats Nos. 5065 and 2477 to total neutron doses of 5×10^{16}, 1.3×10^{18}, 9×10^{18}, 5×10^{19}, and 5×10^{20} neutrons/cm^2. The material is being evaluated at four stress levels at 650°C. Our stress-rupture data for heat No. 5065 to date are summarized below:

Neutron Dose (neutrons/cm^2)	Time to Rupture (hr) at:	
	52,571 psi	39,798 psi
0	51.0	350.0
1.3×10^{18}	21.0	147.0
9×10^{18}	5.1	80.2
5×10^{19}	0.43	23.5

The creep ductility in these tests ranged from approximately 0.5 to 8% and decreased with increasing dose. The creep curves were not significantly different in form. The irradiation merely decreased the plastic strain, and therefore the time, at rupture.

Chemical Development. — Fuel salt samples were removed daily from the MSRE pump bowl throughout the zero-power experiments. Chemical-composition, contaminant-level, and isotopic analyses were made on a regular basis. The salient conclusion derived from these analyses is that no corrosion was sustained by the fuel containment circuitry during the ~1100-hr test period. At the end of the zero-power experiment the fuel salt was drained from the fuel circuit and replaced briefly by flush salt. The small amount of uranium subsequently detected in the flush salt (0.019 wt %) indicated that no appreciable holdup of fuel salt occurred in the drain operation.

Radiation Effects. — A prototype model of the proposed in-pile molten-salt experiment has been operated continuously at ~1200°F for 2150 hr to test long-term reliability of the autoclave assembly. Salt circulation, produced by thermal convection, has been essentially constant at a flow rate of ~10 to 15 cc/min. However, occasional loss of

circulation has been caused by accumulation of gas bubbles in the horizontal return line between the cold leg and the autoclave. This is being corrected by changing to an inclined return line.

Detailed design of a beam hole liner, a shield plug, and a sampling and addition system required for operation of the experiment in beam hole HN-1 of the ORR has been completed.

Analysis of Radioactive MSRE Fuels. – The zero-power experiments produced approximately one sample per day for the determination of individual constituents: Li, Be, Zr, U, Fe, Cr, Ni, F^-, and Mo. The samples were received, prepared, and analyzed in the High-Radiation-Level Analytical Facility hot cells. In general, the operations were normal and the results as anticipated. The uranium determinations indicated a negative bias of ~1.5 wt % from theoretical value based on additions of enriched fuel.

The bias, if determined real, will be investigated for cause and possible methods of correction.

The new apparatus for the determination of oxides in MSRE fuel by the hydrofluorination method has been fabricated and is now being leak checked. Prior to the fabrication it was established that in zirconium-containing melts, unless a temperature of 700°C and an HF-H_2 gas mixture of at least 0.2 atm HF were maintained, it was impossible to remove all the oxide from the melt. When these conditions were observed, the oxide was removed in less than 4 hr, as shown below:

Sample	Oxide Added (mg)	Oxide Recovered (mg)
UO_2	21.4	22.8
	17.6	17.1
	20.7	19.2
ZrO_2	20.2	21.8
	26.0	24.6
	40.2	39.1

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. – The fuel assembly to be used during reactor startup has been completed and delivered to the site. The control over the plate spacing was better by almost a factor of 2 than was required by the specifications. It has now been shown to be possible to machine both annuli to the tight dimensional tolerances specified. The end fittings on critical assembly No. 2 were modified so this element may be used during the low-power and calibration runs of the reactor.

Two complete sets of control-rod components were also completed and delivered to the site. The first set was again a modification of the set

used during the critical experiment, while the second was a new set to be used during initial operation. In spite of the size of these components, approximately 18 in. in diameter and 6 ft long, excellent dimensional control was achieved during plate forming and assembly. The plates were formed using explosive forming techniques and using care to keep the plates under a compressive load. The large cylinder diameter was within the specified ±0.030 in. of an ideal theoretical cylinder.

Component Development and Testing. — The second period of shakedown operation of the HFIR was completed in June. During this period, tests were conducted to determine actual flow rates through each of the reactor component coolant channels to identify and measure vibrations in the reactor components and beam tubes and to determine the operating characteristics of the inner and outer control-rod drive and safety systems. The operating experience gained in this shakedown period indicated some minor modifications to be made on the reactor structural components, the control-rod guidance components, and the control-rod and safety-system components.

The reactor horizontal beam tubes and engineering facility tubes were subjected to thermocycle tests and hydrostatic tests during shakedown operation. During these tests, leakage of some of the Conoseal joints indicated that these joints must be provided with positive alignment during assembly and uniform clamping force around the circumference of each Conoseal flange.

Remote maintenance practice was conducted during the shakedown tests, and changes in components and maintenance procedures have been made as indicated to simplify these operations. Developments were started on remote maintenance procedures for beam tubes and engineering facility tubes.

A program has been completed of hydraulically testing the target region consisting of targets, target tower, target holder, and fuel grid assembly. Flow rate and pressure drop in the various series and parallel flow channels were measured. Individual components and assemblies were tested, and the results were correlated. All experimental work was at room temperature, and the results were extrapolated analytically to operating temperatures. The size of the orifice to the fuel grid was determined to give a target region flow rate of 788 gpm at 120°F inlet temperature and within an overall target region pressure drop of 110 psi. This yields the required 40 fps coolant velocity past the target pins.

Instrumentation and Controls. — Tests of the servo control system were completed using the installed mechanical drive components and instrumentation in conjunction with a simulation of the reactor dynamics by means of an analog computer. Both the level safety instrumentation and the wide-range counting instrumentation were tested from the chambers through to the readout. Performance of both systems was acceptable. The

magnets are now being tailored to obtain the optimum release time—holding force characteristics.

The calibration of the flow Δt and heat power instrumentation of the primary system was continued. Transient studies of the pressure and flow systems were made to obtain the best setting for control and safety parameters.

Preoperational Activities. — The written portion of the reactor operator qualification test has been given to the startup crew, and the oral portion of the test will be given in the near future. Training of the new operators is continuing as rapidly as the field work permits.

The iodine efficiency tests on the off-gas systems have been completed, and the results were satisfactory. Iodine efficiency tests are continuing on the special building hot exhaust system. Some minor modifications may be required to obtain the desired decontamination factors in this system.

During July the reactor was reviewed by both the Advisory Committee on Reactor Safeguards and the Laboratory's Reactor Operations Review Committee. The ACRS gave verbal approval for operation up to a power level of 20 Mw and indicated they would submit through channels some questions which should be answered prior to operation at power levels above 20 Mw. The RORC has not issued its formal report regarding the review; however, no specific problems were revealed during the review.

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PHYSICAL RESEARCH

Reactor Operations. — The ORR operated 88.7% and the LITR 92% of the time during May and June 1965.

A controlled boiling test was run in the ORR by the reduction of flow through an instrumented fuel element. The purpose of this experiment was to record the neutron noise and to analyze this for frequencies characteristic of boiling.

The 240-g fuel elements are being burned to give a range of weights which will permit the reactor to be completely loaded with these elements. The core of 240-g elements will be operated to determine what burnup can be achieved.

A new upper assembly of grids was installed at the LITR, and the No. 1 seat-switch assembly was replaced. This replacement stopped a reactor water leak.

The BSR operated for 137.250 Mwhr, mostly for solid-state research.

The PCA was operated for training ORSORT and University of Tennessee nuclear engineering students.

Waste Disposal. — Release of radioactivity was very low, both in liquid and gaseous systems.

Work is continuing on minor improvements in monitoring and control of liquid and gaseous wastes, and preparations are being made for operation of the new liquid-waste evaporator next winter when construction is completed.

Hot-Cell Operations. — Six fueled capsules were disassembled and examined in detail; included were two capsules containing graphite spheres embedded with coated fuel particles, and four capsules containing coated fuel particles. One UO_2 reactor meltdown experiment was disassembled and examined; the UO_2 meltdown from Nuclear Safety Pilot Plant experiment No. 9 was sampled. Examination of TRU target rod No. 3 was begun; this included diameter and bow measurements, gamma scanning, fission gas sampling, heat generation measurement, and partial disassembly. Sections of TRU target rods 1 and 2 were cut and transferred to Analytical Chemistry for dissolution studies. Other specimens examined included an Army PM-1 prototype fuel tube (dimensional measurements, gamma scanning, dye checking for cracks, and metallography) and one aluminum plate containing Eu_2O_3 and one Ta-Al plate (dimensional and density measurements, defilming, photography, and metallography).

In the ORR cells, specimens were recovered from 18 experiments; these were transferred to other cells for detailed examination. About 100 tensile specimens were annealed, and about 175 tensile specimens were tested. The four in-cell creep machines were in use full time with INOR-8 specimens.

PHYSICS AND MATHEMATICS

Theoretical Physics. — The inelastic cross sections and polarizations for the excitation of some observed 2^+ and 3^- levels of ^{12}C, ^{16}O, and ^{40}Ca

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by 156-Mev protons were calculated in the distorted-wave, impulse approximation, using the particle-hole transition densities of Gillet and collaborators. The results were compared with available experimental data, and good qualitative agreement was obtained: in particular, the particle hole densities provided the necessary enhancements of the cross sections. Fits were also attempted by using the theory of collective excitations. These provide reasonable fits to the cross-section data, but not to the polarization.

The cross section for the inelastic scattering of 156-Mev protons from several light, even-even nuclei has been calculated for the excitation of some strongly excited low-lying states, using the collective theory of excitations. Comparison with experiment produces deformation parameters which are in reasonable agreement with those obtained by other means, provided both the real and imaginary portions of the appropriate volume absorbing optical potential are deformed. Similar treatment of surface absorbing potentials yields cross sections which compare closely in shape to the results for volume potentials, but require unreasonably large deformations. Deformation of only the real part of either type of potential does not yield satisfactory fits.

Low-Energy Neutron Time-of-Flight Spectroscopy. — Simultaneous neutron capture and transmission data were recorded during the 1964 measurements at Rensselaer Polytechnic Institute. The transmission data of the samples of separated tungsten isotopes have now been analyzed for resonance parameters. Twenty-five resonances were observed in ^{182}W up to 1200 ev, and an s-wave strength function of $(2.2 \pm 0.7) \times 10^{-4}$ was obtained. Twenty-two resonances were observed in ^{184}W below 2000 ev, and a strength function of $(2.3 \pm 0.7) \times 10^{-4}$ was obtained. For ^{186}W a total of 26 resonances were detected below 2200 ev, and a strength function of $(1.6 \pm 0.5) \times 10^{-4}$ was obtained. Thirty-one resonances were detected in ^{183}W below 400 ev, but the strength function has not yet been extracted from the data.

The ORNL fast chopper was used to measure the neutron transmission of a tungsten oxide sample enriched to 6.93% in ^{180}W. Six resonances were detected in ^{180}W at energies of 15.9, 49.2, 65.6, 75.0, 87.5, and 230 ev.

Low-Temperature, Nuclear and Solid-State Physics. — Mössbauer studies of the hyperfine structure and of the recoilless fraction have been made on the 77-kv gamma ray of ^{197}Au for alloys containing 1 at. % of gold in copper-nickel. With increasing noble-metal concentration the magnetic field at the gold nucleus is found to decrease. Large quadrupole coupling effects are found in the neighborhood of 10 to 20% noble metal. Progress has been made on a new method of analysis of the experimental data which yields better values for the hyperfine-structure coupling parameters. This method involves a least-squares fit to the experimental gamma-ray line shape in which line broadening due to absorber thickness is taken into account.

High-Voltage Experimental Program: N-D Scattering — Vector Polarization of Recoil Deuterons from a Polarized Neutron Beam. — In N-D scattering two sets of scattering lengths are consistent with the low-energy data: set I, $a_2 = 0.8$ fermi, $a_4 = 6.4$ fermis; set II, $a_2 = 8.3$ fermis, $a_4 = 2.6$ fermis; where a_2 and a_4 are the doublet and quartet lengths respectively. Although current theoretical calculations are converging on set I,

there does not appear to be any unambiguous experimental evidence for either choice. The vector polarization of the scattered neutron and recoil deuteron has been calculated for incident beams of completely polarized neutrons. Low-energy (slow) neutrons produce scattered neutrons with polarizations of 66% (set I) and 65% (set II). On the other hand, the vector polarizations of the recoil deuterons are +50% and −33% respectively. At 350 kev neutron energy the neutron polarizations are +51% (set I) and +49% (set II), while the deuteron vector polarizations are +38% (set I) and −21% (set II). Therefore a determination of the deuteron vector polarization in a triple scattering experiment provides a sensitive test for the proper scattering lengths.

High-Voltage Experimental Program: Neutron Electric Dipole Moment Experiment. – An experiment to detect an electric dipole moment of the neutron is being planned. Very cold neutrons will be used with a split-coil magnetic resonance apparatus. A strong electrostatic field will be applied in the region between the split radio-frequency coils, and we will search for small shifts in the resonance frequency due to the presence of the electrostatic field. The source of neutrons will be the HB-2 beam hole of the LITR. A bent neutron conducting tube immersed in a D_2O moderator will provide a low-pass neutron filter of very large solid angle. The beam-hole liner and the bent tube are being designed, and installation in the LITR is planned before the end of 1965. The requirements for uniformity of the magnetic field, and requirements for the strength and uniformity of the electric field, are quite severe. Models are being made for optimizing the electric- and magnetic-field parameters. We will attempt to have the apparatus for the experiment completed by the time the neutron conducting tube is installed.

CHEMISTRY

Effects of Radiation on Analytical Methods. – Work carried out previously on the effect of gamma radiation on arsenazo III was extended to investigate the effect of ^{60}Co gamma radiation on the determination of zirconium as the zirconium–arsenazo III complex. This complex can be formed in HCl medium, and its sensitivity to radiation was shown to increase as the acid molarity is increased. Irradiations were made of 3, 6, and 9 M HCl solutions containing both reagent alone and reagent-complex. It is known that arsenazo III is readily attacked and destroyed by strong oxidizing agents; so, it is to be expected that formation of chlorine by radiolysis would cause complications. Both arsenazo reagent and zirconium–arsenazo III complex solutions are decolorized in HCl medium when exposed to radiation. The rate of decomposition for both is greater the higher the HCl concentration, and is in line with the rates reported on the formation of chlorine as a function of HCl molarity. A dose of 30,000 rads decreased the absorbance of 9 M solution by 50% and that of 3 M solution by 20%. The sensitivity at 9 M is about four times that at 3 M. A dose below 1000 rads will affect absorbancy readings by less than 3%.

It was reported previously that chlorine formation can be inhibited by the addition of methanol. The addition of methanol up to 5 vol % to

solutions of arsenazo III did not stop the destructive action of irradiation. This seems to indicate that the role of methanol is not to prevent formation of chlorine but rather to remove it once it is formed.

Separations Chemistry Research: Recovery of Macromolecules of Biological Interest. — The investigation of separation methods for the recovery of macromolecules of biological interest is a new field of activity. The first problem undertaken was the recovery and fractionation of transfer ribonucleic acids (t-RNA's) (ORNL-3785). The t-RNA's are critical intermediates in the biological synthesis of proteins from individual amino acids, a different t-RNA being involved in the transport of each of the 20 amino acids to the ribosome, the site of protein synthesis.

A procedure for the recovery of purified, mixed t-RNA's from *E. coli* was developed. It includes a phenol extraction step to recover crude nucleic acids from the cells, an isopropanol fractionation to separate DNA from the RNA, and column chromatography with diethylaminoethyl-cellulose to recover the mixed t-RNA's. The procedure is capable of being scaled up to yield large batches of mixed t-RNA's.

Current work includes the investigation of techniques for fractionating the mixed t-RNA's to obtain highly purified, amino-acid-specific t-RNA's. To this end, a new reverse-phase column chromatographic system was devised which gives greater resolution and is less complicated experimentally than previous fractionation methods. The column consists of a quaternary ammonium extractant, dimethyldilauryl ammonium chloride in isoamyl acetate, supported on hydrophobic diatomaceous earth. By use of sodium chloride gradient elution, 16 amino-acid-specific t-RNA's were identified in the column eluate fractions by means of a bioradiochemical assay. Several t-RNA's appear to be 50 to 80% pure. Experiments are under way to prepare milligram samples of these purified t-RNA's and to obtain a better measure of the actual purity.

Separations Chemistry Research: Synergistic Enhancement of Strontium Extraction. — Earlier studies (ORNL-3543) indicated synergistic enhancement of strontium extraction by di(2-ethylhexyl)phosphoric acid (HA) and sodium di(2-ethylhexyl)phosphate (NaA) by the addition of tri-*n*-butyl phosphate (TBP) or tri-*n*-octyl phosphine oxide (TOPO) in octane or nonane (but not in benzene), and a compound $SrA_2 \cdot 2HA \cdot 2TBP$ was postulated. Further studies of the dependence of the distribution coefficient on pH and on reagent concentration (0.0125 to 0.1 *M* A) show two HA dimers reacting in the presence of 0.2 *M* TBP, $SrA_2 \cdot 2HA \cdot xTBP$. As the TBP concentration is increased to near 3 *M*, the number of HA molecules associated with each SrA_2 decreases to zero, implying increasing replacement by TBP. However, direct attempts to confirm the number of TBP molecules are not yet successful.

Volatility Studies: Sorption of PuF_6 by Metal Fluorides. — A sorption-desorption system for PuF_6, similar to the UF_6-NaF system, would be useful in volatility processes. No sorption-desorption system has been found even after a more exhaustive examination of the fluorides of the group IA and IIA elements. Previously, 21 other metallic fluorides were rejected because the sorption of PuF_6 was too poor. In the more exhaustive testing, 10 mg or less of plutonium as the hexafluoride was passed

with a stream of fluorine through 1 cc of the test sorbent at the test temperature and then into a backup trap of NaF; gas flow of 60 cc/min (STP) was continued for 15 min in each test. Separate tests were conducted at several temperatures between 100 and 700°C.

As to the data presented in the following table, the percentage of plutonium sorbed was calculated from the total collected in the two traps. Only with LiF and BeF_2 was there a pronounced temperature dependence for the plutonium sorbed. The compounds NaF, CaF_2, SrF_2, and BaF_2 appear to be good sorbents. The percentages of plutonium sorbed by KF, RbF, and CsF may be low because of caking and channeling; however, their tendency to cake would preclude their use.

Temperature Dependence for Retention of PuF_6 on Group IA and IIA Metal Fluorides

Sorbent	Percentage of Plutonium Retained at Temperature (°C)						
	100	200	300	400	500	600	700
LiF	29.4	46.8	87.5	88.7	96.9	89.7	90.4
NaF	98.6	99.8	99.6	99.92	99.94	99.97	98.2
KF	97.1	98.5	97.6	97.5	94.2	94.4	98.0
RbF	83.6	90.4	93.8	99.9	99.9		98.5
CsF	99.6	98.6		97.9	99.8		95.8
BeF_2	29.1	20.1	76.6	70.9	71.0	73.4	87.1
MgF_2	98.8	98.2	96.3	96.9	98.6	98.6	94.5
CaF_2	99.99	99.995	99.994	99.997	99.98		99.996
SrF_2	99.95	99.97	99.7	98.9	99.97	99.96	99.97
BaF_2	99.4	99.95	99.95	99.99	99.97	93.93	99.99

Chemical Engineering Research: Aerosol Characterization by Light Scattering. — Development of the electronic analyzing system is complete, and several usable aerosol generators have been built and tested. The response of the system to known aerosols is being tested, and early results show that the analysis section contributes to distortion of the detector signal.

Chemical Engineering Research: Stacked-Clone Contactor Development. — Additional mass-transfer studies were made with the stacked-clone contactor, using the two-phase solvent extraction system composed of hexone and water, with acetic acid as the distributing solute. This system is recognized as one having excellent phase separation properties with resultant high throughputs. Extraction and stripping efficiencies at 75% of flooding averaged 81 and 85% for operation at 25 and 35°C, respectively, and flow ratios (aqueous/organic, A/O) from 0.5 to 0.7. Total

flow rates for these runs were about 3.7 and 4.3 liters/min for the two temperatures.

The performance of the stacked-clone contactor with a viscous aqueous phase was studied by altering the above chemical system to one having an aqueous phase composed of acid-deficient aluminum nitrate, with uranyl nitrate as the distributing solute. Hexone remained as the solvent to take advantage of its favorable flow capacity. This flowsheet has commercial use as the "Hexone Process" for enriched uranium. Flooding rates with this system were 5.9 and 2.8 liters/min (A/O = 3) for 1.0 and 1.7 M $Al(NO_3)_3$, respectively, compared with 7.0 liters/min for the acetic acid–hexone flowsheet. Mass-transfer efficiencies for uranium were about 85% for 1.7 M $Al(NO_3)_3$, 0.2 M acid deficient (viscosity = 4.2 centipoises), at 60% of flooding and a flow ratio of 10.5.

Mechanisms of Separations Processes. – Standard thermodynamic functions pertaining to the extraction of uranyl nitrate and nitric acid by tributyl phosphate (TBP) are being measured or calculated to place our interpretations of these extraction equilibria on a firmer mathematical foundation.

Activities of TBP in TBP-HNO_3-H_2O solutions were calculated by several different procedures, using smoothed and unsmoothed data. All methods included graphical integrations of the Gibbs-Duhem relation for the three-component system. Two different equational representations were integrated by means of the CDC 1604-A digital computer, using smoothed partition data; a third method incorporated original data in one of the equational representations, with evaluation of activity ratios by means of graphical integration. The values obtained by these three methods were averaged to give a table of rational TBP activities for aqueous nitric acid concentrations ranging from 0 to 15 M. Considering the fact that one of the procedures included graphical integration of original data, excellent agreement was observed, with the average maximum deviation from the mean being 1.4% over the complete range of the tabular values.

The extraction of uranyl nitrate and water from aqueous solutions by TBP-organic-diluent solutions has been studied. TBP activity data were obtained for Amsco 125-82 solutions containing 5, 10, 15, 30, and 65% TBP (by volume), in addition to previously reported data on 100% TBP. In all cases the extraction was describable in terms of the ratio of organic uranyl nitrate to TBP concentration ratios. Equilibrium constants were calculated by least-squares analysis of the data, with the standard states being defined as the pure materials for TBP and H_2O and the hypothetical 1 M solutions for uranyl nitrate hexahydrate and the complex $UO_2(NO_3)_2 \cdot 2TBP$. The values and standard deviations are as follows:

Percent TBP	K_2'
100	2.65×10^3 (±2.4%)
65	2.39×10^3 (±3.2%)
30	2.13×10^3 (±1.7%)
15	1.80×10^3 (±1.9%)
10	1.27×10^3 (±2.9%)
5	4.96×10^2 (±1.8%)

Physical Chemistry of Polyelectrolyte Systems: Ion Exchange in Cross-Linked Polystyrene Sulfonates. — Cross-linked organic ion exchangers exhibit many of the properties of concentrated electrolyte solutions, and at the same time they behave like high-molecular-weight polymers. This dual behavior is reflected in the selective uptake of ions by these materials from dilute aqueous solutions, as changes in the ionic interaction free energy and in the configurational free energy of the molecular network of the ion exchanger occur simultaneously in an ion exchange reaction. Usually, the lowering of the interaction free energy is the dominant factor in determining ionic selectivity. However, if one of a pair of exchanging ions is large, the configurational free energy may be quite important. Recent thermodynamic calculations on the exchange of tetramethylammonium ion with the sodium form of variously cross-linked polystyrene sulfonates have shown that the increase in the free energy of the molecular network when this large cation enters the exchanger is sufficient to cause a pronounced reversal in its preferred uptake. This reversal with increased cross-linking also was observed experimentally. The quantitative agreement between the computed equilibrium partition and that found experimentally has been taken as an additional confirmation of the Gibbs-Donnan model on which the calculations were based.

Physical Chemistry of Polyelectrolyte Systems: Thermodynamic Properties of Aqueous Quaternary Ammonium Halide Solutions. — Heats of dilution of tetramethyl, -ethyl, and -n-propyl ammonium chloride, bromide, and iodide, and tetra-n-butylammonium chloride and bromide were measured at 25°C from near saturation to 0.2 m or lower to final concentrations below 0.003 m. The apparent molal heat content data, Φ_L, were differentiated to obtain the relative partial molal heat contents, $\bar{L}_1$ and $\bar{L}_2$, of the solvent and solute respectively. The Φ_L data were combined with previously reported activity and osmotic coefficient (free energy) data to obtain the excess or nonideal apparent molal entropy Φ_S. It was found that the free-energy function Φ_G was small and relatively invariant compared with the very large values and dramatic changes observed in the values of Φ_L and Φ_S within the series $(CH_3)_4N^+$, $(C_2H_5)_4N^+$, $(n\text{-}C_3H_7)_4N^+$, and $(n\text{-}C_4H_9)_4N^+$. Over a large concentration range the values of Φ_S and Φ_L were almost independent of the anion associated with these large tetraalkyl ammonium cations. These results suggest that previous structural inferences based on the relatively insensitive free-energy function may be misleading, and that models of these electrolyte solutions must account for the much larger entropy and enthalpy effects. The independence of the entropy on the size of the anion suggests that the effect of these large tetraalkyl ammonium cations on the water structure is so severe as to overshadow the differences due to the variation in the size of the anion.

Electrochemical Kinetics and Its Application to Corrosion. — The inhibitory properties of passivating inhibitors, such as chromates, have been ascribed variously to their oxidizing power or to effects associated with adsorption of the unreduced anions. We have been able to make a definitive experimental differentiation between these interpretations by utilizing the unique properties of technetium compounds. We have found that iron may be passivated anodically in a pertechnetate solution at a potential that is too noble for reduction of TcO_4^-. Activation on open

circuit demonstrated that the film behaved normally for a passive film formed in the absence of an inhibitor. Yet passivation was effected at a current density far lower than that needed in an uninhibited environment. Also, the charge consumed in passivation was enormously reduced in the presence of the inhibitor. Beta counting proved that the inhibitor was not reduced during passivation. Hence the presence of the inhibitor anions alone accounts for the effects that render passivation so much easier in the presence of the inhibitor. An oxidizing agent is still required, but it need not be the inhibitor itself. Measurements of the cathodic polarization of both chromate and pertechnetate couples showed that their reduction has too high an overvoltage to permit them to induce passivation without the associated effects of the unreduced anions.

High-Temperature Aqueous Solution Chemistry. — Knowledge of the solubility of magnetite (Fe_3O_4) in water at elevated temperatures is of general interest, and is of particular importance to those who are interested in the mechanisms of the geologic deposition of iron or the transport of radioactive iron in pressurized-water reactors. We are studying this solubility at temperatures between 150 and 300°C in solutions containing 0 to 30 $\mu\underline{M}$ HCl. Preliminary values for the concentration of iron in pure water are 1.5 and 0.33 $\mu\underline{M}$ (±50%) at 200 and 260°C respectively. These results were obtained by pumping the water (saturated at room temperature with hydrogen at 1 atm) through a bed of finely divided radioactive Fe_3O_4 and analyzing the effluent.

Physical Chemistry of Molten Salts. — The vapor pressures of the system LiF-BeF_2 are being investigated to derive thermodynamic activities, to determine the significant vapor species, and to obtain data of importance to the Molten-Salt Reactor Program. Manometric vapor pressures were measured for compositions from 25 to 100 mole % BeF_2 in the temperature range 840 to 1280°C. Above 65 mole % BeF_2, where the vapor is virtually pure BeF_2, the activity coefficient of BeF_2 in the melt was found to be above unity. These values of the activity coefficient are consistent with results of the H_2O-HF equilibration studies of the LiF-BeF_2 system.

The compound Li_3AlF_6 was prepared previously in studies of molten-salt solvents for aluminum-matrix fuel elements; recently, single-crystal specimens have been grown for x-ray diffraction analysis. The crystals are orthorhombic with unit-cell dimensions a = 9.54 A, b = 4.88 A, and c = 8.23 A. The probable space group, deduced from systematic absences, is Pnma or $Pna2_1$. An x-ray density of 2.80 g/cm^3 was calculated by assuming four formula weights per unit cell. Determination of the atomic positions is in progress.

Surface Chemistry. — Gravimetric and calorimetric investigations have revealed the presence of the equivalent of three chemisorbed layers of water on the surface of thorium oxide. This water cannot be removed completely in vacuo until the temperature is increased to 900–1000°C. Thorium oxide samples calcined at higher temperatures (1600°C) exhibit three distinct modes of adsorption, whereas the samples calcined at lower temperatures (650 to 1000°C) have considerable surface heterogeneity. This heterogeneity masks the discrete nature of the three modes of chemisorption. Infrared spectra have been employed to more fully elucidate the nature of these interactions. Surface hydroxyl groups have been observed

even after samples have been outgassed at 500°C, with considerably more complex water structure present at lower outgassing temperatures. The overall chemisorption process appears to be an initial formation of surface hydroxyl groups, with subsequent layers involving rigid orientation of water molecules.

Isotope Chemistry. — The Raman spectra of the newly discovered IOF_5 were obtained for both gaseous and liquid states. The band frequencies, intensities, and polarizations are given below:

Raman Spectral Bands of IOF_5

Band Frequency (cm^{-1})		Relative Intensity	Polarization[a]
Liquid	Gas		
205		0+	
305		1	D
341	340	4	D
374		1	D
503		0+	
608[b]		0+	
640	640	9+	P
679	680	10	P
700[b]		0+	
924	928	4	P

[a]P = polarized, D = highly depolarized.

[b]Shoulder.

These data, combined with infrared spectra, were used to derive the valence force constants given in the next table. Also shown are data for the related compounds, IF_5 and $XeOF_4$.

Valence Force Constants for IOF_5 (millidynes/A)

Constant	IOF_5	IF_5	$XeOF_4$	Description in IOF_5
f_0	6.99		7.08	I–O stretch
f_R	4.60	4.68		I–F_5 stretch
f_r	4.42	3.64	3.26	I–F stretch
$f_{rr'}$	0.18	0.18	0.10	Interaction
f_{rr}	0.00	0.06	0.12	Interaction
f_{α/r^2}	0.20	0.17	0.12	F–I–F bend
$f_{\beta/rR}$	0.55	0.63		F–I–F bend
$f_{\gamma/rS}$	0.44		0.53	F–I–O bend
$f_{\beta\beta'/rR}$	0.08[a]	0.16		Interaction
$f_{\gamma\gamma'/rS}$	0.08[a]		0.10	Interaction
$f_{\alpha\alpha/r^2}$	–0.03	–0.02	–0.02	Interaction
$f_{\beta\gamma/r\sqrt{RS}}$	0.06			Interaction

[a] $f_{\gamma\gamma'}$ set equal to $f_{\beta\beta'}$.

The results confirm an octahedral type of structure having C_{4v} symmetry and bond angles close to 90°.

TRANSURANIUM-ELEMENT PROCESSING

Process Development: Preparation of Actinide and Lanthanide Oxide Microspheres. — Laboratory studies of lanthanide sol preparations have continued, and the lanthanide sol-gel procedure was used to prepare microspheres of ^{244}Cm oxide.

In two small-scale experiments (50 mg of ^{244}Cm), curium sol-gel behavior paralleled lanthanide behavior closely. Two radiolytic effects were noted, but neither appeared to limit the usefulness of the technique. During sol concentration, severe foaming occurred at a curium concentration of about 100 g/liter; however, it was possible to form microspheres from sols of this concentration. A second effect resulted in considerable porosity due to the radiolytic decomposition of water in the set gel sphere. This effect was minimized by rapid drying and calcining following sphere formation.

In the second experiment the gel spheres were calcined at 800°C, and black oxide spheres were obtained. However, some of this material appeared to undergo a crystal transformation after standing about 24 hr. The color changed from black to white, and distegration of the particle

was noted. This effect may be due to the adsorption of water on the oxide formed at the rather low calcination temperature of 800°C. Greater stability should be obtained by calcining above 1000°C.

Since it will be desirable to prepare HFIR targets from mixed ^{243}Am-^{244}Cm oxides, mixed lanthanide sols were investigated. Satisfactory calcined microspheres were obtained from mixed praseodymium-neodymium sol preparations. Sol-gel preparation of mixed americium-curium oxides will be investigated as soon as the shielded alpha-gamma facility in Building 3508 is operable.

Crushing strengths were determined for various batches of praseodymium oxide microspheres. It was found that crushing strength increases with the square of the sphere diameter. Typically, crushing strength varied from 130 to 824 g for sphere diameters of 90 and 230 μ respectively.

HFIR Target-Fabrication Development. — Mockup tests of the remote fabrication equipment revealed several items that require design modifications. These are being rapidly completed, and no schedule delays are anticipated. The equipment in cubicle 3 was operated daily for one week, and 60 pellets were prepared, with an average rate of one pellet every 15 min.

The simulated target elements to be used during the criticality experiments of HFIR have been completed. The manufacture of plutonium-bearing pellets for the first target loading in the reactor has begun, and we anticipate completing the task by August 15.

METALLURGY AND MATERIALS

Physical Properties Studies. — A pulse-heating calorimetric technique for measuring the specific heat of electrical conductors over a wide temperature range was developed. A direct current is used to self-heat an instrumented, rod-type specimen suspended in a constant-temperature black-body vacuum chamber. To determine the specific heat, the current flowing through the specimen is increased stepwise. A digital voltmeter capable of recording 300 readings/sec measures the time dependence of the temperature change and power dissipation in a known length of the specimen. Radiation heat losses occurring during the 4-sec heat pulse are accounted for with total hemispherical emittance values obtained with the specimen at constant temperature. An error analysis predicts an accuracy of ±1.0% from 100 to 800°C.

The accuracy and reproducibility of the technique were demonstrated by measurements on pure iron from 100 to 1400°C and by subsequent comparison with literature values. Below 910°C the reproducibility was ±0.5%, and the agreement with the literature was ±2.2%, except in the vicinity of the Curie transformation. The specific-heat results from 910 to 1400°C were within 5% of several literature results.

At 772.8°C the specific heat changed discontinuously from a value of 0.346 to 0.267 cal g^{-1} $(°C)^{-1}$. Consequently, the Curie phenomenon of iron is a second-order transformation, caused by a change in the rate of decoupling of aligned atoms at the ferromagnetic-to-paramagnetic transition.

The thermal conductivity of a 2-in.-diam single crystal of silicon is being measured in the radial heat-flow apparatus. Results to 500°C showed that the thermal resistance is not linear in temperature. A phenomenon other than three phonon umklapp processes must be operating; to further define this behavior we are obtaining data at least every 25°C in this range.

Solid Reaction Studies. — The diffusion of ^{44}Ti in the body-centered cubic alloy V–20% Ti was studied to further characterize the non-Arrhenius behavior we previously reported in β-titanium [Acta Met. 12, 1033 (1964)] and vanadium [Trans. AIME 233, 243 (1965)]. The data appear to be best fitted on an Arrhenius-type plot by a straight line from 1100 to 1350°C and a line of continuous curvature from 1350 to 1650°C. Selected values of the diffusion coefficient D follow:

Temperature, °C	1100	1150	1210	1310	1417	1520	1625	1668
$10^{10}D$, cm^2/sec	0.299	0.649	1.52	5.31	21.8	65.2	192	294

CONTROLLED THERMONUCLEAR RESEARCH

The DCX-1 Injection-Accumulation Experiment. — We have studied the charge-exchange signals from plasmas established by high-energy molecular ion injection into mode I and mode II discharges operated along the axis of the facility. The arc reflectors were mounted outside the mirror coils. The pressure differential required for mode II was established by bleeding deuterium gas into the plasma chamber and isolating this chamber from the reflectors by restricting orifices at the coil throats. The input power for mode II operation was from 20 to 50 kw, and the neutral gas pressures were typically 2 to 5×10^{-4} torr (plasma chamber) and 1 to 2×10^{-5} torr (reflector region). For mode I the values were approximately 5 kw and 5×10^{-4} torr. The injected beams were 600-kev H_2^+ and 400-kev H_3^+ on the standard beam trajectory (turnaround at R = 3.25 in.), and 600-kev H_2^+ with turnaround at the magnetic axis. The experimental observations differ in detail, but both arcs appear to contribute to dissociation. In comparison with gas dissociation at the same indicated ion gage pressure in the plasma chamber, operation with the arcs yielded increased trapping fractions. The evidence was most clear with the beam trajectory displaced to the center of the arc column (displaced to axis); the trapping enhancement then ranged from 10 to 100%. No definite conclusion was reached as to the size of the "burned-out" region possibly associated with mode II. It was not possible to isolate the effects of burnout on fast-proton lifetime from the effects of the gross pumping action displayed by both discharges. If present, the burned-out region was small compared with the orbits of the trapped energetic protons, so the region was not greater than 6 in. in diameter.

Studies of the effects of deliberate changes in trapped proton energy distributions were continued. A cyclotron dee structure suitable for operation at the third harmonic of the proton cyclotron frequency was added to allow some control of this distribution. The structure was fed by the

amplified output of a noise generator filtered to pass signals in a band about 2 Mc wide and centered on the third harmonic. Using this harmonic drive allowed the determination of instability thresholds by observation of the fundamental component of the usual instability rf spectrum. The amount of energy spread required to stabilize at a particular density is not a rapidly varying function of containment time, and may be a constant. At fixed containment time, the spread (full width at half maximum) varies as $(n_+)^{1/2}$, with 30 kev stabilizing $n_+ = 3 \times 10^7$ cm^{-3} at $\tau = 60$ msec. Unfortunately, this method of stabilization results in proton losses against the dee structure itself. These losses have limited the maximum achieved densities to about 3×10^7 cm^{-3}.

A cyclotron structure suitable for operation at the fundamental has been constructed. It is mechanically less elaborate and will perhaps offer a less effective target for these proton losses.

Properties of Plasmas Created by Electron-Cyclotron Heating. — Measurements of the electron temperature and density have continued on the ELMO facility with up to 1 kw continuous wave of 35 gigahertz microwave power. The electron temperature appears to be higher than anticipated, perhaps 500 kev. The density of these hot electrons is in the range of 2×10^{11} cm^{-3}. The amount of energy stored is about 50 times larger than that stored in a 10.6-gigahertz plasma of equivalent volume and input. Similarly, the rate of emission of neutrons produced by Coulomb dissociation is two orders of magnitude higher than that produced at 10.6 gigahertz. Currently, the thickness of the lead wall around the ELMO facility is being increased by a factor of 3 to reduce the x-ray radiation level.

Injection experiments are continuing on the EPA facility. The new mirror coils which provide a 2:1 mirror field are now in operation. The microwave plasma produced in the new field apparently has a higher temperature. The x-ray intensity outside the lead wall has increased by almost an order of magnitude. Additional lead walls have been added for personnel protection.

It has been possible to reduce the x-ray intensity outside the lead wall by means of a radial plasma limiter. The decay time of the trapped 20-kev protons does not appear to be affected by this limiter.

Investigation of the instabilities that can be induced in hot electron plasmas is continuing in the PTF facility. The particle losses have been correlated with oscillations in the plasma. An intense oscillation at $\omega_{ce}/2$ has been observed at the time that bursts of high-energy electrons come out of the magnetic mirrors. This loss of electrons through the mirror is believed to be due to the "mirror instability."

Properties of Plasmas Created by Beam-Plasma Interaction. — We are now able to operate burnout V with 75 kw in the electron beam. This is an increase of 25 kw over the 50 kw reported in March. It still appears that nearly 45% of the beam power is dissipated in the end electrodes. The power increase was made possible by increased radiation cooling of the filament.

Plasma spread in radius has been photographically observed in burnout V as in burnout IV upon the onset of interaction with the electron beam. We have examined by electrostatic and magnetic analysis some of the charged particles escaping the liner (4-in. radius). The analysis so far is still crude and does not rule out the existence of contaminants in the plasma.

We can say that some of the escaping ions are deuterons with energies of 14 to 45 kev. The analyzer current together with an estimate of its aperture gives a density of 3×10^4 cm^{-3} for the 19- to 20-kev bracket.

A Langmuir probe measurement of the electron temperature seemed to indicate 25 to 50 ev at the maximum radius of 4 in. At smaller radii, considerable interference with the plasma was experienced. Here the electron temperature appeared to be between 50 and 100 ev.

In the hot-electron blanket experiments in progress in the beam-plasma facility, both density and temperature have been increased through the use of new designs in arc anode and gas feed distribution. Hot-electron density is now on the order of 10^8 cm^{-3} at a temperature in excess of 100 kev. Langmuir probe studies in the plasma streaming through the mirror indicate a "warm," ionizing trapped plasma having a density about ten times that of the hot-electron density. Evidence for the warm, non-x-ray-producing plasma is seen upon insertion of a 0.005-in.-thick, 1-in.-wide tantalum paddle radially inward through the blanket. X-ray production ceases, but the paddle is cut into pieces in about 10 sec.

Ion Source and Ion Beam Technology. — The 100-kv test stand has been modified to make possible an 8-ft beam drift space instead of the previous 4-ft space. This facility will be used for a more detailed study and a continuing effort to improve the 400- to 500-ma H^+ beam already developed in the energy range 50 to 70 kev. Attempts will also continue to produce an H_2^+ beam of comparable intensity to the H^+ beam. Studies have started to produce an intense hydrogen beam in the energy range 5 to 10 kev that would be suitable for injection into a thermonuclear experiment. These studies will also continue to be performed on the 100-kv test stand until a 10-kv test stand, now being designed, is available.

Theoretical Plasma Physics. — Growing electrostatic plasma waves propagating perpendicular to a confining magnetic field $\vec{B}$ (the so-called flutelike modes) pose a special problem for the controlled thermonuclear program. Since they produce no electric field along $\vec{B}$, these modes are not subject to Landau damping by cold charged particles moving along $\vec{B}$. Also, because the perpendicular electric field will induce an $\vec{E} \times \vec{B}$ drift of particles out of the system, flute modes are expected to be important sources of anomalous diffusion. The well-known hydromagnetic flute instability can be prevented if the confining magnetic field strength increases in all directions away from the center of the plasma, that is, if the plasma is in a magnetic well. However, in addition to hydromagnetic flutes, there also exist flute modes at the ion gyrofrequency, ω_{ci}, caused by resonance between ion gyration and various drift waves, notably the diamagnetic drift associated with the density gradient. Such drift-cyclotron flute modes exhibit a density threshold which has the form

$$(\omega_{pi}/\omega_{ci}) < (R/a)^p$$

for stability, where $R \equiv (n_0^{-1}\, dn_0/dr)^{-1}$, a is the ion gyroradius, and p is a power which depends on the plasma distribution function. For a Maxwellian ion distribution with monotonically decreasing density, $p = 1$. But for loss-cone distributions (having no ions with zero perpendicular speed), $p = 3/4$. And for density profiles which vary as r^2 near $r = 0$

(i.e., hollow cylindrical plasmas), p = 1/2. For a density of 10^{14} ions/cm^3 in a confining field of 10 kilogauss, these plasmas would be stable for (R/a) exceeding, respectively, 100, 500, and 1000. Thus, one is led to expect stability only for machines which are many gyroradii across at reactor densities.

Atomic Cross Sections. — Directed neutral beams which have an energy distribution have been formed by passing beams of H^+ or H_2^+ through an argon gas cell. The gas cell was composed of an eleven-electrode deceleration system with an axial linear decreasing electric field. Without applied retarding potentials the efficiency of conversion of 50-kev H^+ to neutrals was 30%. Application of 20 kev retarding potential caused severe defocusing, resulting in a lowering of the conversion efficiency to 2%. Experiments with exponential electric gradients in the decelerating gas cell are continuing.

Evaluation and development studies of semiconductors used as particle detectors have continued. Cooling a silicon surface-barrier detector to liquid-nitrogen temperatures and using a field-effect transistor preamplifier placed in the vacuum system have provided a resolution (full width at half-maximum amplitude) of 2.85 kev for protons in the energy range 7 to 30 kev.

Crystals of barium titanate have been used as pyroelectric detectors. Particles impinging on the front surface of the crystals induce a spontaneous polarization current which is measured with a sensitive current meter. The measured maximum sensitivity of these detectors was 10^{-4} w with a response time less than 1 msec.

Atomic and Molecular Processes Information Center. — Activities of the Atomic and Molecular Processes Information Center have been mainly concerned with literature search and classification of references. Approximately 8000 references are now awaiting processing. A 136-page annotated bibliography of the 1963 literature has been prepared and is now being printed. A unique card system has been designed and tested for storage of information. The entire journal reference is key punched onto IBM cards which are then sorted by author and journal to provide a working bibliography. After review and evaluation by the technical staff, this additional information is key punched onto the original cards. These cards are then re-sorted and printed out as an annotated bibliography. Plans are now being made to convert our manual system to this type of machine storage system. Systems involving information retrieval are being studied.

A display of the activities of the Information Center has been prepared for the IVth International Conference on the Physics of Electronic and Atomic Collisions.

BIOLOGY AND MEDICINE

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation. — All lithium-drifted gamma-ray detectors currently produced are stabilized against accidental warmup by coating the

N side (lithium-doped side) of the germanium wafer with a thin layer of metallic lithium. This prevents the out-diffusion and subsequent loss of the lithium dopant from the germanium when the detector is at or near room temperature. In the event that a detector is accidentally allowed to warm up, it is usually only necessary to apply a small bias voltage for a few hours to relevel the lithium distribution in the compensated region of the junction.

Most of the commercially available insulated-gate field-effect transistors were experimentally characterized. Measured input capacitances range from 1 to 12 pf, and maximum transconductance ranges from 130 to 1700 micromhos. The noise power spectral density has a dominant low-frequency component which extends beyond 1 megahertz in most devices. The low-frequency density is given empirically by the equation $R_n^{\ell f} = K/f^{\alpha}$, where α ranges from 1 to 1.4. The measured total noise resistance (white noise plus low-frequency noise) ranges from 10 to 300 megohms at 1 kilohertz, 250 kilohms to 20 megohms at 10 kilohertz, 15 to 700 kilohms at 100 kilohertz, and 2.5 to 100 kilohms at 1 megahertz.

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