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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JANUARY 1966

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

January 1966

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JANUARY 1966

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

REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

Compatibility of Oxide Fuel and Graphite Under Irradiation. — We are conducting experiment 05-10 in the ORR poolside facility to evaluate the compatibility of $(U,Th)O_2$ fuel pellets with graphite under high-temperature gas-cooled reactor conditions. The fuel-graphite interface has been maintained near the design value of 1370°C and swept with an inert gas containing 230 ppm of CO. During the last six weeks of irradiation the release rate of ^{135}Xe (expressed as R/B, ratio of release rate to birth rate) increased from 1.5×10^{-2} to 18×10^{-2}, and the indicated CO concentration in the sweep gas decreased from approximately 300 to 205 ppm. We believe that significant changes have occurred within the experimental assembly and plan to remove the capsule for postirradiation examination during the next ORR refueling shutdown.

High-Burnup Coated-Particle Irradiation Experiment. — We are conducting an experiment to evaluate the performance of loose pyrolytic-carbon-coated fuel particles in a graphite container at high burnup (50 at. % of the heavy metal) under conditions of interest to high-temperature gas-cooled reactors. Irradiation continued during the current report period at a measured fuel temperature of approximately 1240°C. The estimated burnup as of January 17 was 37.8 at. % of the heavy metal in the UC_2 particles in the lower fuel compartment and 35.6 at. % of the heavy metal in the sol-gel UO_2 particles in the upper compartment.

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

Postirradiation examination of the second capsule, designated P4B-2, is continuing. Visual examination of the two fuel specimens revealed no evidence of distortion or flattening such as was observed on the specimens

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from the first capsule; the only deformation noted was a slight bowing of the lower fuel specimen. We believe the lower heat rating and cladding temperatures during most of the irradiation of the second capsule contributed to the greater apparent stability of the fuel specimens.

Plasticity Theory as Applied to Graphite. – In order to account for the nonlinearity of the stress-strain diagrams for graphite obtained from uniaxial test data, we have been investigating the feasibility of using plasticity theory as a basic mathematical model for describing the materials behavior. In order to use plasticity theory for graphite, it has been necessary to extend many of the concepts of the basic theory to include such things as anisotropy and the absence of a definite yield point. A computer program, GUSS, was written for solving the problem of a long thick-walled cylinder loaded with a temperature distribution through the wall thickness and/or internal and external pressures. The program was used to study the effect of various assumptions concerning the shape of the stress-strain diagrams on the computed values of stress and strain in a cylinder loaded with internal pressure. The study shows that the assumed stress-strain relationships have a pronounced effect on the results and therefore must be realistically characterized in order to obtain accurate calculations. Additional studies will be made for thermal and irradiation stresses.

Heat Transfer Analysis of Fueled-Graphite Irradiation Experiments. – We carried out a simplified heat transfer analysis for fueled-graphite irradiation capsules to assist in correlating power generation rates in the fueled-graphite spheres, as calculated from flux monitor analyses, with measured temperatures in the capsules adjacent to the sphere surface. A computer program was written to perform the necessary heat transfer calculations and to develop plots of the radial linear heat flow, H, across the container thermocouple in terms of the thermocouple reading, T_d, for each type of capsule employed. For each test sphere an average value of H corresponding to the time-averaged container temperature was read from the appropriate plot. The H values were correlated successfully with the average heat generation rate, P, computed from flux-monitor analyses by the equation

$$P = H/(0.0506 + 0.000221H) \ . \qquad (1)$$

This correlation applies directly to the No. 3 (sweep) sphere in all experiments that contained 6-cm-diam spheres; however, due to heat flow between Nos. 1 and 2 (static) spheres in a given capsule, a correction was applied to determine their actual heat generation rates.

This analysis was applied to operating data from the three AVR spheres irradiated in capsule experiment 01A-8 to determine: (1) the operating power level in each element throughout the irradiation test, (2) the fuel burnup in each element, (3) the effective thermal conductivity of the fueled-graphite core of the element provided with a central thermocouple, and (4) the central temperature of the two fuel elements not instrumented with central thermocouples. The fueled-graphite thermal conductivity was well fitted as a function of temperature by the equation

$$1/k = 0.0887T - 4.18 \ , \qquad (2)$$

[redacted]

where k is the effective thermal conductivity of the fueled core in w cm^{-1} $(°C)^{-1}$ and T is the average fueled-core temperature in °C. Power, burnup, and central-temperature results are summarized in the table below.

Summary of Operating Conditions for AVR Capsule O1A-8

	Fuel Element Position		
	Upper Static	Lower Static	Sweep
Time-averaged container temperature, °C	778[a]	842[a]	853[a]
Maximum container temperature, °C	843[a]	904[a]	878[a]
Time-averaged central fuel temperature, °C	1144[a]	1442	1371
Maximum central temperature, °C	1275[a]	1562	1403
Time-averaged fission power, w	2299	3096	2754
Maximum fission power, w	2495	3228	2800
Burnup, at. % of heavy metal	5.53	7.45	6.62

[a]Measured directly or averaged from measured values; all other values were calculated by using power-temperature correlation.

It should be noted that the thermal conductivity given by Eq. (2) agrees well with values obtained from earlier fueled-graphite irradiation tests. This supports the accuracy of the power-temperature correlation for calculation of heat generation rates. Further, the measured thermal conductivities did not show a significant change over the life of the experiment; this indicates that a progressive shrinkage of fueled core away from the unfueled shell did not occur.

Dynamic Seals for EGCR Charge Machine. – We are continuing development of a piston ring and a shaft seal for the actuating cylinders used to control the various motions of the EGCR charge machine, since this work has general applicability to gas-cooled reactor component technology. A unit with carbon-wool-impregnated Viton-B O-rings and SP-3-type polymer rider rings has operated successfully in a dry helium atmosphere at temperatures up to 177°C for over 273 temperature-pressure cycles, or the equivalent of five years of operation in the charge machine. The total leakage bypassing the rings and the shaft seal has approximately doubled during the test period; however, the present leakage is not considered excessive.

REACTOR FUELS AND MATERIALS

Nondestructive Test Development. – Recent ultrasonic development has included further study on the detection of nonbonding in fuel tubes.

A through-transmission technique similar to the one we developed for flat fuel plates can be conveniently performed in an ordinary laboratory, but it would be difficult to adapt to a hot cell requiring remote handling methods. For this reason we have developed a modified through-transmission technique employing a single transducer as a transmitter and receiver. The ultrasonic energy generated by a piezoelectric transducer is transmitted through a water couplant into the tube wall and across the inside of the water-filled tube, reflected from the inner surface of the opposite tube wall, and then transmitted back along the original path to the transducer (receiver). Thus any nonbond will attenuate the sound transmitted through the tube wall in both directions.

We determined optimum parameters of calibration, transducer-to-specimen distance, etc. Using these data, we then fabricated a Teflon guide block to maintain proper alignment between the transducer and the specimen during evaluation. This technique was successfully applied in a hot-cell remote-handling facility. The method, which incorporated a remote-controlled mechanical system for helically scanning the fuel tubes, could detect and record nonbonds equivalent to or larger than a 1/16-in.-diam electro-discharge-machined reference hole.

REACTOR COMPONENTS

Reactor Controls Development: Neutron Fluctuation Analysis. – The use of reactor neutron-population fluctuations to measure reactor parameters is well established. Analysis of current fluctuations of the output of a neutron-sensitive ionization chamber has been applied to the measurement of reactivity of subcritical reactors. Work in this field has been previously reported. However, the potential advantages of such measuring methods have not been realized in practice, partly because of high gamma-ray intensities associated with shut-down reactors after a period of operation at high power. To overcome this obstacle to the practical use of measurements of fluctuations in shut-down reactivity, we have investigated the use of pulse-type neutron detectors in place of integrating current-type ionization chambers. The advantage of detecting individual pulses is that we can discriminate between neutron-induced signals and smaller, gamma-ray-induced signals, and the gamma-ray signals can be eliminated. A pulse detector is no different in principle from the previously used current-type ionization chamber, since in any case, detection of a neutron is a discrete event, which leads to the collection of charge in the detector. However, the essential difference lies in the electrical details of the associated instrumentation: for pulse-type detectors the fluctuating signal is generated in an integrating instrument; whereas for current measurements, the integration takes place in the detector-amplifier input-circuit capacitance.

A feasibility experiment was made at the Pool Critical Assembly to prove that, indeed, the use of pulse-type neutron detectors yields the same information regarding reactor subcritical reactivity as does integrating current-type ionization chambers. A BF_3 proportional counter and a BF_3-filled ionization chamber were installed in proximity to the shut-down reactor core. For a given subcritical reactivity condition, a value

of −2.26 ± 0.20 dollars was obtained from the proportional counter channel, as compared with −2.47 ± 0.17 dollars from the ionization chamber channel.

Reactor Controls Development: Nuclear Sensors. — In many reactor experiments in which reactor parameters are measured by use of reactor neutron-population fluctuations, the detector location can be a seriously limiting problem. The space available for the detector is restricted and is often so far from the core that the reactor neutron-population fluctuations measured by the detector can be masked by the statistical fluctuations in the detection process. To prevent this difficulty, maximum neutron detection efficiency is required.

A commercially available Reuter-Stokes type 81A ionization chamber was filled at a pressure of 2.5 atm with a gas mixture of 85% helium-3, 10% xenon, and 5% methane to replace the standard filling of 1 atm of BF_3 gas. Helium-3 is the neutral gas which presently is known to have the highest absorption cross section for thermal neutrons. Xenon was added to increase the stopping power of the ionized particles (the stopping power of helium-3 is poor). Methane was added to act as a quenching gas, and to increase the ion mobility and, therefore, reduce recombination. Experimental results indicate that the chamber detection efficiency is increased by a factor of 2 when the chamber is filled with the helium-xenon-methane gas mixture instead of with 1 atm of BF_3 gas. The saturation characteristics for the gas mixture are also superior.

Reactor Controls Development: Reactor Computer Control. — A real-time calculation of reactivity from neutron flux by use of an on-line digital computer has several potential applications: (1) for control-rod worth determination, by observing the reactivity change corresponding to a given change in rod position; (2) as a criticality meter, by continually displaying the system reactivity; and (3) as a reactivity anomaly indicator, by comparing the expected reactivity based on rod position, xenon concentration, fuel burnup, etc., with that calculated from neutron flux behavior.

A simple algorithm was developed to calculate reactivity from $\Phi(t)$ and $d\Phi(t)/dt$ in reactor systems having an on-line digital computer. A space-independent reactor kinetics model is used, and the relevant differential equations are solved numerically. The technique shows good promise of being able to calculate reactivity in real time with a moderate-speed computer, and the accuracy afforded appears to be sufficient for the applications envisaged.

POWER REACTOR FUEL PROCESSING

Aqueous Process Development: General. — Development of mechanical, nonaqueous, and aqueous steps for head-end processes designed to convert power reactor fuels into aqueous solutions suitable for subsequent fuel decontamination and recovery by the well-established methods of solvent extraction is continuing. During the past month the major efforts were on (1) the shear-leach process for multitubular assemblies of metal-clad

oxide fuels for pressurized- and boiling-water power reactors and SAP-clad HWOCR fuel (sintered-aluminum-product-clad fuel for heavy-water-moderated organic-cooled reactors); (2) the grind-leach process for HTGR graphite-UC-ThC fuel (for high-temperature gas-cooled reactors); and (3) hot-cell evaluation of the adsorption of ^{233}Pa from Thorex dissolver solution onto unfired Vycor.

Aqueous Process Development: Shear-Leach Process. — In this process, fuels with nitric-acid-insoluble cladding (stainless steel, Zircaloy-2, etc.) are sheared to expose the fuel oxide, metal, or alloy, which is then dissolved in nitric acid or fluoride-catalyzed nitric acid. The cladding is subsequently discarded as a metal waste. Current studies include (1) computer evaluation of fission product heat generation for various steps in the process and subsequent heat dissipation and pressurization on quenching the fuel-containing basket in a dissolver; (2) reevaluation of scoring and galling problems on the gibs and liners of the 250-ton shear; (3) further evaluation of the hazards involved in the handling of zirconium fines produced in the sawing and shearing of Zircaloy-2-clad fuel; and (4) hot-cell determination of tritium and rare-gas fission product release in the fuel-shearing operation.

Writing and debugging was completed on the computer code BIG DEAL for calculating temperatures in irradiated power reactor fuel during (1) canal storage; (2) mechanical disassembly; (3) residence in the shear feed envelope; and (4) storage of sheared fuel in air in baskets of 5-, 7-3/4-, or 10-in. ID in a four-by-four array. The code also computes the pressures developed in a batch dissolver resulting from the quenching of the baskets of sheared fuel in cold nitric acid. Spent-fuel temperatures for burnups of 8000 to 50,000 Mwd per ton of uranium, specific powers of 10 to 70 w per gram of uranium, and cooling times of 180 to 1080 days were calculated. These calculations indicate that excessive temperature does not appear to be a serious problem in shear-leach processing and that operation of the dissolver appears to be safe from a pressure-buildup standpoint.

The existing Stoody-1 gibs and type 420 stainless steel liners of the 250-ton shear were completely decontaminated from natural uranium prior to machine grinding. Grinding is being done to remove all previous indications of scoring or galling in preparation for additional wear studies. In addition, a D-2 tool-steel gib and a Stellite 6B liner will be fabricated to evaluate the wearability of this combination of materials. The surface of the gibs and liners will be given a No. 16 finish. This will be followed by hand scraping (fishtails 1/4 in. apart) over 20% of the total area.

Tests were conducted to evaluate further the hazards of handling zirconium fines produced in abrasive-disk sawing (to remove end boxes) and shearing of Zircaloy-2-clad fuel elements. Material for these tests was produced by both wet and dry abrasive-disk sawing of reactor-grade Zircaloy-2 rods and by shearing of 0.439-in.-diam by 32-mil-wall Zircaloy-2 tubing filled with UO_2 pellets. All samples were then sieved into eight particle-size fractions between 4760 and 44 μ. The UO_2 in the sheared material was dissolved away from the zirconium fines with 6 M HNO_3; no attempt was made to separate the abrasive-disk debris from the zirconium fines in the sawed material, where the volume ratio of abrasive to

zirconium particle was 7 to 1. Ignition tests were made on all samples with a 20,000-v spark discharged from a Tesla coil type of probe. The samples were also tested with a Phillips electron-probe analyzer to determine the extent of zirconium conversion to ZrO_2 during mechanical processing. In the ignition tests, none of the fines produced by sawing and only the particles smaller than 149 μ produced by shearing could be ignited. The electron-probe analysis showed that most of the sawing fines are apparently converted to oxide but that the Zircaloy-2 shearing fines are not oxidized during either shearing or leaching.

The results of these tests indicate that the fines resulting from sawing are safe because they are largely converted to ZrO_2 and are highly diluted with abrasive-disk debris. It was also concluded that particles larger than 149 μ produced by shearing are safe but that smaller fines should be periodically removed from the dissolver and dissolved in HNO_3-HF.

To determine the extent of tritium and rare-gas fission product release to the environment during the shearing of gastight, metal-clad oxide fuels, several stainless-steel-clad fuel samples containing 96% ThO_2–4% highly enriched UO_2 pellets (96% of theoretical density) irradiated to 25,000 Mwd/ton were sheared in special hot-cell equipment designed to trap and measure the amounts of tritium and rare gases released. Shearing one end of the fuel samples released a maximum of 0.026% of the calculated fission yield of tritium; a maximum of 0.269% was released when the fuel capsules were completely sheared into 1/2-in. segments. The release of ^{85}Kr by a single cut was 0.5%; 1.07% was released by multiple shearing. Tritium was found to diffuse very slowly from the dense oxide fuel pellets when they were exposed. Further gas-release tests during shearing of UO_2 fuel and during leaching of both ThO_2-UO_2 and UO_2 fuels are scheduled.

Aqueous Process Development: Grind-Leach Process. – A grind-leach process is being studied as a possible alternative to the burn-leach process for high-temperature gas-cooled reactor fuels that contain pyrolytic-carbon-coated UC_2-ThC_2 or UO_2-ThO_2 particles either as dispersed or as consolidated fuel-bead compacts in graphite. In the grind-leach process, the fuel is crushed to –6 mesh and subsequently ground to –140 mesh to break all the pyrolytic carbon coatings on the fuel particles. The ground fuel is then treated with 13 M HNO_3–0.05 M HF. The undissolved graphite is filtered and washed to permit complete removal of the dissolved uranium and thorium. Both engineering- and laboratory-scale tests have been made with unirradiated General Atomic–Peach Bottom ThC_2-UC_2-graphite fuel compacts (3% U–14% Th–83% C). The –6 mesh material was easily produced from the fuel compacts in a hammer mill. This material was passed five times through a roll crusher with 8-in.-diam by 2-in.-wide rolls. About 82% of the –6 mesh fuel was reduced to –140 mesh (105-μ) material. Because of agglomeration of graphite particles into flakes (3000-μ-diam by < 125-μ-thick) on multiple passing of the material through the rolls, sieve analysis of the ground material was largely meaningless; however, subsequent engineering-scale leaching tests indicated that very nearly all coatings had been ruptured. In these tests, 0.35% of the uranium and 0.67% of the thorium were lost to the water-washed graphite residue. The mixed leachate-wash composite solution was 0.17 M in Th, 0.035 M in U, and 6 M in H^+. The losses of uranium and thorium to the graphite residue were further reduced to 0.2 and 0.5%, respectively, by

an equal volume of additional washing. The permeability of the crushed fuel to acid and water ranged from 4 to 25 gal hr^{-1} ft^{-2} at a pressure gradient of about 18 psi per foot of bed depth. These results were confirmed by parallel laboratory tests in which uranium and thorium recoveries of about 99.7 and 99.4%, respectively, were achieved by leaching ground fuel (−140 mesh) for 5 hr with either boiling 13 M HNO_3 or 13 M HNO_3–0.05 M HF and then washing the residue three times with water. The volume of each wash was equal to the volume of leachant used. After leaching, 20 to 50% of the carbide carbon was found in the solutions as soluble organic species. This result was expected from previous studies with pure carbides and shows that the finely powdered graphite was not attacked appreciably by the acid during leaching. These preliminary results indicate that a grind-leach process for graphite-base fuels is potentially feasible.

Aqueous Process Development: Advanced Fuels. – Methods are being developed on a laboratory scale for processing fuels proposed for use in heavy-water-moderated, organic-cooled reactors. One reference fuel contains UC clad in SAP (a sintered dispersion of Al_2O_3 in aluminum). A possible processing method for this fuel consists in shearing the fuel, contacting the sheared pieces with steam at high temperature, and dissolving the residue in nitric acid. Reaction with steam (pyrohydrolysis) converts the UC to UO_2 and results in removal of the carbide carbon, primarily as CO_2.

To simulate a pyrohydrolysis-leach process, UC and SAP (at a UC/SAP weight ratio of 8) were exposed to steam for 4 hr at 750°C, and the residue was subsequently digested for 5 hr in boiling 8 M HNO_3. The final solution contained 97.2% of the aluminum from the SAP and practically all the uranium from the carbide. The solution had a composition of about 1 M $UO_2(NO_3)_2$, 1 M $Al(NO_3)_3$, and 0.7 M HNO_3. The high percentage of SAP dissolved is surprising in light of earlier, less promising results. Consequently, attempts to duplicate the above results will be made, with emphasis on explaining the high rate of dissolution of the SAP after exposure to steam.

Aqueous Process Development: Protactinium Adsorption on Unfired Vycor. – Additional hot-cell experiments to demonstrate the adsorption of ^{233}Pa on unfired Vycor glass and subsequent elution with 0.5 M oxalic acid were performed to obtain additional information on loading and elution characteristics, the effect of Vycor mesh size, and the effect of cyclic loadings and elutions. Three Zircaloy-2-clad rods containing vibratory-compacted sol-gel ThO_2 were irradiated in the ETR to 5000 Mwd/ton and then dissolved and processed after only 28 to 39 days of decay. Preliminary results, using 60 to 80 mesh Vycor, showed that 95 to 97% of the protactinium had been adsorbed from the filtered feed solution [0.5 M $Th(NO_3)_4$, 10 M HNO_3, 0.1 M $Al(NO_3)_3$, 0.05 M F, containing 0.084 mg of ^{233}Pa per ml] when the initial protactinium breakthrough reached 10% of the feed concentration. Loading of the Vycor column averaged about 3.5 mg of ^{233}Pa per gram of glass. The adsorbed protactinium was successfully eluted with 0.5 M oxalic acid, and the same column was reloaded with additional protactinium without difficulty.

Fluoride Volatility Processing: Tests on Chemical Decladding and Fluorination of Stainless-Steel-Clad UO_2 Fuel in a Fluidized Bed. — In the volatility process proposed for stainless-steel-clad UO_2 fuels, the cladding is removed by reaction with a mixture of HF and oxygen in a fluidized bed of alumina, and the uranium is subsequently volatilized as the hexafluoride by reaction with fluorine.

In recent tests with a 1-in.-OD fluidized-bed reactor, one of the important factors studied was the optimization of fluorination conditions to get minimum lumping of uranium material and to achieve complete comminution of the stainless steel fluoride (SSF) residue. The possibility of reuse of the alumina, the behavior of chromium, and entrainment of material into the disengaging section of the reactor were also investigated. In a 2-in.-OD reactor, larger-scale tests were made with 12-in.-long four-tube assemblies of Consolidated Edison prototype fuel to define operating conditions for different sizes and types of alumina and to study the operation of analytical in-line instrumentation.

In the 1-in.-OD reactor tests, a ratio of alumina to fuel of 2.3 to 1 was used with 40 g of Norton RR fused alumina. The fuel consisted of small UO_2 plates wrapped in 5-mil type 302 stainless steel. The alumina was nominally 90-mesh material without the -100 mesh particles. Fluoride-catalyzed oxidation of the stainless steel was by a standardized method: 40% HF—60% O_2 for 30 min, then 20% HF—80% O_2 for 60 min. Subsequent treatment with fluorine at either high initial concentrations or at initial temperatures of 600 to 650°C resulted in sintering due to the rapid reaction of U_3O_8 fines. Use of a low fluorine concentration at 450°C gave no lumping of uranium-containing material, but complete pulverization of the SSF residue was not obtained. In boat tests, nearly complete comminution of the SSF residue was attained only by using a low concentration of fluorine at 450°C, followed by higher concentrations. For fluidized beds the fluorination program adopted was: for the first hour, with the reactor at 450°C, increase the concentration of fluorine in helium from 10 to 70%; then for the last 3 hr, use 70% fluorine at 550°C; maintain superficial gas velocity at about 0.7 fps. Tests of this program resulted in a free-flowing bed with no sintering of alumina, good SSF pulverization, and over 99.9% uranium volatilization.

One experiment was made using a second cycle of decladding and fluorination in the original bed but with a fresh batch of fuel. The SSF's and about 10% of the first charge of uranium remained in the bed after the first cycle. No difficulty was encountered, but at the end of the run about 5% of the SSF was present as a fine powder coating the disengaging section. Such a fine powder could sorb a significant amount of plutonium. Except for this possible difficulty with the fines, reuse of the bed presents no difficulties.

In the larger-scale tests with the Consolidated Edison prototype fuel, three runs have been completed. These runs included decladding with 40% HF—60% O_2, followed immediately by fluorination.

One run with −35 +70 mesh Norton RR alumina produced a free-flowing bed containing less than 0.5% of the original uranium. A very small amount of porous sinter was formed, as a result of starting the fluorine flow before the fluidizing flow of diluent nitrogen had been started. Two other tests with −100 +140 mesh alumina, Norton RR in one case and Norton abrasive grain in the other, were unsuccessful because of failure to complete the decladding reaction.

The gas chromatograph was operated during the last two runs and furnished data on HF/O_2 and F_2/N_2 ratios in the effluent gas during decladding and fluorination respectively. During one test, the ultraviolet spectrophotometer was operated to monitor UF_6 in the fluorination effluent; because of the decladding failure, no UF_6 was produced in fluorination, and the test was of no value.

Results of the tests, along with earlier work, indicate that satisfactory process conditions can be specified if gas velocity and concentration and run time are related to the requirements of the fluidizing medium. Also, maintaining an adequately low ratio of fuel to alumina is very important.

Concentrations of reactant gases and UF_6 product in the reactor offgas can be measured by in-line analytical instruments. Transporting a representative sample to instruments remote from the reactor without introducing undesirable lag time is difficult. Procedures for sampling are being refined so that they can be specified for the Fluidized-Bed Volatility Pilot Plant.

Fluoride Volatility Processing: Separation of UF_6 from Bromine Fluorides by NaF Sorption. – Studies on the evaluation of sorption by NaF as a means for separating UF_6 from BrF_5 and BrF_3 in the interhalogen fluoride volatility process were continued. Currently, UF_6 delivered to the AEC may not contain more than 5 ppm of bromine (30 Fed. Reg. 14821, Nov. 30, 1965).

The experiments consisted generally in fluorinating 2 g of UF_4 in a boat for 2 hr at 500°C with BrF_5 and then sorbing the product UF_6 in a 4-g bed of granular NaF (Harshaw Company pellet material, prepared in an activated form by decomposing $NaHF_2$). Various modifications of this method were used to show that bromine contamination of the UF_6-NaF bed is due probably to BrF_5 and to BrF_3, the latter being formed by reduction or dissociation of the BrF_5 or present as an impurity in the BrF_5. Bromine trifluoride is believed to form a complex with NaF. The pentafluoride apparently forms no complex at the sorption temperature of 150°C. The use of small NaF particles and fluorine-pressure pulsation cycles were studied as a means of minimizing bromine contamination by conversion of any residual BrF_3 in the NaF back to BrF_5.

A test with −12 +20 mesh NaF gave a bromine containation of 44 ppm, based on the weight of sorbed UF_6, using a 1-hr fluorine flush period after the BrF_5 step. Changing the particle size of the NaF to −20 +40 mesh gave a bromine contamination value of less than 18 ppm. Further improvement in the desorption of residual BrF_3 was obtained by ten cycles of pressurization and depressurization of a −12 +20 mesh NaF bed in the 1-hr fluorine treatment step. This gave a final bromine contamination value of less than 14 ppm. In a special run, using no uranium, 50% BrF_5–50% F_2 was passed through a 1-in.-OD reactor held at 500°C into a −12 +20 mesh bed of NaF at 150°C. The final bromine content of the bed was less than 20 ppm.

These results indicate that probably almost any degree of separation of bromine from UF_6 is achievable by the NaF process. The bromine contamination is apparently due to either BrF_3 or BrF_5 carried into the interior of the NaF particles along with the sorbed UF_6. Cyclic fluorine pulsation or use of small NaF particles minimizes this effect. The use of fluorine evidently reconverts BrF_3 to BrF_5 and minimizes formation of a BrF_3-NaF complex.

Fluidized-Bed Volatility Pilot Plant (FBVPP). — Design continues and fabrication of equipment has begun for the FBVPP, to be installed in cells 1, 2, and 3 of Building 3019. The processing of zirconium-clad and stainless-steel-clad UO_2 power-reactor fuel elements will be studied there.

The design of the main cold traps was completed, and fabrication was begun. Fabrication of the backup filter for the off-gas from the primary reactor has also started. The last tests made at ORGDP of nickel filter elements gave satisfactory results, and we can now place an order for both nickel and Inconel elements. In these tests, ten cycles of a sequence of HCl and hydrogen (5 hr, 315°C), dilute oxygen (10 hr, 100°C), and dilute fluorine (10 hr, 100°C) were run, for a total of 250 hr.

A preliminary flowsheet for phase 2 (separation and purification of plutonium and uranium fluorides) was issued.

Removal of equipment from cell 3, Building 3019, has started. FBVPP head-end equipment will be located in this cell. Equipment from the old Molten-Salt—Fluoride-Volatility Pilot Plant is being dismantled and removed from cell 1, where processing of solid wastes from the FBVPP will be studied next. All equipment has now been removed from the upper portion of the cell, and the lower portion is being cleaned.

Critical items of equipment from cell 1 are being examined to evaluate corrosion. Thickness measurements of the dissolver wall confirmed the conclusion from in situ measurements (by pulse echo) that the rate of corrosion during aluminum processing was not greater than (and probably significantly less than) that during zirconium processing.

FUEL-CYCLE STUDIES

Extrusion of Sol-Gel Fuels. — We are investigating the extrusion of clays derived from sol-gel material as a method of making ceramic fuels. We hope that this will lead to an economical way to make small-diameter fuel rods remotely. The effect of reducing the moisture content of a 10 M ThO_2 clay by slow air-drying before extruding has been studied to see whether shrinkage that occurs on firing could thus be lessened. It is felt that such shrinkage leads to microcracking. Unfortunately, drying did not help and may have made the results worse. Other ways of dealing with this problem will be studied.

Microsphere Forming Studies. — Gel microspheres of thoria, urania, and thoria-urania mixtures are formed by extracting water from sol droplets as they fall through a tapered column containing 2-ethyl-1-hexanol flowing countercurrently. It is necessary to add surfactants to the 2-ethyl-1-hexanol to prevent the sol droplets from sticking to each other or to the column, and to keep them from being malformed. Also, the flow of 2-ethyl-1-hexanol up the column must be modified by proper column design so that channeling of the gel microspheres, leading to insufficient residence time in the column, does not occur. In a continuing study, we are searching for better surfactants and improved column designs. We have screened a variety of alcohols, esters, amines, and proprietary surfactants but have found none as good as the Span 80 and Ethomeen S/15 we are already using.

Alternatives to the fluidized bed are being considered for suspending sol drops during extraction of the water to cause gelation. Long lengths of polyvinyl chloride tubing (100 ft in one set of tests and 200 ft in another) were used to provide residence time for the suspended drops. When the tubing was wound on a cylinder about 18 in. in diameter, the drops settled to the wall and coalesced wherever the tubing was not vertical. Operation was very poor with the axis of the cylinder vertical, but reasonably good with the axis horizontal. Settling was further reduced by using 18-in. vertical runs connected by bends of 3-in. radius at the top and bottom. From the results observed, the use of a coiled tube appears less attractive than vertical stages of moderate velocity connected by short transfer tubes of reduced cross section. In preliminary tests, three 6-ft vertical stages successfully gelled ThO_2 spheres of up to 300 μ diameter after calcination.

Preparation of Sol-Gel Thorium Dicarbide Microspheres. — Thorium dicarbide microspheres can be made from mixed thoria-carbon sols by dispersing the sol in 2-ethyl-1-hexanol containing surfactants. This method leads to a relatively low-density product (< 90% of theoretical), and the results of early studies suggest that complete removal of the last traces of ThO_2 may be difficult. Present studies are designed to see what control can be achieved over residual oxide (or free carbon) and density in the fired dicarbide product.

In the past month, numerous surfactants were investigated for improved sphere-forming properties. It was found that only a Span 80–Ethomeen S/15 combination gave completely acceptable gel spheres. This combination was used to prepare ThO_2-C gel microspheres for use in studies on thorium nitride and sulfide preparation. In other studies, several preparations of gel spheres containing different ThO_2/C ratios were fired at temperatures up to 1900°C, and the products were analyzed to determine the extent of conversion to monocarbide and dicarbide and the amount of unreacted carbon. Results indicate that about 0.123 mole of carbon per 100 g of starting material was consumed in side reactions (presumably with residual nitrate ion, present in the original sol). Incomplete conversion to dicarbide in the presence of excess carbon suggests that higher temperatures or longer reaction times may be required to obtain complete conversion.

Vibrational Packing of Microspheres. — A laboratory investigation of low-energy vibrational packing of microspheres has been started to determine the optimum ratio of large and small spherules to obtain high packed densities. So far, particles having diameters <35 and >40 mesh have been packed with <325-mesh particles. For this system, a ratio of 37/13 for the coarse/fine fractions gave the highest densities, which were 83 to 85% of the theoretical density of the pure, crystalline material. Such packed densities are reasonable to accommodate thermal expansion and fuel swelling, and the studies will be extended to include different sizes for the coarse and fine fractions.

Thoria-Urania Sol Preparation. — The objective of these studies is to extend sol-gel technology to include mixtures of thoria and urania in all ratios. The original sol-gel process is limited to mixtures containing less than 10% uranium, which is added to thoria sol as uranyl nitrate

solution or UO_3 slurry. During the past month, ThO_2-UO_2 (Th/U = 3.5) sol was prepared by blending a paste of steam-denitrated thoria containing excess nitrate ion with a UO_2–5% ThO_2 paste containing insufficient nitrate ion to cause peptization. The mixture immediately became a somewhat viscous sol, which was formed into excellent-appearing 300-μ-diam gel microspheres by dispersing it in 2-ethyl-1-hexanol containing Span 80 and Ethomeen S/15 surfactants. Firing studies of these gel spheres are in progress.

Thorium-Uranium Recycle Facility. – This facility (TURF) is now about 40% complete. TURF is to provide sufficient shield space for the development of remote processing and fabrication of Th-^{233}U fuel assemblies of various types. All the facility design and procurement of items are progressing satisfactorily with the exception of the shielding doors, which are a little behind schedule.

Sol-gel process equipment design effort was concentrated on examining and revising drawings of liquid-handling equipment. Preliminary flowsheets were drawn, and critical items are being identified for the microsphere-forming system.

^{233}U Storage and Distribution. – Oak Ridge National Laboratory serves as a national storage and distribution point for ^{233}U. A processing facility is being installed to permit cleanup and packaging of ^{233}U in various forms as requested by users. The ^{233}U solution storage system, consisting of five tanks, each capable of storing 80 kg of ^{233}U at a concentration of 180 g/liter, was placed in service during the month. Installation of the geometrically safe ^{233}U dissolver, capable of dissolving 5 kg of $^{233}UO_2$ per day, is almost complete.

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

The microsphere-forming column was operated with both a rotary disperser and two-fluid nozzles during this report period. The rotary disperser, which had 12 0.010-in.-diam orifices, produced an undersirable amount of fines at 270 rpm, at which rate the spherules after firing were 240 to 300 μ in diameter. Smaller orifices are apparently needed to make smaller spheres. Two-fluid nozzles produced them in the range 210 to 280 μ in good yield, so these nozzles will be used to produce 210- to 250-μ-diam spheres for use in coating studies.

Other work performed in the CPDL during this report period was directed toward a study of the effects of changes in the design of nozzles in the particle coater and toward coater component evaluation and the coating process itself. With one nozzle studied, a considerable amount of bed slugging was observed over the range of gas flows used. The resulting coating was somewhat faceted, indicating that a slugging bed gives inadequate particle motion. With another nozzle, soot buildup caused early termination of the run, but good sphericity was achieved. From these experiments and others, we concluded that a dual-flow multiorifice graphite nozzle is the best system we have studied.

We are developing an in-line process monitor for measuring the average thickness of the pyrolytic carbon coating on ThO_2. X-ray attenuation

was used for measuring the thicknesses. Work during this report period was directed toward improving the multiplier phototube voltage-divider network. We were able to reduce the required x-ray energy from 85 kvp to 50 with no loss in sensitivity, making it possible to consider the use of the 60-kv x rays from ^{241}Am.

Gas Evolution from Sol-Gel Thorium Oxide Fuels. – Previous studies have been concerned with gas evolution from chunks of ThO_2–3% UO_2, prepared by the sol-gel process and suitable for vibratory compaction into fuel rods. Recently, the emphasis of the work has changed, and now gas evolution from ThO_2 microspheres formed by the ORNL sol-gel process is being studied. Excessive release of gas from such fuels could adversely affect their performance in reactors. Preliminary studies have shown that, although large amounts of gas (more than 20 cm^3 per gram of ThO_2) are evolved at temperatures up to about 260°C, a relatively small amount (about 0.27 cm^3/g) is evolved above 760°C. The gas is primarily CO_2, CO, and H_2, a composition similar to that found in the sol-gel chunks.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. – The problem of excessive temperatures above the radiator, which has delayed operation of the coolant system, was overcome. Air leakage from inside the radiator was reduced, and ducting was installed to draw cooled ventilation air through the space between the door hoods. Heater leads and thermocouple junction boxes were moved to cooler locations, and high-temperature electrical insulation was installed where necessary.

Fuel salt was circulated continuously at 1200°F except for a three-day period following an accidental drain. (The drain valve thawed faster than expected when cooling air was stopped as part of a control circuit checkout.)

After the radiator was ready, the coolant loop was filled and circulation was started. The program of power calibrations and dynamics experiments, which had been restricted to 10 kw or less before the coolant system was operative, was continued as the power was raised. On January 24, operation at 1 Mw was begun, in a program of gradual increases in power until the reactor is operating at the design level of 10 Mw (thermal).

Metallurgy. – A short section of graphite pipe and a graphite-to-metal joint successfully contained molten salts for 500 hr at 700°C and 150 psig. The graphite pipe was machined to 1-1/4 in. OD by 3/4 in. ID from a bar of MSRE graphite. Molybdenum end caps were brazed to the ends of the pipe, using braze metal ANM-16 (35 wt % Au–35 wt % Ni–30 wt % Mo).

Molten salt was fed to the inside of the graphite pipe through an Inconel tube brazed into the top molybdenum end cap. The salt was pressurized with an argon cover gas, and a vacuum of <5 μ was maintained on the outside of the graphite.

The test monitoring system indicated that no leak occurred during the 500-hr test period, and posttest radiographic examination indicated

that the salt was contained as planned. Metallographic examination revealed no penetration of salt in the graphite and no detectable corrosion of the braze material.

This test, which operated satisfactorily despite a primitive joint design and a relatively thin-walled graphite pipe, encourages us to believe that larger graphite tubes and graphite-to-metal joints can be developed for the molten-salt breeder reactor.

MSR Processing: Design Studies. — A preliminary design and engineering evaluation (ORNL-3791) was made of a plant for treating the fuel and fertile streams of a molten-salt breeder reactor. The plant was designed to operate continuously as an integral part of a 1000-Mw (electrical) power reactor; the required plant capacity is 15 ft^3/day of fuel salt and 105 ft^3/day of fertile salt.

In the flowsheet on which the study was based, the fuel stream is fluorinated for removal of uranium and distilled semicontinuously under vacuum for removal of rare-earth and other nonvolatile fluorides. Fission products volatilized with the uranium during fluorination are removed by passing the gas stream through beds of NaF and MgF_2. The fuel salt is then reconstituted by combining the purified UF_6 with salt recovered from the distillation step and with makeup salt; after filtration it is returned to the reactor. Treatment of the fertile stream consists only of fluorination followed by decontamination of the UF_6 on NaF and MgF_2 sorbents. Discard of a small portion of the fertile stream suffices to keep the fission product concentrations at acceptable levels.

The chief conclusions of the study are: (1) the proposed processes comprise a compact and relatively simple system that can be engineered with a normal amount of developmental work, and (2) integration of the processing plant into the reactor facility is both feasible and economical and is the logical way to take advantage of the processing possibilities of a fluid-fueled reactor. The capital and operating costs were found to contribute about 0.2 mill/kwhr to the fuel cycle cost when the reactor operates at an 80% plant factor and capital charges are 10%/year.

MSR Processing: Relative Volatilities of Rare-Earth Fluorides in LiF. — A proposed method for removal of rare-earth fluorides from the fuel stream of a molten-salt breeder reactor (MSBR) is semicontinuous vacuum distillation. One measure of the potential for this process is the relative volatility of the rare-earth fluorides as compared with the less volatile fuel salt; this quantity is defined as

$$\alpha_{AB} = \frac{Y_A/X_A}{Y_B/X_B} ,$$

where α_{AB} = relative volatility of component A compared with component B,

Y_i = mole fraction of component i in vapor phase,

X_i = mole fraction of component i in liquid phase.

Measurements of relative volatilities of several rare-earth fluorides in LiF have been completed. The experimental method consisted in heating a mixture of LiF and rare-earth fluorides in a 1-in.-diam nickel

vessel to the desired temperature in the presence of an inert gas at 1 atm. The still was then evacuated to 0.1–1 mm Hg, and after the temperature and pressure in the system had reached an apparent steady state, a surface located approximately 4 in. above the liquid surface was quickly cooled to below the melting point of the salt for 2 to 4 min, during which time a vapor sample consisting of ~0.01 g of salt was collected. At this point, sample collection was terminated by raising the system pressure to 1 atm by introducing inert gas. Relative volatility values, calculated from analyses of liquid and vapor samples, are as follows:

Relative Volatilities of Rare-Earth Fluorides in Lithium Fluoride

Rare-Earth Fluoride	Liquid Mole Fraction	Average Relative Volatilities			
		900°C	950°C	1000°C	1050°C
CeF_4	0.0067	0.133	0.167		0.208
SmF_3	0.01	0.033			0.009
NdF_3	0.01	0.025			0.016
PrF_3	0.001		0.038	0.020	0.014
EuF_3	0.001	0.041	0.037	0.028	0.012
CeF_3	0.01	0.043	0.033		0.018
LaF_3	0.001	0.035	0.024		
LaF_3	0.01	0.051	0.027	0.011	0.008

MSR Processing: Fuel Reconstitution. — A step in the current method for processing the fuel stream of a molten-salt breeder reactor is that of combining purified UF_6 with molten salt from the distillation step. A proposed method is that of contacting the UF_6 with molten salt containing UF_4, which results in absorption of the UF_6 by the formation of a nonvolatile intermediate fluoride of uranium such as UF_5. The intermediate fluoride is then reduced to UF_4 by contacting the mixture with hydrogen.

Three experiments were carried out at 600°C; in these experiments UF_6 was introduced at the rate of 1.5 g/min through a 1/4-in. tube at a point 12 in. below the surface of a molten $LiF-ZrF_4$ mixture containing ~0.5 mole % UF_4. Complete absorption of the UF_6 was observed during each of the tests, in which 147 g of UF_6 was absorbed in 98 min.

MSR Processing: Chromium Fluoride Trapping. — At the conclusion of tests on the MSRE, uranium will be recovered from the fuel salt as UF_6 by sparging the salt with F_2. Fluorides of chromium will be present in the fuel salt as a result of corrosion of reactor piping and of equipment used for hydrofluorination or fluorination of the salt. During the recovery of the uranium, volatile fluorides of chromium (CrF_4 and CrF_5) in the fluorinator off-gas contaminate the UF_6 product and also render equipment inoperative by depositing in lines, valves, etc. Data obtained in a recent study will permit the design of a trapping system for removing these fluorides from the off-gas, which will also contain UF_6 and F_2.

Experiments were carried out in which 1 liter (STP) of F_2 per minute was sparged through a molten $NaF-LiF-ZrF_4$ mixture at 650°C which contained

0.5 to 4 wt % CrF_3. The resulting off-gas containing fluorine and volatile fluorides of chromium then passed through beds of pelleted NaF at 400°C for removal of chromium fluorides. In some tests, a UF_6 flow of 100 cm^3/min was added to the F_2.

It can be concluded that:

1. Fixed beds of NaF at 400°C are effective in removing fluorides of chromium from a gas stream which also contains UF_6 and F_2.
2. Pelleted NaF having a surface area of 0.074 m^2/g and a void fraction of 0.277 is superior to material having a surface area of 1 m^2/g and a void fraction of 0.45, and has an effective capacity of about 20 g of chromium per 100 g of NaF.
3. Uranium losses to the 400°C NaF bed of less than 0.01 % are achievable when working with a gas stream that contains 0.4 mole of CrF_5 per mole of UF_6 in F_2.

MSR Processing: Removal of Fission Product Zirconium. — The processing methods presently being considered for the MSBR, fluorination and low-pressure distillation, will not remove fission product zirconium sufficiently to prevent it from being a significant poison. At least 40% should be removed per pass through the process, and some influence on fuel cycle cost is seen until 90% per pass is removed. Bench-scale studies have been made on an alternative method using lithium or beryllium metal at 550 to 800°C to partially reduce the constituents of the salt, forming precipitates which can be separated by filtration. Acceptable decontamination factors of 30 to 2200 were obtained in all tests. These covered a range of initial zirconium concentrations from 60 ppm to 11.7%. The low-level analyses of the treated salts were run by neutron activation analysis, and, in most cases, the final zirconium concentration was at or below the detectability limit. In tests involving high concentrations of zirconium reduced by lithium, the zirconium-bearing solid phase was identified at room temperature to consist largely of α-zirconium metal.

Analysis of Radioactive MSRE Fuels. — The MSRE has operated for some 300 hr at power levels below 25 kw, in preparation for the full-power experiment. During this period the fuel salt was sampled regularly and chemically analyzed.

Significant advances in analytical methods have been incorporated into routine practice in the laboratory since the zero-power experiment was completed in July 1965. The increased accuracy which is apparent in current chemical analyses shows more clearly than ever that composition and purity of the reactor salts can be ascertained accurately and economically on a routine basis and that current methods now afford excellent monitoring practices to molten-salt reactor technology. Of greatest significance is that oxide content of the salt can now be determined routinely and accurately at concentrations lower than 100 ppm, by purging samples of the molten salt with H_2 and HF. Oxide concentration is then calculated from the amount of water recovered from the effluent gas stream (ORNL-3782).

Samples of flush and fuel salt taken during the December startup of the MSRE were analyzed by the hydrofluorination method. The results are summarized below:

	Time of Fuel Circulation (hr)	Oxygen (ppm)
Flush salt	24	47
	29	72
	47	106
Fuel salt	4	120
	24	85
	32	106[a]
	52	66

[a]Slightly exposed during sampling.

These results were in excellent agreement with analyses made by the $KBrF_4$ procedure on samples taken during the same period. $KBrF_4$ values paralleled the trends shown by hydrofluorination analysis and averaged 20 ppm higher, a not unexpected bias, since the pulverized samples required for the $KBrF_4$ determination are easily contaminated by atmospheric moisture. The $KBrF_4$ method gave very erratic results last summer when the humidity was high. Tests have shown that consistent results can be obtained by the hydrofluorination method during periods of widely varying humidity.

A refinement in the method of determining uranium concentration in the MSRE fuel was recently introduced into routine practice and has reduced the apparent bias [MSR-65-40 (internal memorandum)] between computed inventory figures and chemical analysis to less than 0.4%. In the past, values of uranium concentration, as derived by the controlled-potential coulometric method, were determined by titration to a 5-μa background. The accuracy of this method is now improved by inferring the 5-μa end point on extrapolation from the 50-μa level. The average value for the concentration of uranium in the fuel using this new method is 4.652 wt %, within 0.4% of the inventory value.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. – The first fuel assembly has been received from the fabricator. It conforms completely with the specifications. The second and third assemblies have been assembled and welded and are now being final machined. Both of these elements also meet the plate spacing specification. The average plate spacings have been increased from 48.3 mils found in both elements of the first assembly to 49.3 mils for the inner and 49.0 for the outer element.

Work has started to add spacers to the present HFIR fuel elements. Both round wires and flat spacers with a thin lip have been attached to a plate by ultrasonic welding. However, the bonds have not yet been made reproducible, some being good and others hardly attached down a single strip.

Erratic results have been encountered in the preparation of the europium oxide for the control rod plates. Three production batches have been purchased from the same company and to the same set of specifications. The original material was arc melted with no difficulty, and a single melting was sufficient to furnish adequate material. A subsequent batch outgassed badly, making it very difficult to maintain the arc, and also reacted with the mold, causing frequent replacement. Three or four meltings were required for this material. The last batch delivered acted like the first and was satisfactory. The impurities causing these differences have not yet been determined.

Component Development and Testing. — The performance of the target tower assembly and the target holder assembly during the shakedown flow test (November 9 through November 23) indicated that these components were unstable at normal HFIR operating flow rates. A test program was initiated to pinpoint the cause of the instability and to provide the information necessary to establish design criteria for modification of the existing tower design to eliminate instability.

As a result of these tests, the target tower and the target tower spider have been clamped together to provide a rigid assembly in order to eliminate relative rotation of these two parts. The target holder assembly will be strengthened and equipped with self-contained clamps to retain the target rods, instead of relying on the weight of the target tower to clamp the rods. In addition, the rather flexible linkage connecting the target holder and target tower assembly will be eliminated. The tower and the target holder will be handled separately, thus providing the reactor operator with direct viewing and feel while installing and removing the target holder.

The primary coolant strainer has been received from the vendor and is installed in the reactor primary piping system. This strainer has operated satisfactorily for approximately 300 hr.

Heat Transfer Analysis. — In conjunction with the modification of the HFIR heat transfer analysis to include more sophistication and thus perhaps remove some of the conservatism, the possibility of using an additive burnout correlation for estimating the burnout heat flux has been investigated. The advantage in using this technique is that the calculated difference between incipient boiling and burnout remains consistent over the full range of operating conditions, whereas the use of separate correlation sometimes did not. In the present analysis it is conservatively assumed that burnout occurs at the same moment that incipient boiling occurs. The modified analysis will attempt to take advantage of the actual difference between incipient boiling and burnout, at least for transient cases. For nominal HFIR conditions the difference is about 40%.

A contemplated modified core design will include axial spacers in the fuel element coolant channels. A literature search was made to obtain information on the effect of spacers on burnout heat flux. An extrapolation of experimental data obtained at SRL indicated that the reduction in burnout heat flux associated with the addition of 0.050-in.-diam axial spacers to a dimensionally perfect channel would be about 6%. The advantage gained by controlling the thickness of the coolant channel with the spacers will be much greater than 6%.

Reactor Operations. — Critical experiments have been conducted by using various combinations of the HFIRCE-3 first-production and second-production fuel elements with the standard and first-production control rods. The postneutron critical experiments are essentially finished, and final preparations are under way to commence power escalation.

The experimenter's collimator and shield plugs and the rotating shutters were installed in HB-2 and HB-3. Low-intensity beam measurements were made by personnel from ORNL and ANL. High-intensity measurements will be made during power escalation.

A 10-in. bypass valve has been installed around the main secondary valve to provide better control of the secondary water flow through the primary heat exchangers. During the period of conducting the critical experiments, considerable experience was gained in remote handling of the reactor components; many improvements were made in the remote handling techniques as a result of this experience. The HFIR is now being manned three shifts a day, seven days a week.

Fabrication of the shipping cask for spent fuel elements is complete.

PHYSICAL RESEARCH

Reactor Operations. — The ORR operated approximately 89% of the time in November and December. One unusual experiment was begun, and two significant improvements were completed:

1. Irradiation of nine ^{241}Am samples for production of curium.
2. Operation of a demineralizer in series with the primary water degasifier. This further reduces the contamination escaping from the primary system to the pool water.
3. A new quick-opening access cover was installed on the reactor tank. This not only reduces the time required for changing fuel, but provides a much better seal and reduces the chance of contamination reaching the pool.

Small reactor power oscillations were observed and were determined to be flow-dependent. It was concluded that the oscillations were due to movement of one of the shim rods at certain positions and at certain water-flow conditions.

Waste Disposal. — The amount of radioactivity released to the environment in liquid waste continued its downward trend in 1965. The average monthly release of strontium to White Oak Lake in 1965 was 450 mc as compared with an average of 600 mc in 1964, and of this, only 120 mc, or 27%, came from the process waste. The remainder came from miscellaneous sources, probably mainly from the White Oak Creek bed, which was contaminated years ago when the Laboratory discharges were relatively high. Ruthenium, accounting for 84% of the total release of radioactivity, was reduced from an average of 30 curies/month in 1964 to 12 curies/month in 1965.

The ^{131}I released from the gaseous waste systems was an average of 1.4 curies/month in 1965, compared with 7.0 curies/month in 1964. The particulate activity in 1965 averaged 39 mc/month, compared with 10 mc/month in 1964; 87% of the particulate activity in 1965 was ^{137}Cs and the remainder ^{131}I.

Hot-Cell Operations. — Sixty-one high-burnup UO_2 fuel pins, some clad in Zircaloy and some clad in stainless steel, were examined; these pins will be inserted into the ORR for a short irradiation and then used in melt experiments in the Nuclear Safety Pilot Plant. The pins were cleaned, gamma scanned, checked dimensionally, and placed in an autoclave at 285°C for 24 hr; the pins were then weighed again, and any weight gain greater than 1 part in 10,000 was cause for rejection.

ORR-AGCR capsule P4A-1 was examined; this included visual examination, photography, dimensional measurements, gamma scan, and metallography. Experiment MTR 43-89, containing multiple compartments of coated particles, was disassembled and examined. The No. 4 TRU target rod was visually examined, gamma scanned, and partially disassembled, and the fission product heat generation was determined. One NSPP meltdown (fuel spiked with radioisotopes) and one in-pile UO_2 meltdown were disassembled. Examination of Army fuel element PM1-M2 continued, and examination of ThO_2-UO_2 capsule U-5 was begun.

Diameter and bow measurements were made on the LITR beam-hole liner from HB-2 to aid in fabrication of another liner. The corrosion specimens were removed from the Volatility Pilot Plant fluorinator, and samples for metallurgical examination were cut from the vessel walls. In-cell creep test machines and tensile test machines continued in full operation.

PHYSICS AND MATHEMATICS

Charge Spectrometry. — When an x ray interacts with an atom, it usually does so by the photoelectric ejection of an electron from one of the inner shells of the atom. In the readjustment to this vacancy, mainly by a series of Auger processes, extensive ionization can occur. A comprehensive study has been in progress for examining the charge spectra resulting from x irradiation of gaseous atoms in a specially designed mass spectrometer. Besides offering an opportunity to understand the basic nature of multiple ionization, the knowledge of how much ionization occurs when an inner shell vacancy is formed should be of intrinsic value to those concerned with radiation damage and its detection. With this second goal particularly in mind, studies were made on xenon and mercury.

The relative abundances of the differently charged ions were measured following the x irradiation of xenon ($Z = 54$), and in separate experiments of mercury ($Z = 80$). These studies were carried out for a variety of x-ray energies, so as to obtain data as a function of the initial inner shell vacancies. From the data we derived the charge spectra that are the consequence of producing an initial vacancy in each of the following shells: the K, L_I, $L_{II,III}$, M_I, $M_{II,III}$, $M_{IV,V}$, and N shells of xenon and the L, M, N, and O shells of mercury. These data were correlated with earlier measurements on He, Ne, Ar, and Kr; and empirical rules were set up, whereby one may estimate the average charge resulting from the atom readjusting to a vacancy in any shell of any atom.

Theoretical Physics: Two-Nucleon Interaction. — At present the most accurate fits to the two-nucleon data by potential models are still those of G. Breit *et al.* (Yale) and of Hamada and Johnston (HJ), which include phenomenological hard repulsive cores. When expressed in a common form the values of the two potentials differ at most by a few percent outside nearly identical core radii. Both give about 8 Mev/nucleon of binding in nuclear matter. One might expect that the two potentials would give similar results for properties of nuclei. However, a Brueckner theory calculation of the ground-state binding energy, single-particle energies, and matrix elements of the two-body reaction matrix (effective nucleon-nucleon interaction) in ^{16}O, using an extension of the Eden-Emery method, gives significantly different results for the two potentials. For example, the HJ potential gives about 3 Mev/nucleon more binding. The main source of these differences is the slight difference in core radii. When the core radius of the Yale potential is reduced slightly to the HJ value,

the calculated quantities become very similar to those obtained with the HJ potential. The greater sensitivity to the core radius in finite nuclei as compared with nuclear matter is caused by the presence of the shell-model potential, which enhances the probability of two nucleons being close together. A slight decrease in core radius results in a significant increase of the correlation function throughout the region of the attractive part of the potential. This work indicates the rather great sensitivity of the effective nucleon-nucleon interaction in nuclei to the details of the free two-nucleon interaction and a need for increased accuracy of the latter.

Theoretical Physics: Optical-Model Analysis of the Scattering of 24.7-Mev Alpha Particles. — The measured elastic scattering of 24.7-Mev alpha particles from the nuclei O, Mg, Al, Si, Ca, Mn, Ni, Co, Cu, Ge, Zr, Ag, In, Sn, Hf, W, Au, Bi, and U was analyzed using the optical model. Fits were obtained using a four-parameter Woods-Saxon potential. No marked improvement was found by using a more general potential. The fits for medium and heavy nuclei are good, but those for the lighter nuclei are noticeably poorer. Potential ambiguities were studied in some detail for nickel and magnesium, and potentials with depths of about 150 and 200 Mev were obtained for all the nuclei in addition to a more shallow potential. The medium-weight nuclei show a preference for the deep potentials, the light nuclei for the shallow ones. The behavior of the predicted scattering matrix elements was also investigated.

Theoretical Physics: Hartree-Fock Calculations for Finite Nuclei. — A velocity-dependent two-body potential of the type investigated by Green, but acting in relative s-state only, has been used in a completely self-consistent Hartree-Fock (HF) calculation for finite nuclei. The potential saturates nuclear matter at the correct energy and density; the second-order corrections to the energy at saturation are small. The HF calculations have been done neglecting the Coulomb force and for nuclei with Z = N. Single-particle energies, wave functions, total binding energies, and root-mean-square radii have been calculated for the closed-shell nuclei ^{16}O, ^{40}Ca, and ^{80}Zr and for the nonclosed shell nuclei ^{12}C, ^{28}Si, and ^{60}Zn. The density distributions have been calculated in configuration space and also in momentum space. The single-particle HF potential is nonlocal and thus, in configuration space, depends upon three variables. Various approaches to the study of this potential have been made. For example, we have attempted to find various local and velocity-dependent potentials which are "equivalent" to the nonlocal HF potential.

Low-Temperature, Nuclear and Solid-State Physics. — Recently, a study was made of the correlation between the electrical resistance, which gold introduces as an impurity in an alloy, and the energy of the resonance gamma ray of the gold. On the basis of this correlation, it was concluded that at least in the vicinity of the gold nucleus, the gold atom became negatively charged relative to metallic gold in all the alloys investigated. It was concluded that the gold nucleus in its first excited state is larger than in the ground state. Because of the complexity of the theory of dilute alloys used in the above correlation, it seemed worthwhile to further investigate some of the basic concepts used

in this theory. This was done by making a study of the dependence of the resonance gamma-ray energy on the density of the metallic gold. Using the Mössbauer method the resonance gamma-ray energy was measured as a function of pressure up to about 1.2×10^6 psi at 4.2°K. It was found that the gold gamma-ray energy increased approximately linearly with pressure. The energy change from zero to maximum pressure was $(70 \pm 10) \times 10^{-9}$ ev. These results confirm the conclusions drawn from the earlier study of the correlation between the electrical resistance and the gamma-ray energy.

High-Voltage Experimental Program: Fast-Neutron Scattering Using Time-of-Flight Techniques. — Time-of-flight spectra for the scattering of 5-Mev neutrons from ^{56}Fe, ^{23}Na, ^{39}K, ^{40}Ca, and ^{89}Y have been obtained at 30, 43, 55, 68, 80, 93, 105, 117, and 130° (previous work given in ORNL-TM-1271). Spectra for iron were also taken at eight evenly spaced angles between 43 and 132° at neutron energies of 4.5 and 5.5 Mev. All samples were right circular cylinders of naturally occurring elements, and each was approximately 1/3 mean free path thick to 5-Mev neutrons. Standard samples were carbon and polyethylene [for (n,p) scattering]. Samples were placed at 0°, 7 to 12 cm from the center of a double-foil deuterium gas target, which was bombarded with an approximately 1-nsec-wide burst of deuterons from the 3-Mv Van de Graaff accelerator to produce a pulsed beam of monoenergetic neutrons.

Scattered neutrons were detected by a well-shielded scintillation detector (which utilized neutron-gamma discrimination) located 4 m from the sample. Time-of-flight spectra of the scattered neutrons were taken with a 4096-channel, two-dimensional, pulse-height analyzer in which input pulses were sorted according to their pulse height and their time-of-flight amplitude. Output from this analyzer was fed via a data link to a CDC 160A computer, where the spectra were corrected for time-walk perturbations and then listed as a printer plot. A time-of-flight monitor was used to monitor the target neutron yield. It was located 3.4 m from the target at an angle of −40°.

Absolute differential cross sections for elastic scattering and for inelastic excitation of individual levels in the struck nucleus are being extracted from the time spectra. Existing computer programs are being adapted to ease the repetitive and routine part of the considerable hand work that must be done in reducing each spectrum to absolute cross sections. Final corrected cross sections will be used to satisfy cross-section requests (WASH-1047) made by other groups and agencies and also to support a program of theoretical analysis.

With the thought of extending the present neutron scattering program to higher energies, a study of the $^{51}V(p,n)^{51}Cr$ reaction was carried out on the 5-Mv Van de Graaff accelerator at proton energies of 3.2, 3.7, 5.0, and 5.3 Mev. The accelerator was operated in its pulsed mode, and the electronic and mechanical apparatus used was the same as that for the (n,n') work discussed above. A large number of time spectra were taken at 0 and 25° for flight paths ranging between 1 and 9 m. The primary interest in these spectra is to understand the reported level structure of ^{51}Cr and to hunt for new levels in ^{51}Cr.

High-Voltage Experimental Program: Protons on ^{14}N. — The new precision gas scattering chamber has been used to measure the spectra of the reaction product produced by the proton bombardment of nitrogen from about 4 to 5.6 Mev bombarding proton energy. The proton beam was obtained from the 5.5-Mv Van de Graaff. The chamber was operated in the differentially pumped mode. We observed not only the elastically scattered particles and those inelastically scattered from the first excited and the second excited states, but also, at the higher energies, alpha particles from the (p,α) reaction. Yield curves of good resolution were obtained at six scattering angles, and detailed angular distributions were measured at eleven selected energies. These data are now being arranged on magnetic tape in a form suitable for processing on the large computer.

Spectroscopy Research. — Data have been obtained on the wavelengths of 96 lines of ^{208}Po, on the wavelengths of 8 lines of Pb I, and on the hyperfine structure of 31 lines of ^{209}Po. From these data, relative values of 26 energy levels of ^{208}Po I have been deduced. The hyperfine structure splittings of 14 of these levels in ^{209}Po have been determined. Some tentative identifications of the levels have been made, based on a determination of the absolute value of the fundamental level $6p^4\ {}^3P_2'$ by application of a Ritz formula to three levels taken as $6p^3({}^4S)np^5P_3'$, n = 7, 8, 9. The ionization potential of ^{208}Po has been tentatively estimated as 8.414 v. From the measured splittings of the energy levels of the configuration $6p^4$ of ^{209}Po I, the nuclear magnetic moment of ^{209}Po is estimated to be 0.7_6 nuclear magneton. Mrozowski's identifications of some lines of Po I were examined. It was found that his identifications of λ 4611 and λ 5940 are confirmed by coincidence of frequencies and by hyperfine structure patterns, but that his identifications of the other lines and his determination of $6p^4\ {}^1S_0'$ could not be confirmed by the data of this investigation.

CHEMISTRY

Effects of Radiation on Analytical Methods. — The deoxygenated ferrous sulfate solutions used for high-temperature dosimetry studies gave G values of 7.6, which were below those previously observed. Values of this order were obtained even at room temperature, where the value should be 8.2. It was suspected that the argon used to displace oxygen was introducing some reductant. Tests were carried out using stringent purification procedures on the argon, as well as substituting helium for the argon, with no significant improvement in the G values. An alternate deaerating procedure was attempted using vacuum degassing accompanied by alternate freezing and thawing. Argon gas was introduced at the final stage to ballast the tubes against excessive inward pressure during sealing. This procedure gave ambient-temperature G values of 8.2. The G values at 150 and 250°C averaged 8.1 and 8.0. The close agreement of the ambient-temperature value is reassuring and indicates that these values

are more reliable than those reported previously. The past difficulties are believed to have resulted from the decomposition of dosimeter solution adhering to the glass tubing at the point of the glass seal. Careful manipulation during solution entry and degassing to prevent any solution from wetting the sides of the tube near the point of seal were found to be of the utmost importance.

Separations Chemistry Research. — In the Biochemical Separations Program, whose purpose is to devise new methods for the separation and recovery of biological macromolecules, continued progress has been made toward the goal of preparing pure samples of the transfer ribonucleic acids (t-RNA's) from *E. coli*. The sample of phenylalanine-accepting t-RNA ($t\text{-}RNA^{Phe}$) obtained as previously described (ORNL-3896) by reversed-phase chromatography and gel filtration was very pure, as determined both by phenylalanyl-t-RNA formation and by terminal adenosine nucleoside content. One of the several leucine t-RNA's, $t\text{-}RNA_4^{Leu}$, has been purified by similar experimental techniques, and measurements of the purity of that sample are being made. The molecular weight of the $t\text{-}RNA_4^{Leu}$ was estimated by chromatography on calibrated columns of Bio-Gel P-150 polyacrylamide gel. A preliminary value of 29,000 was obtained. This is higher than the value of 26,000 obtained for $t\text{-}RNA^{Phe}$ by the same technique.

Foam Separation Studies. — The effect of foam reflux on volume reduction and surfactant distribution in the water-dodecylbenzenesulfonate (DBS) system is being studied as part of a basic study of the mechanisms of foam separation. The apparatus, in which any desired fraction of the condensed foam can be returned to the countercurrent (liquid vs air) foam separation column, gave excellent mechanical performance under conditions of 20, 50, and 80% foamate reflux when distilled water containing DBS in low concentration was used as feed. The DBS was added continuously to the column in two places. About 80% entered the top of the column with the feed (DBS concentration about 82 ppm, feed throughput rate about 46 gal ft^{-2} hr^{-1}); the other 20% was added slowly (about 0.15 gal ft^{-2} hr^{-1}) to the bottom of the column as a more concentrated (5000 ppm) solution. The countercurrent air-to-liquid volume ratio was maintained at 13 to 15. Steady-state operation was reached in less than 2 hr in all cases.

The DBS concentration in the effluent varied directly with the amount of foam that was refluxed; however, the concentration increased only from 76.9 to 85.0 ppm over the wide foam-reflux range studied. On the other hand, the overall volume reduction factor, corrected for the fraction of collapsed foam returned to the column, increased markedly, from 7.06 to 109.5. Corresponding average DBS concentrations in the foam phase increased from about 1000 ppm to almost 2700.

The surfactant concentrations in the column effluents and foamates were determined by analysis of the ultraviolet spectra, since DBS has characteristic absorption features at 2090 and 2550 A.

Volatility Studies. — A study of the sorption of PuF_6 on various metal fluorides is being made in conjunction with the development of

volatility processing. The PuF_6 sorption values for the fluorides of the elements in groups I-A and II-A of the periodic table, in the group of 31 sorbent fluorides previously tested, have been improved by changes in the experimental technique and by minimizing the amount of sorbent used in each test. The sorbents (0.1 to 1.0 mg) were deposited in test dishes by evaporation of various types of solutions in order to make the material more susceptible to gas-solid reaction. The walls of the dish were increased in height to avoid loss from spattering. The sample size was adjusted to give good analytical precision without introducing significant problems of self-absorption. The new values for the Pu/metal atom ratios are as follows: LiF, 0.90; NaF, 1.13; KF, 1.31; RbF, 1.35; CsF, 1.06; BeF_2, 0.04; MgF_2, 0.16; CaF_2, 0.49; SrF_2, 0.57; and BaF_2, 0.37. The temperature of sorption was 350°C for the first exposure to PuF_6, with a final temperature of 125°C.

The proximity of the above values to simple integral ratios suggests that compounds may be formed. This conclusion is supported by failure of the PuF_6 to react beyond the ratios indicated when the PuF_6 concentration was significantly increased. The solid phases formed were white or light yellow except for the following: NaF, bright red; RbF, red; and CsF, brown-red.

Chemistry of Carbides and Nitrides. — The reactions of the uranium carbides with 3 to 9 M HCl at 80°C are being studied as part of a basic research program on the chemistry of metal carbides. Uranium monocarbide gave the same gaseous products with the hydrochloric acid solutions as with water, and the sesquicarbide showed only minor differences. In contrast, uranium dicarbide yielded significantly more free hydrogen and wax and less gaseous hydrocarbons when hydrolyzed in 3 to 9 M HCl instead of in water. Uranous chloride solutions were obtained in all cases. Relative reaction rates for the mono- and dicarbides were: H_2O = 3 M HCl = 6 M HCl > 9 M HCl; and for the sesquicarbides, 3 M HCl = 6 M HCl > H_2O = 9 M HCl.

Chemical Engineering Research: Stacked-Clone Contactor. — The stacked-clone solvent extraction contactor, by virtue of its rapid control response, low holdup, and high throughput, is potentially useful for processing small inventories of solutions which may be highly radioactive. However, feed solutions containing small particles may be encountered.

We have made tests to identify and evaluate potential operating problems when using such feed solutions. We learned that the degree of control upset and product loss depends on the type of impurity. Sudden injections of 30-g powder packs of diatomaceous earth, fuller's earth, and infusorial earth to the aqueous feed stream (0.08 M HNO_3) when operating at steady state at 40°C and about 60 to 80% of flooding with Amsco as the solvent produced the following results: In all cases, a momentary control upset occurred when the slug of solids was sensed by the interface control system, but recovery was complete in a few seconds without manual adjustment. Diatomaceous earth remained in the aqueous phase, and more than 90% of the solids passed out the raffinate end of the contactor within a minute. Fuller's earth and infusorial earth produced more serious effects in that emulsions were formed, contaminating the organic product stream for 7 min before normal operation was resumed.

Chemical Engineering Research: Macromolecular Biological Separations. — The initial step of the program of separating kilogram quantities of purified mixed transfer ribonucleic acids (t-RNA's) from E. coli has been completed. About 300 kg of E. coli cells was processed to yield 632 g of mixed t-RNA's with acceptable activity. The next step, the separation of phenylalanine-accepting t-RNA from the bulk of the mixed t-RNA's, is now in progress. The separation process consists in a gradient elution from a diatomaceous earth column saturated with a quaternary ammonium compound. This process has been highly successful on a small scale but must be scaled up manyfold to handle the large quantity of feed material now available.

A procedure has been developed for preparing large batches of column packing of acceptable and consistent quality. A 1/2- and a 1-in. chromatographic column and the necessary auxiliary equipment have been installed and several runs made. In the first runs, the packing apparently became contaminated by iron that resulted from corrosion of equipment. These traces of iron destroyed the phenylalanine t-RNA and obscured the results of the runs. Once the trouble was identified, the columns were cleaned and the sources of the iron contamination were removed. In subsequent runs, acceptable separation of phenylalanine t-RNA from other t-RNA's was obtained.

Mechanisms of Separations Processes. — The electrolysis of water to produce hydrogen is being studied as a means of utilizing large blocks of cheap electrical power which might be available from dual-purpose desalination reactors. The cell being utilized has porous nickel electrodes. The electrolysis products, hydrogen and oxygen, are evolved from the reverse sides of the electrodes. This arrangement permits very close spacing of the electrodes, for our work 0.010 in., and thus minimizes the voltage drop due to electrolyte resistance. Asbestos paper is used as a matrix to hold the aqueous KOH electrolyte in contact with the two working electrodes. The electrolyte concentration in the asbestos matrix is maintained constant by diffusion of the circulating electrolyte (in contact with the reverse sides of both electrodes) through the porous electrodes. The electrolyte reservoir is maintained at constant temperature, and carbonate contamination of the electrolyte is held below 0.1% by the use of Ascarite tubes at all system openings. The cell has been operated successfully at temperatures up to 80°C and with current densities up to 2400 ma/cm^2 (2230 amp/ft^2).

Cell voltages have been measured at several different temperatures and two different concentrations of KOH (27 and 35%). The gain in cell efficiency (20 to 40 mv decrease in cell voltage) observed in going to the higher concentration was negligible considering the fact that it involves a 36% increase in electrolyte inventory. Cell voltages of 2.01 and 2.34 v were measured for current densities of, respectively, 1000 and 2400 ma/cm^2; the cell temperature was 80°C, and the electrolyte was 27% KOH.

Current efficiencies were determined at current densities of 1000, 1500, and 2000 ma/cm^2 by measuring the volume of hydrogen evolved. The efficiencies ranged from 97.2 to 98.5%, with the lowest value obtained for the highest current density.

Published data are available for another type of electrolysis cell, in which plate electrodes with a catalytic coating of Raney nickel are used. The gaseous products are evolved between the working electrodes, with gas separation achieved by a membrane. The published voltage for this cell at atmospheric pressure and 75°C is 2.06 v at a current density of 400 ma/cm^2 and with a 0.25-in. electrode spacing. (Increased electrode spacing is necessary because of electrolyte voids caused by gas bubbles.) Using our cell with the porous electrodes, the voltage, measured under comparable conditions, was 1.84 v. Thus our system showed a higher cell efficiency by about 11%. This increase should be even greater at higher current densities (at atmospheric pressure) because release of the product gases takes place behind the electrodes and thus electrolyte resistivity is not affected.

Electrochemical Kinetics and Its Application to Corrosion. — The rates of the surface reaction between HF molecules and zirconium metal were determined in a variety of solvents by use of the rotating disk electrode. Measurements of the $HF-H_2SO_4$ system were completed, and the experiments were extended to the HF-HCl system. The results were compared with the previously completed $HF-HNO_3$ system, where it was found that rates depend primarily on the concentration of HF and the rotational velocity, with only a slight dependence on the concentration of HNO_3. In $HF-H_2SO_4$, the sulfate, if present in large enough concentrations, reduces the rate and changes the reaction order with respect to HF from one to two. These results indicate that sulfate is adsorbed and prevents the HF from attacking the zirconium at the site of adsorption. In the HF-HCl system, results were almost identical with those found in $HF-HNO_3$, except that pitting and pitting potentials were observed. The pitting potentials were identical with those found when the electrode had relatively thick passive films in solutions free of HF. The values of the pitting potentials were independent of rotational velocity. Experiments with electrodes that were irradiated with fast neutrons showed that there was no difference in rates from electrodes that were not irradiated.

Physical Chemistry of Molten Salts. — Crystal-structure analyses have clarified the relationships among double salts of alkali fluorides (XF) and tetravalent heavy-metal fluorides (MF_4) having the stoichiometric ratio of $7XF:6MF_4$. Compounds of this type have been observed in a dozen or more molten-salt mixtures. In the cases of $7LiF \cdot 6UF_4$ and $7LiF \cdot 6ThF_4$, which form tetragonal crystals, a complete structure determination showed that the ratio is actually 1:1; that is, the formulas are $LiUF_5$ and $LiThF_5$. The U^{4+} (or Th^{4+}) ion is coordinated by nine F^- ions arranged in the shape of a trigonal prism with pyramids on the prism faces. These polyhedra share corners and edges to form an intricate network in which the Li^+ ions are able to occupy irregular octahedral holes. The other 7:6 compounds are of rhombohedral symmetry; a structure determination of $Na_7Zr_6F_{31}$ has confirmed the stoichiometry and offers a good explanation for its existence. Six Zr^{4+} ions are bridged together by F^- ions to form a "cage" which contains one F^- ion inside it. This F^- ion and an Na^+ ion elsewhere in the structure cause the observed deviations from the 1:1 ratio.

Isotope Chemistry. — Operation of the Oxygen-17 Facility was interrupted November 28 when an electrical transformer which supplied power to the water distillation cascade ignited. The electrical outage lasted 25 min; normal operation was restored through the use of a standby transformer. The interruption caused some mixing in the distillation columns, and the peak ^{17}O concentration decreased from 1.8% to 1.6%. The peak ^{18}O concentration remained at 90%. Operation of the thermal diffusion cascade was normal during this report period. The peak ^{17}O and ^{18}O concentrations in the thermal diffusion cascade reached 24 and 99.8% respectively. With the exception noted above, equipment performance and productivity improved during the first half of FY 66. More than 270 g of isotopically enriched O_2, H_2O, and D_2O, worth $85,000, was produced, and all commitments to customers were met.

TRANSURANIUM-ELEMENT PROCESSING

TRU Operations. — Testing and checkout of much of the Transuranium Processing Plant building service equipment and out-of-cell chemical process equipment are now under way. The capacity of the compressed air system is marginal, and steps are being taken to increase it. The propane-gas piping system had many leaks, and all threaded steel pipe behind the laboratory benches has been replaced with silver-soldered copper tubing. Balancing and testing of the ventilation systems have been started. It was necessary to reseal many points in the exterior wall to minimize air inleakage, since the building is operated at 0.3 in. H_2O gage vacuum at all times.

The ten control systems for metering solutions into the cell, which use the pressure drop across fixed capillary restrictors as a means of indicating flow, were checked out and now function satisfactorily, after a number of minor changes. Several of the restrictors had to be resized.

Transuranium Processing Plant Design and Construction. — All equipment racks and piping in the main-line chemical processing system, with the exception of the centrifuge and its associated piping, have been installed in the TRU cell bank, and a leak check of this system is in progress. Final testing of the all-Zircaloy-2 centrifuge was completed satisfactorily following seal-welding of the outer overflow tank. The block walls of the office building addition have been erected, and installation of the stressed-beam-supported roof is in progress.

Curium Recovery Facility. — While awaiting the fabrication and installation of several major equipment replacements necessary for a ^{242}Cm production run scheduled to start in February, the Curium Recovery Facility is being used to test alternative flowsheets. Two were tested during this report period: the Talspeak and Cerex processes. In the Talspeak process, the lanthanide-actinide separation is achieved in a complexed lactic acid system, whereas in Cerex, only cerium in the rare-earth group is removed, this by oxidation to Ce^{4+}. The preliminary results obtained from three demonstration runs, using highly active ^{244}Cm solutions as feed, are not definitive enough to warrant the replacement of the flowsheets used in previous ^{242}Cm recovery programs.

Process Development. — Chemical processing to recover transplutonium elements from the first HFIR prototype target rod, which was irradiated in the Savannah River High Flux Reactor, was continued. The plutonium fraction recovered by HCl anion exchange was additionally purified by HNO_3 anion exchange. The transplutonium elements were separated by chromatographic elution from cation exchange resin with ammonium α-hydroxyisobutyrate to isolate californium, einsteinium, and berkelium.

Analytical results to date indicate that the rod contained 5.4 g of Pu, 1.6 g of Am, 2.6 g of Cm, 5 μg of ^{252}Cf, and 0.2 μg of ^{253}Es at reactor discharge. Analysis of the berkelium fraction has not been completed.

METALLURGY AND MATERIALS

Physical Properties Studies. — We are continuing detailed measurements of the physical properties of high-purity single-crystal and polycrystalline silicon from 400 to 1300°K, with the purpose of examining high-temperature heat transport mechanisms in a material that in many ways resembles refractory oxides. Our measurements of the electrical resistivity (ρ) show a linear dependence of log ρ on 1/T from 600 to 1200°K, which is the expected intrinsic behavior; deviations from linearity, typical of extrinsic behavior, occur below 600°K. Extensive thermal conductivity measurements from 325 to 1325°K have revealed two ranges in which the thermal resistivity is linear with temperature, 325 to 675 and 675 to 1100°K, with a 1.5-fold slope increase occurring at 675°K, which is near the Debye temperature of silicon. Between 1100 and 1325°K the thermal resistivity exhibits a nonlinear temperature dependence, which is indicative of a significant electronic component that increases the thermal conductivity above that expected from lattice conduction alone. These results confirm previous high-temperature observations by workers at the General Electric Research Laboratories, but the results contradict the high-temperature measurements made by Ames Laboratory workers. Our high-temperature thermal conductivity results are being analyzed in terms of the measured electrical behavior, which should allow prediction of this effect in other intrinsic materials, such as UO_2.

Direct Observation of Lattice Defects. — In our program aimed at improved understanding of factors affecting the strength of high-temperature metals, we have developed an equation describing the frequency of cross slip in body-centered cubic metals and alloys based on the Peierls mechanism of dislocation motion. The equation can be expressed in terms of temperature, stress, strain rate, crystallographic orientation, and composition. We feel that the structure of a slip band (i.e., its stress-concentrating ability) is determined to a large extent by the frequency with which dislocations move off the most highly stressed {110} plane. Our equation shows that this is an extremely strong function of temperature, which could explain the abrupt change from ductile to brittle behavior observed in these materials. We currently are using an etch-pitting technique to observe slip-band structure directly as a function of the deformation variables.

Physical Ceramic Studies. — We are continuing our efforts to establish the mechanism of void formation at grain boundaries during deformation of fluorite-structure materials, to enable us to understand and improve the strength of ThO_2 and UO_2. We observed the formation of porosity on the grain boundary of a bicrystal of UO_2 that experienced grain-boundary sliding at a stress of 3000 psi at 1500°C. Also, the extent of void formation appeared to depend on the boundary orientation. The results of recent creep tests on polycrystalline UO_2 indicate that little dislocation motion should occur in this stress range during creep in UO_2 and that the transport of material should be by a diffusional flow.

Solid Reaction Studies. — Continuing our study of tracer diffusion in the body-centered cubic form of the transition element titanium, we have measured the tracer diffusion of ^{110}Ag in the beta phase from 900 to 1600°C. The Arrhenius plot, like that for ^{46}Sc, is linear over the whole temperature range. The diffusion coefficient varied from 2.5×10^{-10} cm^2/sec at 900°C to 2.7×10^{-7} cm^2/sec at 1600°C; it is represented by the Arrhenius equation $D = 3 \times 10^{-2} \exp(-43{,}000/RT)$ cm^2/sec.

CONTROLLED THERMONUCLEAR RESEARCH

The DCX-1 Injection-Accumulation Experiment. — A paper summarizing the mode identification studies previously described was submitted to Physical Review Letters for publication. Since comparisons between DCX-1 and DCX-2 phenomena might be valuable in determining the mode and role of microinstabilities in DCX-2, we sought conditions with gas dissociation for which DCX-1 would give experimental results more like those observed in DCX-2. The most sensitive parameter in these studies was the radial magnetic field gradient. With small gradients (field index approximately 0.002), DCX-2-like rf emission amplitude and frequency structure and large energy spreads (250 kev full width at half maximum) were obtained in DCX-1. In addition, a sharp transition (at a pressure of 1×10^{-5} torr) in the rf structure and cold plasma properties was found; this transition was later observed in DCX-2.

From plasma stabilization in the calutron by "end wall" treatment, reactive end walls were developed and applied to a DCX-1 plasma of 10^7 cm^{-3} density and 1 sec lifetime (gas dissociation). One pair of end walls was indeed able to completely suppress all rf emission when optimized in axial and azimuthal position. This encouraging result will lead us to return to the region of operation where instability losses dominate in order to see if these walls still maintain their stabilizing effects. In the meantime a version of the Beam-Plasma Interaction "Burnout" series is being operated in the DCX-1 Facility by that group.

Properties of Plasmas Created by Electron-Cyclotron Heating. — The EPA Facility has been renamed INTEREM, which stands for Injection of Neutrals and Trapping on an Electron Resonant Environmental Medium. The facility is still undergoing extensive modification to provide diagnostic access to the trapped 20-kev protons. A long evacuated tube is being

added to the south side to provide the necessary flux reduction of the charge-exchange neutrals. With this reduction, it will be possible to perform a time-sequential energy analysis of the neutrals entering a cooled silicon barrier detector located about 655 cm from the machine axis. This measurement will determine the nature of that component of neutral current which has an e-folding time an order of magnitude longer than the dominant component after beam turnoff. In addition, the charge-exchange current will be measured as a function of the axial position to look for evidence of spreading of the trapped 20-kev protons.

Improvements have been made on the neutral-beam ion source. The neutral beam current should be up by more than a factor of 2 over the previous nominal value of 60 ma equivalent.

The new ELMO cavity, which had a stainless steel central section, was found to be too lossy. A thin copper plating is being added to the interior. This should increase the surface conductivity for microwave efficiency without seriously sacrificing its diamagnetic response time.

Investigations are continuing on the instabilities that have been observed in the electron-cyclotron-heated plasmas in the PTF. The purpose of the present investigations is to determine the conditions at the threshold for the growth of gradient B drift waves (flutes). The frequencies and phase velocities of these waves near threshold are also being measured. The phase velocities that have been observed are near the calculated hot-electron gradient B drift velocity. The fundamental frequency for these waves in the PTF device is about 0.7 Mc. The frequencies of waves that are observed to grow at the surface of the plasma are from 7 to 10 Mc. However, these waves appear to become amplitude modulated at the fundamental of 0.7 Mc, and as the flute grows and begins to transport plasma to the walls, the high-frequency waves "wash out" and only the fundamental is observed near the wall. For some conditions near threshold, the 7- to 10-Mc waves are observed to grow and decay in a period of 2 to 3 μsec with little or no transport of plasma to the wall.

Properties of Plasmas Created by Beam-Plasma Interaction. — In previous experiments we had inferred that some ions in the "Burnout" plasma machines were being heated to quite high energies (several kilo-electron volts). These experiments included the observation of high-energy ions escaping through openings in the circumference, neutrons emitted from D-D reactions, and Langmuir probe measurements. We have demonstrated by Doppler broadening and tilting of the Balmer β line, emitted by recombining ions, that the ions in Burnout IV have a mean energy corresponding to about 1 kev.

Two electrode modifications allow operation of the hot-electron plasma blanket experiment at much higher power input and stronger magnetic field. Operation of the anticathode above 16 to 20 kw power input has been achieved by locating the anticathode in an iron enclosure. The resulting beam spread has lowered the power dissipated per square centimeter by a factor of 5.

It has been found that operating conditions are significantly influenced by variations of the magnetic field in the anode region. A 4-in.-ID, 3/8-in.-thick iron cylinder placed around the anode increased the optimum operating axial midplane field from about 1400 gauss to about

1900 gauss. In addition, the power input has been increased to about 37 kw, the maximum output of the supply. These results may indicate the presence of an instability in the anode region of the type studied by Hoh [Phys. Fluids 6, 1184 (1963)] or Simon [Phys. Fluids 6, 382 (1963)], which extinguishes or shorts out the discharge at sufficiently high magnetic fields.

Ion Source and Ion Beam Technology. — An intense effort has been made to incorporate the new H_2^+ ion source into the 600-kv accelerator. The plasma boundary from the standard version of this source resulted in a very divergent beam, with several milliamperes impinging on the accelerator electrodes. After much experimentation a source has been built with a nonferromagnetic (copper) target cathode, which with only brief evaluation delivers a 100-ma, 550-kv H_2^+ beam through the DCX-2 injection geometry. This is equal to the best previous performance using the standard duoplasmatron ion source. Since this new source has been only briefly evaluated and one is not limited by a low percentage H_2^+ production, one can possibly increase the usable H_2^+ beam with more test-stand evaluation.

All of the components of the DCX-2 end injector have been tested, and beam alignment and injection efforts are now in progress.

Theoretical Plasma Physics. — Criteria have been derived for the relative cold-plasma density required to stabilize the drift waves (associated with the negative radial gradient of the magnetic field in a simple mirror trap) in energetic ion or electron plasmas separated from a grounded conducting end plate by a cold plasma. We consider three cases:

1. In a dense ($\omega_{pi}^2 \gg \omega_{ci}^2$) hot-electron plasma in which $\omega_{ge} \ll \omega_{ci}$, where ω_{ge} and ω_{ci} are the electron precession frequency and ion gyrofrequency, respectively, the required relative cold-plasma density is

$$\left(\frac{N_{cold}}{N_{hot}}\right)_{critical} \approx k_\perp^2 \frac{\omega_{ge}}{\omega_{ci}} \frac{L_{hot}}{2} L_{cold} \frac{m}{M} ,$$

where L_{hot} and L_{cold} are the lengths of the hot and cold plasmas, respectively, m/M is the electron-to-ion mass ratio, and $k_\perp$ is the azimuthal propagation vector of the wave considered. This criterion is similar to one derived by Babykin et al. [Soviet Phys. JETP 20, 1096 (1965)] and seems to be supported by experiments reported by that group.

2. A dense hot-electron plasma in which $\ell\omega_{ge} \approx \omega_{ci}$ for some small integer ℓ leads to a corresponding criterion which is roughly given by

$$\left(\frac{N_{cold}}{N_{hot}}\right)_{critical} \approx k_\perp^2 \frac{1}{\delta} \frac{L_{hot}}{2} L_{cold} \frac{m}{M} ,$$

where $\delta = 1 - \ell\omega_{ge}/\omega_{ci}$ and $0 < \delta < 1$ is required. Tentative data from PTF appear to be in rough agreement with this condition.

3. Low-density ($\omega_{pi}^2 \ll \omega_{ci}^2$) hot-ion plasmas are stabilized if a similar criterion is satisfied. Again, to within an order of magnitude,

$$\left(\frac{N_{cold}}{N_{hot}}\right)_{critical} \approx 10k_\perp^2 \left(\frac{\omega_{gi}}{\omega_{ci}}\right) \frac{L_{hot}}{2} L_{cold} \frac{m}{M} .$$

We have considered the cold electrons to be the dominant effect in deriving these criteria. If the cold ions dominate, the mass ratio, m/M, must be replaced by unity.

The striking inference of this model is that very little cold (electron) plasma is required to stabilize the long-wavelength precessional drift waves. Additional experimental data with which to test the validity of the model are being sought.

Atomic Cross Sections. — Comparison of H_3 counting rates of a silicon surface-barrier detector having a large angular acceptance with that of a proportional counter having a low angular acceptance has confirmed the existence of stable excited "Rydberg" states of H_3, similar to those found in H and H_2. The capture into repulsive levels of H_3 dominates the electron capture process when H_3^+ is passed through hydrogen gas.

Studies of silicon surface-barrier detectors for the detection of low-energy heavy particles have continued. By using detectors with 1000-μ depletion layers the resolution (full width at half maximum) is 2.8 kev for 50 mm^2 of active surface. Energy loss in the normally 50-$\mu g/cm^2$ gold barrier for 10-kev protons was determined to be approximately 200 ev.

Measurements with a refrigerated McLeod gage indicate that the "mercury vapor pumping effect" can be eliminated by immersing the gage in a region where the ambient temperature is −15°C. Calibration of a commercial capacitive differential manometer, using a refrigerated McLeod gage, has been made to determine the usefulness of such a gage for a pressure standard in measuring condensable vapors usually found as residuals in vacuum systems. Present results indicate nonlinear behavior for pressures less than 10^{-3} torr.

BIOLOGY AND MEDICINE

SOMATIC EFFECTS OF RADIATION

Medium(Low)-Level Long-Term Effects of Radiation. — Female RF/Un mice were exposed to 50, 100, 200, or 400 rads of 14-Mev whole-body neutron radiation at ten weeks of age. Selected groups of mice from sham-irradiated controls and from each radiation level were examined for

hematologic changes at 100-day intervals throughout life. Total leukocyte counts in the irradiated groups did not vary significantly from those in controls, decreasing slightly with time in all groups. The lymphocyte counts decreased correspondingly with time, whereas the neutrophil counts increased slowly with time in all groups of mice up to 600–700 days of age. These changes were not significantly correlated with radiation dose. The packed red-cell volume in all groups decreased slowly with time. After 200 days, packed red-cell volumes in the irradiated groups were often significantly lower than those in controls. The blood findings in individual aging mice were dependent on the health of the animals in question, being significantly affected in the presence of diseases such as leukemia. Individual variations of this type, however, are not thought to have accounted for the age-dependent trends observed. The absence of long-term dose-dependent changes in peripheral blood contrasts with the presence of early hematologic changes and the induction of leukemia in the irradiated populations.

Physiology of Blood Platelets. – It has been reported that vinblastine (VLB), an alkaloid which inhibits mitosis and thus has a radiomimetic action in mammals, depresses proliferation of myelocytic and erythrocytic cells of the bone marrow but not megakaryocytes. Since a differential effect on various elements of hemopoietic tissue would be valuable in studying hemopoiesis and especially megakaryocytopoiesis, we have carried out additional experiments to extend the knowledge of the action of this drug. Specifically, we have investigated some effects of daily, sublethal doses of VLB on megakaryocytes and platelets during a five-day period. Erythrocyte, leukocyte, and platelet counts were taken daily. Two VLB-injected and two control rats were killed at 2, 3, 4, and 5 days after beginning injections, at which time bone marrow was taken for smears and sections. Platelet counts dropped sharply, reaching less than 10% of a normal count by five days. The total white blood cell count gradually declined. There were no changes in erythrocyte counts or hematocrits. The average number of megakaryocytes in sections of bone marrow of control rats was 14.1 per high power field, whereas that of VLB-treated rats had dropped to 3.3 by the fifth day. In addition, the proportion of naked megakaryocyte nuclei was greatly increased, and markedly fewer maturing megakaryocytes were present in treated rats than in controls. Other abnormalities in the morphology of megakaryocytes were also observed. It is therefore clear that VLB has a marked effect on megakaryocytic cells of the marrow as well as on erythrocytic and myelocytic elements. In addition, this drug may directly destroy circulating platelets or increase their rate of usage.

Virology and Leukemogenesis. – Cytopathogenic effects (CPE) and transformation, in an established culture of BALB/c mouse cells, have recently been associated with infection by the Rauscher leukemia virus. The CPE and transformation are not grossly apparent when the infected cells are cultured on modified NCTC 109 medium with 25% human serum and 5% calf serum. When, however, infected cells are subcultured in modified Eagle's basal medium (BME) with 10% calf serum, CPE and transformation are manifested as follows: Immediately after subculturing in BME, the infected cultures become pretransformed, exhibiting enlarged cells with

ill-defined borders, cytoplasmic vacuolization, increased cytoplasmic eosinophilia, and nuclear distortion. Two to three weeks later, such cultures are gradually overgrown by transformed fibroblastic cells growing in a criss-cross pattern. A posttransformation stage is characterized by an abrupt rounding of the transformed, criss-cross cells. Subcutaneous injection of the infected, transformed cultures into newborn BALB/c mice resulted in 100% incidence of myxofibrosarcoma at the site of inoculation within 25 days. Injection of untransformed, infected cells, grown in NCTC 109, resulted in a 35% incidence of myxofibrosarcomas within 30 days. Injection of BALB/c mice with untransformed, uninfected control cells resulted in a 10% incidence of spindle cell sarcomas within 40 days after inoculation. The recognition that interactions between leukemia viruses and host cells can thus be studied in vitro promises to expedite the investigation of viral aspects of radiation leukemogenesis in providing an assay system more amenable to analysis than that of leukemia induction in vivo.

RADIATION GENETICS

Human Cytogenetics. — Pre- and postflight peripheral leukocyte samples have been obtained from the GT-6 and GT-7 crews. Analysis of the chromosome preparations is virtually complete. Biological testing of the temperature and irradiation profiles anticipated for the GT-11 S-4 experiment is in progress. The first S-4 thermoelectric refrigeration unit has been completed, and qualification testing is under way.

In order to further elucidate the mechanism by which cytosine arabinoside induces chromosomal aberrations, fractionation experiments with human leukocytes are being conducted, testing whether the drug will keep x-ray-induced chromosomal breaks open and whether x-ray-induced breaks can interact with cytosine arabinoside-induced breaks.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — Bacteriologic sampling in our experimental animal facility housing 120,000 animals showed a high incidence of a nonpigmented *Pseudomonas* (*P. stutzeri*). When *Pseudomonas* is present in animals receiving acute lethal whole-body radiation, early deaths frequently occur which preclude use of these animals in radiation protection and recovery experiments. To determine if *P. stutzeri* was involved in the early death syndrome in x-irradiated mice, a comparison of the pathogenicity of *P. aeruginosa* with that of *P. stutzeri* was made in mice on the basis of time of death and subsequent recovery of the inoculated organism(s) from animals dying after irradiation. Adult $C3BF_1$ mice 10 to 14 weeks of age were separated according to sex, then randomly divided into groups of five mice per cage. Fresh water bottles containing 10 to 14 ppm free chlorine were placed on the cages three times per week during the experiment. Before the animals were used in the experiment, fecal

samples from each pan of mice were tested for Pseudomonas sp., and the mice were tested for Salmonella sp. Neither organism was found. All irradiated animals received 950 r of 250-kv x rays. Twenty-five mice were each inoculated intraperitoneally with 1×10^6 P. aeruginosa organisms approximately 2 hr before irradiation. Twenty-five animals were inoculated with the same dose by oral lavage 24 hr before irradiation. Ten control mice were inoculated (5 intraperitoneally, 5 orally) with the same number of organisms but were not exposed to x rays. Ten additional mice were held in the same room with the inoculated mice but were not given Pseudomonas or exposed to ionizing radiation. Identical inoculation and control procedures were used with those mice given P. stutzeri, except that a dose of 1×10^7 organisms per mouse was used. Data show a correlation between incidence of early death, which began on day 2, and recovery of pigmented Pseudomonas administered intraperitoneally. In the irradiated group given the pigmented organisms orally, death began to occur on day 5; again, there was a good correlation between incidence of early death and recovery of the organism. Deaths that occurred in the irradiated group given the nonpigmented Pseudomonas were not associated with the presence of the organism. Thirty-two of fifty mice receiving P. aeruginosa died within eight days after irradiation; 29 of the 32 showed pigmented P. aeruginosa on heart blood culture. Fewer deaths were seen with the oral as compared with the intraperitoneal route of administration. Two out of fifty mice given P. stutzeri died within eight days of exposure to x rays. Neither showed the organism on heart blood culture. Under the conditions of this study, P. stutzeri was not implicated in the early-death syndrome.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation: A New Detector for Neutron Diffraction Systems. — Existing neutron diffraction systems have a single detector, which is moved from point to point in space to determine the space-intensity pattern of the neutrons diffracted from a crystal whose structure is being studied. This procedure is usually time consuming, because it does not make efficient use of the limited number of available neutrons. A new detector is being developed which we believe will significantly reduce the data collection time.

The new thermal-neutron scintillation detector will provide information on the intensity and location of the neutron-scintillator reactions. The detector area will depend either on the availability of image intensifiers having large input photocathodes or on the speed and aperture of the optical systems which might be used to couple the scintillator screen to the image intensifier. Detector areas up to 75 in.^{2} seem to be feasible. The image intensifier output screen will be scanned by a television camera tube whose scanning pattern will be synchronized with the advancing of a computer memory, each of whose cells corresponds to a small area on the scintillator screen. When a neutron is detected, a count will be added to the cell appropriate to the location of the detected neutron in the scintillator. In this way, a neutron diffraction pattern will accumulate in the memory as the experiment proceeds.

We are seeking suppliers of suitable electro-optical components and neutron-sensitive phosphors.

Basic Instrumentation: Charge-Sensitive Preamplifier. – A charge-sensitive preamplifier was developed for field use. The input section is a new wide-band low-noise design reported earlier (ORNL-3825). The output section is a new cable driver which will linearly drive ±22 ma into a 93-ohm load with a 10 to 90% rise time of 40 nsec with no overshoot. The voltage gain of the driver is 5 when terminated by a 93-ohm load. The gain-bandwidth product is 1 gigahertz. The equivalent noise voltage is sufficiently low to allow the charge-sensitive input section to control the noise performance of the preamplifier. The temperature stability of the driver is less than +35 ppm/°C in the range from +15 to +50°C.

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