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Argonne National Laboratory
REACTOR DEVELOPMENT PROGRAM
PROGRESS REPORT
January 1962

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REACTOR DEVELOPMENT PROGRAM
PROGRESS REPORT

January 1962

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FOREWORD

The Reactor Development Program Progress Report, issued monthly, is intended to be a means of reporting those items of significant technical progress which have occurred in both the specific reactor projects and the general engineering research and development programs. The report is organized in a way which, it is hoped, gives the clearest, most logical over-all view of progress. The budget classification is followed only in broad outline, and no attempt is made to report separately on each sub-activity number. Further, since the intent is to report only items of significant progress, not all activities are reported each month. In order to issue this report as soon as possible after the end of the month editorial work must necessarily be limited. Also, since this is an informal progress report, the results and data presented should be understood to be preliminary and subject to change unless otherwise stated.

The issuance of these reports is not intended to constitute publication in any sense of the word. Final results either will be submitted for publication in regular professional journals or will be published in the form of ANL topical reports.

The last six reports issued
in this series are:

July 1961	ANL-6399
August 1961	ANL-6409
September 1961	ANL-6433
October 1961	ANL-6454
November 1961	ANL-6473
December 1961	ANL-6485

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I. WATER COOLED REACTORS

A. EBWR

1. 100 Mw Modifications

a. Reboilers - Installation of the reboilers was completed in December. The units and piping that were reinstalled were subjected to hydrostatic and leak tests. All tests on the system were passed successfully. The reboiler plant is now ready for operation.

b. Instrumentation and Control - During the past few months the intensity of electrical noise in the nuclear instrumentation has been increasing, particularly on the period circuit. The substitution of a battery power supply for the electronic chamber power supply has reduced the pickup appreciably. As time permits, the source of noise will be located and eliminated.

2. Experimental Equipment

a. Transfer Functions - Testing of the start-stop logic circuits which control the reactor computers has been initiated. A model has been built and has successfully operated the analog computer. The digital computer will be tested when it is received back from the manufacturer.

b. Neutron Flux Distribution in EBWR - The modification to the east instrument nozzle is nearly complete. In the new nozzle assembly the nozzle will extend out from the reactor at a 27-degree angle to horizontal to its termination above the main floor of the reactor building. The modified nozzle will be used for neutron flux measurements by the bead chain method, for thermocouples, and possibly for water and steam samples from the reactor vessel.

3. Calibration of the New Control Rods with Zircaloy Followers

The new boron-stainless steel control rods with Zircaloy followers were installed in the reactor and calibrated over the range of about 14 in. to 32 in. by an approximate method (period measurement). The reactivity worth of the nine rods was determined by comparison with results previously obtained by adding known amounts of H_3BO_3 to the reactor. The calculated reactivity changes are given by:

$$\delta\rho = \frac{\bar{\Sigma}_A B}{\bar{\Sigma}_A + \bar{\Sigma}_A B}$$

where $\bar{\Sigma}_A^B$ (the effective boron capture cross section) was calculated and $\bar{\Sigma}_A$ (the effective reactor absorption cross section) was determined by period measurements.

This coarse calibration showed that the new control rods are worth approximately the calculated value and that they have about the same reactivity worth as the fueled follower rods. More detailed and accurate measurements are planned later.

B. BORAX-V

1. Installation and Modification of Reactor and Components

Approval has been received from the Commission for cold zero-power operation. Preparation and checkout for operation is now complete and loading of the fuel has started.

Earlier in the month preparations were made for forced-circulation flow distribution tests. Rod drop tests were run. As a result of the reduced control rod channel spacing in the core structure shroud and the advisability of inspecting the reactor again, the control rod drives and seals were dismantled and the control rods, the forced-convection baffle, the feedwater sparger and the core structure were removed. The control rod channels were restored to design dimensions and checked with a cruciform gage. The core structure, control rods, and associated reactor vessel internals were reinstalled. Dummy and empty fuel assembly boxes, and sources and instrument thimbles were loaded into the core structure and aluminum and stainless steel corrosion sample bundles were installed on the outside of the shroud. The four in-vessel startup counters, extension spool, and source drive were installed and checked out. Rod drop tests were repeated and all applicable startup checkout lists were completed before loading of fuel commenced.

Modification of the boron addition tank piping and heating system has been completed and the system has been hydrostatically tested. Tubular heaters were clamped to the bottom of the tank. The tape heaters were reinstalled on the boron system piping and level gauge glasses. Wiring of the control circuit and heaters was completed and the system checked out.

The electrical wiring for remotely controlling the position of the antimony source with relation to the beryllium has been completed. This includes a position-indicating system. All in-vessel thermocouples have been connected to their respective recorders and are ready for service. The downcomer density and velocity-measuring probes and instruments are now ready for service. To provide for timing of control rod drop tests for drops to the 4-inch position the circuit design has been modified and equipment has been ordered. This will permit all manipulation from the control room for timing rod drops, either to the dash pot or including the dash pot.

All components on the sampling station are operational except the oxygen analyzer and the steam fission product monitor.

2. Procurement and Fabrication

a. Superheater Fuel - Inspection was completed on the 380 central and 30 peripheral superheater enriched plates received at Argonne, Illinois, with nine plates rejected. Rust spots were found on an additional 21 type FCE plates. Final acceptance of these plates depends on the results of corrosion tests. Two hundred and sixteen type HPE and 64 type FPE plates have also been received.

No brazing work on four-plate superheater fuel elements was done this month. A second brazing furnace has been installed and is being checked out. Preparations have been made for a five-element braze in two furnaces.

It was inferred in the November Progress Report (ANL-6473, p. 11) that exposure to water at 300°C for two weeks caused a four-plate vacuum brazed Borax-V superheater assembly to bow an estimated $\frac{1}{16}$ in. in the long direction. However, it is more likely that the assembly was bowed prior to test. The same test was repeated for another assembly and bowing of only a few mils occurred during the test.

b. Reactor Components - Fabrication of the end closure pieces for new control rods of revised design continued. Modifications to strengthen the mechanical joint between the aluminum follower and stainless steel cruciform extension have started.

Six 17-4PH stainless steel control rod extension shafts have been chrome-plated, honed, and are ready for final inspection. The remaining extension shafts are ready for plating.

c. Experimental Components - Fabrication of the duplex flux wire counter is essentially complete and the machine is being calibrated and tested using flux wires irradiated in TREAT.

Fabrication of the remotely-controlled, high pressure steam sampling probe is nearly complete. This device will also be used as a source drive during cold and hot critical operation.

Twelve high-temperature, Ta-sheathed, W-W/26% Re thermocouples have been successfully installed in boiling fuel rods which are ready for assembly into instrumented fuel assemblies. Ten are in 4.95% enriched rods and two in 9.90% enriched rods. The latter two are for experiments with individual fuel rods. Installation of the remaining thermocouples in boiling fuel rods is being delayed pending satisfactory in-core performance of the 12 thermocouples on hand.

All three instrumented boiling-fuel-assembly exit flow meters have been received. Brazing development work for the pressurized terminal box seal plates and superheater fuel plate thermocouples has restarted.

Fabrication of the superheated steam fission product monitor was completed. An automatic, continuous chloride monitor has been procured (on loan from Westinghouse) and adaptation for installation on BORAX-V is in progress. Fabrication and procurement for the H_2 - O_2 recombiner test rig has started.

Specifications are being prepared for the purchase of two types of high temperature in-core neutron flux detectors. Dual purpose miniature fission chambers and/or fission heating devices are being proposed for total fission rate and thermal fission rate measurements.

3. Design

Detailed design was completed on the variable speed oscillator rod drive with sine potentiometers, the boiling fuel rod gamma-scanning machine, and the movable high pressure steam probe and its control circuits. Detailed design continued on the fuel handling coffin.

Engineering design was completed and detailed design started on equipment to test the performance of a platinum catalyst H_2 - O_2 recombiner in steam at 600 psig saturated conditions. If these tests are successful, it is planned to add a platinum-coated wire layer to the central superheater demister type steam dryer to reduce the oxygen content of the steam entering the superheater and thus to inhibit the corrosion rate of the superheater fuel.

Engineering design of the root-mean-square noise analyzer was completed and all components have been ordered.

Design was started on a pneumatic "rabbit" type step function generator to be used as an additional method of determining reactor stability.

Special loading fixtures for the initial non-radioactive loading of boiling fuel rods into boiling fuel assemblies were designed and built.

Stress calculations and mechanical design for the new system of anchors and guides on the superheater drain line were completed.

Check calculations were run using a two-dimensional PDQ cell problem to verify the safety of the cadmium-lined boiling fuel storage cells located in the water storage pit. The k_∞ of the present arrangement is calculated to be 0.77; the actual reactivity of the array is considerably less. A criticality analysis of the 45 cadmium-lined superheater fuel storage racks proposed for the old BORAX reactor vessel indicates a k_∞ of about 0.92 for peripheral superheater assemblies.

4. Development and Testing

a. Plant Tests - The control rods were installed and test operated with the reactor vessel at room temperature and atmospheric pressure. Rod drop times were measured to be between 1.56 - 0.90 sec for full 29-in. travel including the dash pot, and from 0.56 - 0.49 sec for 25-in. travel from the 29-in. position to the beginning of the dash pot at the 4-in. position. The tests were repeated with the reactor vessel at 600 psig and at room temperature. The times were measured to be from 0.62 to 0.40 sec for full travel, and from 0.26 to 0.24 sec for 25-in. travel from 29 in. to the dash pot at the 4-in. position. The control rod seals leakage rates at these conditions with the control rods at the "down" position was between 2 and 20 gph. The leak rates with control rods "up" was greater than 20 gph.

During these tests the force necessary to raise control rods after dropping them with the reactor at pressure was found to be excessive. The seals, control rods and shrouds were inspected. The control rods were found to be in good condition. The control rod channel spacing in the X-8001 aluminum was found to be less than normal, but not small enough to cause interference with the control rods. The cause of the reduced spacing in the shroud channels was attributed to the low pressure created above the rods during high pressure control rod drop tests. This low pressure is caused by the restriction of the flow control orifices temporarily installed in the top of the channels. Hydraulic calculations indicate that with the orifice plate the differential pressure across the shroud is about ten psi and without the orifice plates the differential pressure is less than 0.5 psi, which is insufficient to affect the control rod channel shrouds. The flow control orifice plates for these channels have been removed since they will not be required for core sizes proposed at present.

The cause of the excessive force required to raise the control rods was found to be due to the seal supply water not being on. Therefore, pressure on the underside of the dash pot plunger was not normal, allowing a differential pressure of 600 psi across the dash pot plunger. The seals were found to be in good condition.

The startup and loading nuclear instruments were checked with control rods in various positions and the source in various positions. Additional instrument thimbles were fabricated to permit the positioning of four channels of nuclear instruments in the reactor vessel for startup. This will provide measurement of unmultiplied source level neutrons on four channels. Additional testing and adjustment was done to reduce the period circuit noise.

After the boron addition system piping and heating revisions were made, a hydrostatic test was satisfactorily completed and heating tests were made. The level gauge glasses and piping can be maintained at temperatures above 150°F when the tank pressure is brought to ten psig

and saturation temperature by the immersion heaters and maintained there by the tank bottom heaters. Further tests at 600 psig and saturation temperature are planned.

b. Control Rods - Successful non-warping furnace anneals of dimpled stainless steel cladding angles were made by clamping the sheet in a fixture. Attempts to fabricate a prototype rod with dimpled cladding were unsuccessful because of misalignment of the dimples due to non-uniform spacing. A clamping type die was used in an attempt to maintain precise dimple location, but without success. As a parallel effort, successful spot welding techniques were developed using the original double interface slug design except that the slugs are prelocated by spot welding to one side of the cladding before the rod is assembled. A prototype control rod of this design is being fabricated. The decision has been made to use the slug design on the new set of production control rods.

c. In-Core Instrumentation Development - One instrumented superheater fuel assembly venturi tube assembly (venturi tube plus riser tubes) was returned from Alden Hydraulic Laboratories with an incomplete calibration. The assembly is now back with Alden for completion of the required tests and submission of data called for in the contract. Data received as a result of the first tests indicate the unit to be useful in both forward and reverse flow directions (second and first pass superheater installations, respectively).

The test contract with Thermatest Laboratories for calibration of the high temperature W-W/26 Re thermocouples has been completed. Performance of the last group of four thermocouples in matching the published curve of emf vs. temperature was far superior to the first test group up to 4000°F. Each group had one mechanical failure in the sheath junction, at temperatures below 3000°F, which affected thermocouple performance in an erratic manner. Metallurgical and chemical tests are planned to determine the causes of the two failures.

d. Exponential Experiment - The water tanks, cadmium shell, fuel rod racks, track, drain-and-fill lines, counting circuits, etc. for the exponential experiment to test the cadmium ratio technique of void determination were installed in the north hole of the TREAT shield and checked out.

II. LIQUID METAL COOLED REACTORS

A. General Research and Development

1. Evaluation of Carbide and Oxide Ceramic Fuels for Large Fast Breeder Reactors (See also Section III.A.5).

An evaluation is being made to assess the ultimate potential of uranium oxide and carbide fuels in terms of obtaining minimal fuel cycle costs for large fast breeder reactors. Effort is directed toward a parametric heat transfer analysis of fast breeder cores using these fuels. This heat transfer data will be used in conjunction with the CYCLE code to calculate fuel cycle costs for typical breeder cores using each type of fuel.

The parametric heat transfer analysis of fast reactor cores using the two fuels is based on the following conditions:

(1) Maximum cladding temperature	1200°F
(2) $\int k(\theta)d\theta^*$ for oxide	20 watts/cm
(3) $\int k(\theta)d\theta^*$ for carbide	250 watts/cm
(4) Radial maximum/average power	1.5
(5) Axial maximum/average power	1.3
(6) Hot channel factor on coolant rise	1.3
(7) Hot channel factor on heat flux peaking	1.5
(8) Coolant temperature rise	300°F
(9) All cores are right circular cylinders	

All core outputs were normalized to a power of 1338 Mw by trial and error. Rod diameters of 0.125, 0.250, and 0.500 in. were studied. In addition, two limiting criteria were considered for each case: (a) a limit of 30 ft/sec was imposed on coolant flow velocity, in accordance with current practice in liquid metal cooled systems; and (b) the coolant velocity was permitted to increase until the flow-pressure drop through the core resulted in a pumping loss of 1% of thermal power (TP). The results of these calculations are presented in Table I.

As can be seen from the data in Table I, the 30 ft/sec limitation imposes a more severe restriction on the carbide fuels and it is necessary to have flow velocities on the order of 100 ft/sec if their full potential is to be realized.

In removing power from high power density reactor fuels, the mechanical and thermal properties of the cladding may eventually limit the power output.

*In each case the values quoted permit a 50% power transient before center fuel melting occurs.

Table I Comparison of Carbide and Oxide Fuels

Radial Diameter (in)	Fuel	Limiting Criterion	Velocity (ft/sec)	Power Density (Mw/L)	Coolant Volume Fraction	Heat Transfer Coefficient, Btu/(hr)(ft ²)(°F)	Temp Drop (°F)	Coolant Temp (°F)	Core Length (ft)
0.25	Carbide	1% TP	109	2.23	0.3438	45,900	108.5	1070	3.00
	Oxide	1% TP	42	0.220	0.1910	49,600	7.9	1107	6.50
	Carbide	v = 30 ft/sec	-	0.961	0.7173	10,400	478.8	795	3.98
	Oxide	v = 30 ft/sec	-	0.203	0.2536	35,000	11.4	1106	6.66
0.125	Carbide	1% TP	142	6.34	0.5336	47,500	210	1025	2.12
	Oxide	1% TP	55	0.731	0.3269	54,000	14.8	1105	4.35
	Carbide	v = 30 ft/sec	-	1.38	0.8987	7,443	1335	<600	3.51
	Oxide	v = 30 ft/sec	-	0.553	0.4911	28,100	28.3	1100	4.76
0.50	Carbide	1% TP	85	0.679	0.2003	41,900	59.5	1090	4.50
	Oxide	1% TP	33	0.0608	0.1049	44,500	4.5	1108	10.0
	Carbide	v = 30 ft/sec	-	0.474	0.4422	13,900	179.1	1040	5.0
	Oxide	v = 30 ft/sec	-	0.0603	0.1126	41,400	4.8	1108	10.0

The general design criteria for structures which have a combination of thermal and mechanical stress has been to restrict the total stress (sum of mechanical and thermal stresses) to a value not exceeding the yield point of the material. In the present connection, the design of fuel claddings severely restrict the fuel power density, especially the carbide fuels. Accordingly the procedure proposed in ANL-6389¹ has been used for cladding design for the above cases. In this method the mechanical stresses are restricted to a value less than the yield point; however, the additional thermal stresses are permitted to take the total stress to three times the yield point. Under creep conditions in the reactor the thermal stresses are relieved so that the total stress is then equal to the mechanical component only. This appears to be a reasonable approach, particularly so since the total plastic deformations required to accommodate the thermal stresses are small, approximately 0.20%. Using these standards, it is found that Inconel-X is a satisfactory cladding material for carbide fuels and Type 347 stainless steel is satisfactory material for the oxide for the highest burnups considered (50,000 Mwd/tonne, ~5 a/o).

The heat transfer data and corresponding coolant and cladding volume fractions are presently being put into CYCLE.

2. Isotopic Power Sources

A study has been started to explore the feasibility of making Pu²³⁸ and Cm²⁴² as useful byproducts in the operation of fast power reactors. Appropriate neutron cross sections have been estimated and the isotope production equations have been programmed for machine computations.

A literature search revealed that fast neutron cross section data do not exist for the americium and curium isotopes and that little data exist for the neptunium isotopes. Although the absolute values of the fast

¹S. Fistedis, "Design Criteria for Steel in Nuclear Reactors," ANL-6389 (July 1961).

cross sections, which are obtained by theoretical means, are not accurate, theory can predict the general energy dependence of the cross sections. This is expected because the neutron energy is almost great enough to apply the classical model for neutron interaction.

The values of cross sections of the uranium and plutonium isotopes (both capture and fission) are available from the literature. The values of $\sigma(n, \gamma)$ for the neptunium, americium, and curium isotopes are obtained by assuming that they are of the same magnitude as the $\sigma(n, \gamma)$'s of the thorium, uranium, and plutonium isotopes. This would be true for the total cross section in the classical model of interaction ($\sigma_T \propto \pi R^2$). A minimum and maximum value of $\sigma(n, \gamma)$ of the americium and curium isotopes are obtained from maximum range of the values of the $\sigma(n, \gamma)$'s for the thorium, uranium, and plutonium isotopes in each energy range. This results in a factor of two between the minimum and maximum $\sigma(n, \gamma)$'s of the americium and curium isotopes. The values of $\sigma(n, f)$ are similarly approximated from the range of experimental $\sigma(n, f)$'s of the heavy isotopes having the same change in pairing energy upon the absorption of a neutron. For example, $\sigma(n, f)$ of Cm^{242} is assumed to lie within the values of $\sigma(n, f)$ for U^{236} , U^{238} , Pu^{240} , and Pu^{242} . The value of $\sigma(n, 2n)$ for U^{238} is obtained by averaging the cross section with the fission spectrum above the threshold (6 Mev) for the reaction. Considerable error ($\pm 50\%$) however may be expected in this cross section.

The computer program calculates the quantities of the isotopes from the value of the cross sections (maximum or minimum), the flux for each energy group, the decay constants, the irradiation time, and the initial concentration of the isotopes.

The quantities of isotopes which are produced for various reactor conditions and possible methods of separating the isotopes will be investigated next.

3. Metallographic Examinations of Irradiated Refractory Metal-Clad Fissium Alloys

Irradiations were concluded late in 1961 on a group of refractory metal-clad uranium-fissium and uranium-plutonium-fissium alloy specimens. The specimens were prototype EBR-II fuel elements and were irradiated in instrumented capsules in the CP-5 reactor. Irradiation temperatures for most of the specimens were maintained near 700°C.

A total of 12 uranium-fissium alloy specimens were irradiated for periods ranging up to 13.5 months. Results from the initial postirradiation examination of these specimens are given in a previous Progress Report (November 1961, ANL-6473, pp. 32-33). The maximum burnup achieved in the specimens was estimated in that report to be 2.5 a/o. Mass spectrographic analyses recently completed on adjacent specimens indicate that the maximum burnup achieved was in fact approximately 4.1 a/o.

Metallographic examination of the specimens is now in progress. Initial examinations show that a thin reaction layer occurred between the fuel and cladding in the specimens clad with niobium. Little or no reaction was evident between the fuel and vanadium cladding. Extensive porosity was evident in all specimens examined, with the largest voids occurring near the center of the specimen.

A total of 18 uranium-plutonium-fissium specimens were irradiated for periods ranging up to four months. The maximum burnup achieved is estimated to be near 2.3 a/o. As reported in an earlier Progress Report (October 1961, ANL-6454), many of the plutonium alloy specimens were damaged by overheating. In some specimens temperatures as high as 1000°C were experienced, well above the fuel melting temperature of 820°C.

Metallographic examinations of these specimens are also being made. The initial examinations show extensive alloying between the fuel and cladding in most of the specimens in which melting occurred. As in the case of the uranium-fissium alloys, alloying was greater with niobium alloy cladding than with vanadium. The specimens irradiated above 700°C were highly porous as is shown in Figure 1. Figure 2 shows the extensive porosity which was also observed in the niobium cladding on the specimens which were exposed to the highest temperatures. This is probably the result of plutonium diffusing into the niobium and subsequently fissioning. A metallographic section made on a specimen clad with Nb-1 w/o Zr alloy, and believed to have been irradiated near 650°C is shown in Figure 3. The specimens showed little porosity in the fuel and a relatively thin reaction layer with the cladding alloy. It therefore appears that Nb-1 w/o Zr alloy may be a satisfactory cladding for the uranium-plutonium-fissium alloy (reference fuel for EBR-II Core II) under EBR-II design conditions.

Figure 1. Interface between Niobium Cladding and Molten Uranium-plutonium-fissium Fuel Alloy which was Deposited on the Outside of the Cladding. The Estimated Irradiation Conditions were 2.3 a/o Burnup at a Maximum Temperature Exceeding 820°C.

Figure 2. Interface between Niobium Cladding and Uranium-plutonium-fissium Fuel Alloy. The Estimated Irradiation Conditions are 2.3 a/o Burnup at a Maximum Temperature near 800°C .

Figure 3. Interface between Nb-1 w/o Zr Alloy Cladding and Uranium-plutonium-fissium Fuel Alloy. The Estimated Irradiation Conditions are 1.5 a/o Burnup at a Maximum Temperature near 650°C .

4. ZPR-III - Experimental

The program of experiments to be performed with the nickel-reflected core of the Fermi B mockup, Assembly 35, was completed. Included in the final work of this program were traverse of fission counters and reactivity coefficient samples, measurement of Be and BeO reflector worths, measurement of the Fermi control rod worth, a Fermi Be source experiment, and determination of Rossi-alpha. Based on the results of the series of reflector experiments, APDA considers that nickel oxide would

possibly be used in the Fermi reactor. Therefore, the next experiment will be a replacement of the nickel reflectors with a nickel oxide composition and a repetition of a few measurements made in the nickel-reflected core.

In the December 1961 Progress Report (ANL-6485) the relative worths of Ni, NiO and Fe₂O₃ reflectors were listed. The values reported in that table were in error. These are correctly tabulated in this report with the recently measured Be and BeO reflector worths (Table II).

Table II. Results of Reflector Experiments

A. Experimental compositions and worths of 18 drawers.

Reflector Type	Composition, g/cc						Worth Rela- tive to Ni Reflector (inhours)
	Ni	Na	Fe	SS	O	Be	
Ni	5.43	0.16		.96			0
Fe ₂ O ₃		0.16	1.74	1.35	0.69		-49
"NiO"	2.72	0.16	1.07	.96	0.43		+6.5
Be		0.16		.96		1.17	+66
"BeO"		0.08	1.07	.83	0.43	.78	+43

B. Reactivity coefficients in reflectors

Reflector	Material worths, inhours/kg					
	Ni	Na	Fe	SS	O	Be
Ni	1.6	7.8	1.4	1.5	7.0	11.3
Fe ₂ O ₃	2.0	5.7	1.4	1.6	6.6	
Be	1.5	5.1	1.2	1.2	6.3	6.9
BeO		5.9	1.5	1.6	5.9	8.4

a. Fission Counter Traverses - Traverses of U²³⁵, U²³⁸ and Pu²³⁹ fission chambers and a BF₃ counter were run at the radial edge of the core parallel to the core axis. The counters ($\frac{3}{8}$ in. in diameter by 2 in. long) were moved remotely in a tube running through drawers 1 and 2-H-16. In Table III the count rates (normalized to 100% at the core midplane) are given for several distances from the midplane.

Table III. Counter Traverses at Core Radial Edge, Assembly 35

Axial Distance from Midplane (in.)	Relative Count Rates (% of Midplane Count)			
	Enr. U	Nat. U	Pu ²³⁹	BF ₃
32	3.1	0.3	4.4	6.0
24	17.5	3.7	16.2	15.8
20	35.4	12.9	33.2	32.4
18	46.2	30.2	44.1	43.0
17	50.8	40.7	49.0	48.4
16	55.3	51.9	55.7	53.0
15	60.0	57.8	-	58.5
14	63.2	62.7	65.2	62.6
13	69.3	-	-	-
12	-	72.6	73.7	71.6
8	85.8	88.3	85.0	85.8
6	90.5	94.0	91.0	90.9
4	95.6	98.3	93.4	95.9
3	97.2	99.5	95.5	96.0
2	98.6	100.3	98.3	96.5
1	100.1	98.9	98.9	98.0
0	100.0	100.0	100.0	100.0
- 1	98.9	100.9	102.4	99.0
- 2	99.8	99.2	98.9	101.8
- 3	-	-	98.3	97.4
- 4	96.5	98.8	96.8	97.8
- 6	-	-	90.7	93.4
- 8	87.9	91.1	89.6	87.4
-12	-	-	75.9	74.6
-13	-	-	68.2	-
-14	66.0	-	64.9	66.0
-15			60.8	

b. Reactivity Coefficient Traverses - Small samples of enriched and natural uranium were also run through the traverse tube set in 1- and 2-H-16. The worths of these samples were obtained at several axial positions. The traverse mechanism was next set up to run radial traverses through column-16 drawers near the core midplane. The worths of the uranium samples were then obtained at several radial positions. Table IV presents the experimental results.

c. Be and BeO Reflector Experiments - Experiments were carried out with Be and BeO reflectors as a continuation of the program started in December, 1961, for finding the relative worths of differently composed radial reflectors.

Table IV. Uranium Reactivity Coefficient Traverses

A. At Core Radial Edge Parallel to Axis ($r = 17.5$ in.)

Axial Distance from Midplane (in.)	Worth of 93% Enriched U (lh/kg)	Worth of Depleted U (.2%25) (lh/kg)
45	0	0
32	0.8	
28	0.8	
24	2.1	
20	2.5	
18	4.1	+0.5
16.5	6.6	
15	9.1	
12	-	
9	15.7	-0.8
6	-	
3	21.1	
0	23.1	+0.3
- 3	23.4	
- 9	18.8	
-15	9.6	
-16.5		
-18	6.1	

B. Radial Traverses Near Core Midplane ($z = \frac{3}{8}$ in.)

Radial Distance from Core Center (in.)	Worth of 93% Enriched U (lh/kg)	Worth of Depleted U (.2%25) (lh/kg)
32.8	0	0
28.3	0	+0.6
22.9	6.5	
20.7	15.3	+0.9
19.6	16.6	
18.5	20.1	-0.5
17.0	23.6	
16.0	26.6	+1.1
14.0	33.6	
12.0	46.2	+1.1
10.0	55.4	
8.0	67.2	+0.9
6.0	74.1	+0.9
3.0	80.2	-0.6
0	82.9	-1.7
-3.0	81.1	-2.2

In these experiments, the different compositions were placed in 18 drawers comprising a one-row thick reflector segment about one quadrant of core half #2. For the Be reflector composition, given in Table II, the reference loading was the normal nickel reflector.

Replacement of the nickel with beryllium was done in two steps of nine drawers each. The first nine-drawer substitution was worth +14.3 in-hours, whereas the second nine drawers surprisingly were worth +52 inhours. To check whether the increased worth was due to the presence of the first nine drawers, the first nine were changed back to nickel while the second nine were in place, with a resulting loss of 18 inhours. It was then thought that the major part of the difference of worths between the first and second nine drawers was a question of fuel proximity, so three columns of enriched uranium near the core edge were each moved about one inch closer to the first reflector drawers. These nine drawers were then changed again from Ni to Be with a resulting worth of +42 inhours. This shows a definite fuel-Be proximity effect. It would have been interesting, had there been time, to investigate the effect further to see whether neutron moderation or the Be (n,2n) and Be (γ ,n) reactions are principally responsible.

With the Be reflector in place, reactivity coefficient measurements were made to provide corrections in reactivity for differences between the experimental composition and a possible Fermi design composition. Next, the 18 drawers were changed from a Be reflector to an approximate BeO reflector where Fe_2O_3 was used to provide about 40% of the oxygen required in a BeO composition. Again material worth measurements were made to adjust the experimental composition to a possible Fermi design. A summary of the reflector experiments done in December and January is given in Table II.

It is interesting to note the variation of the material worths according to the type of reflector in which the worths were measured. The data obtained allow us to calculate the worth of possible compositions for a full-row reflector around the Fermi core. Table V lists possible design compositions for a reflector in the Fermi reactor and the reactivity worths for a full row (2.8 in. thick).

Table V. Possible Design Compositions and Worths for a Reflector in Fermi B Reactor

Design Type	Composition (g/cc)						Reactivity Worth Relative to Ni (inhours)
	Ni	Na	Fe	SS	O	Be	
NiO	3.22	0.23		1.21	0.88		+832
Fe_2O_3		0.23	1.92	1.21	0.82		- 90
Be		0.23		1.21		1.01	+450
BeO		0.23		1.21	1.06	0.60	+849

It thus appears that the optimum reflector for use in the Fermi reactor is the BeO design. However, a large uncertainty is present for the worth of this reflector due to the anomalous results and fuel-proximity effects noticed during the Be experiments.

d. Fermi Control Rod Experiment - Control of the Fermi reactor is maintained by two ten-inch-long enriched boron carbide control rods in subassemblies near the core axis. A mockup was made of a rod whose B^{10} content would be worth approximately -2 dollars reactivity at the core center. This was then "inserted" to the top of the core and a worth obtained. For the central worth measurement, the rod was inserted in two steps, putting a five-inch-length in half #1 and then in half #2. The worths were found by subcritical methods. Next the enrichment of ten inches of B_4C at the center was changed to two lower values to obtain approximate worths of \$1.50 and \$1. This was done by replacing a portion of the 90% enriched B_4C with natural B_4C . Table VI gives the experimental compositions and results of these control rod experiments.

Table VI. Fermi Control Rod Experiments

Poison Section - 2.2 x 3.3 x 10 in. - 1.19 liters

B^{10} Enr.	B^{10}/cc (g)	B^{11}/cc (g)	C/cc (g)	Na/cc (g)	SS/cc (g)
90%	0.217	0.024	0.096	0.443	1.77
65%	0.167	0.088	0.093	0.443	1.77
44%	0.118	0.152	0.089	0.443	1.77

Worth of 90% enriched rod relative to empty Na channel,
 - to top of core -30 inhours
 - symmetric about midplane. -495 inhours
 Worth of 70% enriched rod, relative to Na channel,
 - symmetric about midplane. -407 inhours
 Worth of 50% enriched rod, relative to Na channel,
 - symmetric about midplane. -316 inhours.

e. Beryllium Source Experiment - A mockup was made of the assemblage of beryllium used in the Fermi source. This amounts to about 65 volume percent Be (35% Na and SS) in a 2.7 x 2.7 in. subassembly channel at the radial edge of the core for a length of 26 in. The worth of this mockup relative to average core composition was found to be +8.8 inhours. The main purpose of the experiment, however, was to obtain the fission rate of U^{235} in the vicinity of this source to permit calculation of the heating effects. Therefore, foils of enriched uranium were irradiated during a 35-watt-hour run and their activations determined. The results showed a U^{235} fission rate next to the Be which was about 30 percent higher than at the core center.

f. Fission Ratios and Rossi-alpha - A final set of fission rate measurements has been made with Kirn-type absolute fission chambers to obtain the fission ratio of U^{233}/U^{235} , U^{234}/U^{235} and Pu^{240}/U^{235} . At the end of the month, work was under way for the determination of the Rossi-alpha factor in this assembly. The interest in this experiment concerns the effect of the nickel reflector and the large core size on the methods used in the measurement.

5. ZPR-III - Theoretical

The Rossi- α ratio, $\alpha = \beta_{\text{eff}}/\ell_p$, where β_{eff} is the effective delayed neutron fraction and ℓ_p is the prompt neutron lifetime, has been calculated for the aluminum diluent ZPR-III Assembly No. 23. The perturbation theory method was used with diffusion theory group fluxes and adjoints obtained using the 16-group YOM set with the modifications suggested in ANL-6473 (Progress Report for November 1961).

The $\alpha_{\text{calc}} \approx 7.2 \times 10^4 \text{ sec}^{-1}$ is about 27% larger than the reported¹ experimental value $5.65 \times 10^4 \text{ sec}^{-1}$. The deviation is about the same percentage as previously found² for the ZPR-III Assemblies Nos. 6F, 9A, 22, 24, and 25 containing U^{238} as diluent and calculated using a ten energy group cross section set. This suggests that the calculated spectrum of Assembly No. 23 is also not sufficiently degraded in energy.

6. ZPR-VI and ZPR-IX

a. Building - The cells in Building 335, as reported last month, were found to be quite porous under pressure test. To reduce the leakage rate, it was decided to coat the interior walls of the cells with a phenolic resin base paint. Bids have been received for the painting of cells Nos. 4 and 5. Bids have also been received for the argon gas purging system for the cells. These are being evaluated. Specifications are being prepared for the emergency venting system for the cells. The installation of the fuel storage racks in the vault and vault workroom has been completed.

b. Bed and Table Assembly - An additional solenoid operated table travel stop is being designed for installation on the movable table. This will simplify the interlock circuitry. All installation work has been postponed until the painting of the cells has been accomplished.

¹ Long, J. K., et al., Seminar on Physics of Fast and Intermediate Reactors, SM-18/48, Vienna (1961).

² Meneghetti, D., Seminar on Physics of Fast and Intermediate Reactors, SM-18/37, Vienna (1961).

c. Matrix Assembly - A total of 163 matrix tube bundles have been assembled by Central Shops. This is sufficient for the matrix assembly. The bundles will be stored until installation in the facility can begin.

d. Control and Safety Rods - The dual purpose control/safety and the insertion safety rod drive mechanisms are being delivered by the manufacturer. These drives will be wired and tested.

e. Control Console and Instrumentation - Testing of the instrumentation of the two control consoles is in progress. A preliminary list of discrepancies was given to a representative of the vendor for corrective action.

7. JUGGERNAUT Critical Experiments

The JUGGERNAUT reactor went critical January 11, 1962, with a fuel loading of 3.44 kg of U^{235} . The measured excess reactivity with this loading was 0.43% $\Delta k/k$ and the fuel worth was 0.76% $(\Delta k/k)/100 \text{ gm } U^{235}$, leading to a critical mass of 3.38 kg. The calculated critical mass was 3.3 kg (in error by 0.6% $\Delta k/k$), and the calculated fuel worth was 0.78% $(\Delta k/k)/100 \text{ gm } U^{235}$.

The worth of each shim-safety rod has been determined by rod drop and subcritical multiplication methods. The measured rod worths are in fair agreement with theory though certain discrepancies are apparent.

The combined worth of the shim-safety system was measured with the reactor in the cold, clean condition as 7.7% Δk . With the beam holes open it is expected that the total worth of the system will be reduced to approximately 7.0% Δk . The worth of the shim-safety system was calculated assuming four rods to be symmetrically located around the periphery of the core. The latest and best calculated value for this configuration is 4.8% Δk . This compares to a measured value (i.e., the worth of the four safety rods) of 4.6% Δk . Assuming the worth of the three-rod shim system to be equal to three-fourths the worth of the four-rod system leads to a calculated shim worth of 3.6% Δk , which is in good agreement with the measured value of 3.7% Δk . In order to obtain the worth of the total shim-safety system (seven plates) the worth of a four-rod system with the same plate area as the seven-rod system was obtained. The calculated worth of the system is 6.4% Δk , compared to a measured value of 7.7% Δk . It is now obvious that this method of obtaining the total worth of the seven-rod configuration gives too large a shadowing effect between rods.

The worth of the fine control rod was measured as 0.13% $\Delta k/k$ compared to a predicted worth of 0.15% $\Delta k/k$. The total effect of removing the graphite plugs from the four beam holes was measured as -0.6% $\Delta k/k$ which is in fair agreement with the calculated effect of -0.92% $\Delta k/k$.

On January 18, 1962, the fuel loading was increased to 3.54 kg ($K_{ex} = 1.1\% \Delta k$) and certain rod worth measurements were repeated. The reactivity effect of removing graphite stringers from various experimental facilities is being measured.

B. EBR-I

1. Operations

In recent months, reactor operating time has been negligible. However, routine maintenance and some special work has been performed in preparation for the Mark IV core. Primarily, modifications have been made to the prompt scram circuits to make them entirely fail-safe on loss of power.

During the past month, the neutron source which had decayed to a low level, was replaced. A new antimony slug, irradiated for one MTR cycle during the month of November in an unperturbed flux of 2.9×10^{14} , was installed. The source strength is estimated to be 120 curies and is worth a factor of 160 in neutron count at the startup instrumentation with the reactor in the shutdown position. No operation other than low power criticals will be run until instrumentation changes have been completed and checked out.

2. Fabrication of Core IV Fuel Elements

A total of 555 Core IV fuel and blanket rods are being fabricated for use in EBR-I. This number includes 420 fuel rods, 120 blanket rods, 10 fuel thermocouple rods and 5 blanket thermocouple rods.

ANL fabrication of jacket tubing for 100 fuel rods is continuing. Base tubing to provide an estimated minimum of 43 jackets for nondestructive testing was extruded at ANL. A total of 154 tubes are in process.

The Laboratory's Shops have completed finish-machined fuel connectors for top end closure of 100 jacket tubes.

Several years ago equipment was designed to test the wire used as spacers on reactor fuel elements. The effectiveness of this equipment has been re-evaluated and a number of changes have been made in both the equipment and operating procedure. New standards have been obtained and enough destructive tests have been performed to prove that the equipment is in fact doing the job for which it was designed. This equipment is now being used to test the spacing wire used on the Mark IV fuel. To date about 140 ft of wire have been tested and less than 5 in. has had to be scrapped because of defects.

C. EBR-II

1. Reactor Plant

During this month, work continued on preparations for the primary tank dry heat test and for wet critical. Of the work list of items remaining prior to filling the primary tank with sodium, approximately 50 (of the total of 130) were completed. This included all preparations for the dry heat test.

The heated air test has three objectives: (1) to determine temperature distributions throughout the primary tank for the temperature at which sodium will be introduced into the tank; (2) to verify calculated temperature drops through the primary tank cover and resulting deflections and rotations at elevated temperature; (3) to check operation of the fuel handling system at an elevated temperature. Although it will not be possible to attain normal operating temperature (700°F) in the primary tank without sodium, it is expected to be possible to reach a temperature of 300°-350°F during this test.

Deflection measuring devices were installed in several nozzles in the primary tank cover and on the primary tank hangers to be used to obtain deflection data during the test. Approximately 40 temporary thermocouples were added in the primary tank to obtain a better survey of local temperatures on the primary tank near the immersion heaters. These thermocouples will show any local hot spots which might occur in a major structure causing undesirable temperature gradients during heating. All permanent thermocouples to be used during the test were also calibrated and connected to recorders or indicators.

Three groups of subassemblies were installed in the reactor for checking fuel handling operations at the elevated temperature. One group is composed of dummy core subassemblies loaded into the center of the core region. Two groups of outer blanket subassemblies were loaded at the outer edge of the outer blanket in two islands located 120° apart. This will provide a check of fuel handling over a large region of the reactor.

Six special fans, to provide air circulation in the tank, were installed and/or connected. Three of these fans were previously used during the dry critical experiment isothermal temperature coefficient test. The three new fans can be removed from the operating floor and will be used to circulate the argon in the primary tank when heating prior to filling with sodium. To determine their effectiveness, they will be the only fans used until the primary tank reaches the sodium filling temperature (approximately 250°F at the bottom of the tank).

At 0830 on January 23rd the dry heat test was started. A complete set of deflection measurements were made at the beginning of the test to establish ambient temperature conditions. At month's end, the primary tank temperature has been rising satisfactorily at a rate of 1-2°F per hour. No unexpected or excessive temperature gradients have been observed thus far.

2. Power Plant

The turbine lube oil system was cleaned by completely draining the main oil tank and all pockets in the system, then completely wiping the system and finally returning the oil to the tank through a filter press. The oil itself appeared clean upon removal. However, a layer of dirt was present on the bottom of the tank. A sample of the oil was given to the vendor's representative for analysis.

The startup boiler feed pump was prepared for service and started. Its operation was satisfactory, and a great deal of dirt was flushed from the feedwater systems.

Prior to an attempt at synchronization of the turbine-generator check out of the electrical system was performed. The check revealed that the phase connections from the generator had been wired in reverse sequence. These were reconnected in the proper sequence. Further, the original connections were apparently not tight enough. This situation is being corrected.

3. Sodium Boiler Plant

The welding of stainless steel secondary sodium piping commenced following satisfactory laboratory chemical analysis of deposited weld metal. Installation of the superheaters continued. This work packet is nearing completion with stress relieving of the chrome moly welds currently being accomplished. Some welding remains on the joints to be cold sprung. The repaired evaporator was helium tested, set, aligned, and welding begun. Work continued on several items of both continuance and Package IV work.

The modification of the surge tank supports was completed. The sodium tank car unloading piping including electrical heaters was essentially completed. All the accessible stainless steel welds made by the original contractor were inspected to determine the ferrite content by magnetic test per contract specifications. The testing of welds made under corrective or Package IV work was begun.

4. Engineering

a. Special Subassemblies for Sodium Cleanup - Two general types of subassemblies are to be employed during the sodium cleanup period immediately following the filling of the primary tank. One type is the special filter subassembly. All of these have been fabricated and delivered to the field. The other type is the "plug" subassembly, which is used to block off flow through certain of the reactor grid holes. Fabrication of the latter type has started, but no delivery to the field as yet has been made.

5. Fuel Reprocessing Facilities

a. Fuel Cycle Facility - The Fuel Cycle Facility is slightly more than 98 percent completed. The low rate of finishing work results from the fact that most of the manpower has had to be assigned to corrective work.

Effective January 13 the uncompleted Package 3 contract work was deleted from the construction contract. All the remaining construction work will be done by the J. F. Pritchard Company, under the Package 4 contract, which is essentially an equipment installation contract under the direct supervision of ANL field personnel.

Corrective work in the Fuel Cycle Facility was devoted primarily to lapping the corroded service sleeve flange faces and to welding backup flanges to those flanges which had become undersized as a result of the lapping operation.

Neoprene and natural rubber gaskets, typical of those for the large transfer locks, are undergoing γ -irradiation tests. Irradiation of the neoprene sample compressed in a test rig was terminated after a total exposure of 3.0×10^8 rad. The neoprene sample had hardened and it exhibited very little resiliency. Irradiation of a sample of gum rubber compressed in the same test-rig has been started.

Samples of silicone rubber for use as a gasket material in the refueling machine and interbuilding coffin were irradiated. After exposure to 2×10^7 rad the silicone rubber was still quite flexible. After exposure to 4×10^7 rad, the material was flexible, but fractured when bent 180 degrees. This is approximately the same radiation life as that possessed by another high-temperature elastomer, Viton. It is estimated that this level of radiation resistance will permit the use of these materials for about six months under processing conditions.

An attempt was made to oxidize a seven-kilogram uranium ingot in a prototype skull oxidation furnace. The experiment indicated that additional measures need to be taken to insure an even oxidation rate.

b. Fuel Subassembly Dismantler and Fuel Element Decanner - A prototype Fuel Subassembly Dismantler is being developed for removing the fuel and blanket elements from a fuel subassembly. (ANL-6473, p. 28, November, 1961). The lathe type bed which supports and positions the various cutters and drives has been set up on its support structure. The hex tube pipe-type cutter, an experimental hex tube longitudinal slitter, the pipe-type cutter for the fuel subassembly shank, and the rotation drive have been given a preliminary check-out.

Testing is now being carried out on the remaining principal subassemblies of the Fuel Element Decanner which were delayed two months because of improper fabrication by the commercial supplier. These included major items, such as the fuel element shears, the feed and wire stripping mechanism, and the fuel chopper unit.

6. Core II Research and Development

Niobium-1 w/o zirconium alloy, the references cladding material for Core II, is being prepared for jacketing some irradiation test specimens. This tubing has been radiographed and sectioned to remove defects, (cracks, tears, etc.), as determined from evaluation of the radiographs. Following this the tubing was hydraulically pressure tested at 4,000 psi, which was 400 to 800 psi below the yield point. The tubing was then cleaned and leak checked with a helium mass spectrometer and found to be satisfactory for its intended use.

7. Process Development

a. Melt Refining Process Technology - Preparations are being made for the investigation of the behavior of condensable fission products volatilized during the melt refining of highly irradiated EBR-II prototype fuel alloy. The two fission products of primary interest are cesium and iodine. The small-scale equipment and remote procedures have been tested in experiments utilizing about five grams of unirradiated uranium-fissium alloy.

b. Skull Reclamation Process - Reduction of uranium-fissium oxide in a beryllia crucible under the same conditions as those employed in the reduction step of the skull reclamation process was carried out to evaluate the possible contamination of the metal phase with beryllium. The reduction was carried out at 800°C in a slip-cast beryllia crucible, 2-in. ID by $4\frac{3}{16}$ in. deep. The charge consisted of 19 g skull oxides (79.5 percent uranium), 14.8 g magnesium, 282 g zinc and 74.5 g magnesium chloride - calcium chloride - magnesium fluoride flux. A sample of the metal phase taken after 14 hours of reduction showed that only about 60 ppm beryllium were in solution. This low concentration of beryllium suggests that beryllia merits consideration as a container material for the reduction step of the skull reclamation process.

Besides showing promise of utility in the reduction step, beryllia continues to be a promising container material for the last three steps of the skull reclamation process. These steps are (1) precipitation of the uranium-zinc intermetallic compound and removal of the supernatant, (2) decomposition of the intermetallic compound with magnesium to precipitate uranium, and, following removal of the supernatant, (3) retorting to remove residual zinc and magnesium. Because hairline cracks develop in isostatically pressed beryllia crucibles upon usage, the possibility of fabricating jacketed crucible assemblies which might permit continued operation with the cracked crucibles is being considered. Also under consideration is the possibility of fabricating large, process-size crucibles by a new forming method, namely, thixotropic casting. Crucibles formed by this method have a lower average density than those formed by isostatic pressing (83 percent as compared to 90 percent of theoretical density). However, thixotropically cast crucibles have fewer internal stresses and may, therefore, prove to be less subject to cracking.

Transfer tube units which will permit transfer of molten salts and metal phases and separation of the two phases have been undergoing preliminary tests. Although a considerable amount of metal transferred with the flux phase in the initial run, the quantity of metal transferring with the flux was greatly reduced after the units were improved. The metal loss after the improvements were made amounted to 78 grams for 3.5 kg of metal transferred. This loss may be tolerable in the skull reclamation process; however, it is believed that the loss can be reduced further by additional improvements of the equipment.

When uranium oxides are added to molten salts containing magnesium chloride, a compound, phase "D," (see Progress Report for November 1961, ANL-6473, page 31), is formed which is believed to be magnesium uranate, MgUO_4 . A correlation appears to exist between the presence of the uranate and the reducibility of uranium oxides by zinc-magnesium alloy in the presence of halide flux. Investigation of the formula and structure of the compound is continuing.

c. Blanket Processing - In the blanket process, uranium blanket material is dissolved in about 14 weight percent magnesium-zinc alloy at 800°C to produce a solution of about 14 weight percent uranium. The addition of magnesium to a 50 percent concentration precipitates the uranium from the solution while the plutonium remains in solution. The uranium solubility in the supernatant phase is reduced to about 0.03 weight percent by cooling to 400°C prior to the removal of the plutonium-bearing supernatant. Isolation of the plutonium from the supernatant will probably be accomplished by distilling away the magnesium and zinc.

A fourth demonstration of the blanket process has been started. In this latest run, direct dissolution was made of the stainless steel-clad uranium blanket pins and plutonium. A number of the major fission product elements (inactive) which are expected to be present, such as strontium,

zirconium, molybdenum, ruthenium, rhodium, palladium, tellurium, barium, lanthanum, cerium, and neodymium, were added to the original charge. The run has progressed through the uranium precipitation step. Analytical results indicate that the uranium and plutonium concentrations in the dissolution step and in the precipitation step were unaffected by the presence of iron, chromium and nickel from the stainless steel cladding and the various fission products. Final evaluation awaits the completion of the run and additional analytical results. To determine the fates of iron, nickel and chromium it is planned to repeat the experiment without plutonium so that analytical procedures may be simplified.

d. Plutonium Recovery Processes - Two methods of possible process interest for the separation of rare earth fission products from uranium and plutonium are being examined. The first method consists of selective extraction of the rare earths into zinc-calcium solutions. The results of a recent exploratory experiment indicate that 99 percent plutonium and greater than 99 percent uranium can be precipitated from a zinc-63.5 percent calcium solution at 424°C while 95 percent or more cerium remains in solution. The second method involves selective oxidation of the rare earths by magnesium chloride. By contacting a magnesium chloride-containing flux with a zinc-magnesium solution, the rare earths favor the flux phase, while the uranium and plutonium are expected to remain in the metal phase. Recent determinations of distribution coefficients (weight percent rare earth in flux/weight percent rare earth in metal phase) for yttrium, praseodymium, and samarium show the rare earths to favor the flux in general, but with minimum values in the region of 5 to 10 weight percent magnesium in zinc.

Preliminary results from a redetermination of the calcium-zinc phase diagram by differential thermal analysis show a significant departure from the literature data in the calcium-rich portion of the system. Additional work is needed, however, to identify the compounds and to clarify the details of the eutectic region around 30 atom percent zinc.

e. Reduction of Thorium Dioxide - Additional studies have been completed on the reduction of thorium dioxide to the metal by zinc-magnesium solution in the presence of certain fluxes. The optimum composition regions in calcium chloride-magnesium chloride-calcium fluoride flux systems for rapid, complete reduction are being defined. It appears that a minimum of 45 atom percent magnesium cation with about 11 mole percent calcium fluoride is required for complete reduction in four hours at 750°C.

f. Fused Salt Studies - Quantitative absorption spectra of uranium trichloride and uranium tetrachloride over the range 400 to 2400 $m\mu$ have been determined in lithium chloride-magnesium chloride melts. Comparison with known curves for these compounds in other molten halides shows no particular features peculiar to the lithium chloride-magnesium chloride system.

g. Materials and Equipment Evaluation - Tungsten continues to show considerable promise as a container material for the noble metal extraction and reduction steps of the skull reclamation process (and for halide flux-zinc-magnesium systems, in general). The stability of uranium-magnesium-zinc solutions in tungsten was confirmed by an additional solution stability run. A pressed-and-sintered tungsten crucible was unaffected by a series of cycles in which a two-phase flux and metal system was alternately frozen and remelted. Some of the cooling cycles extended to room temperature. In a preliminary test, tungsten appeared resistant to corrosive attack by a halide flux containing zinc chloride.

The adherence of vapor-deposited tungsten coatings on tantalum and molybdenum specimens was improved by annealing the coated specimens at 1500°C for three hours.

The purification system for the argon-filled dry box which is to be used as a welding facility employs a getter of titanium sponge and not of tantalum as reported in the Progress Report for December 1961, ANL-6485, page 30.

D. Fast Reactor Test Facility (FARET)

The general engineering and physics parameters for an experimental facility to test characteristics of advanced fast reactors are being examined. These studies are directed toward a preliminary design of a test facility showing general feasibility.

Because one of the design criteria for FARET is system temperatures, severe stress and thermal distortion problems arise in the design. For example, the reactor vessel cover temperature will be held closely to the coolant operating temperature to minimize temperature gradients between components. This, however, aggravates the sealing problem at the cover because of the high temperature at this point. The problem has been solved by the use of two closures, one gasketed closure containing the control rod mechanisms for use during reactor operation at high temperature and one replacement closure with rotating plugs and liquid seals for use during reactor loading operation at lower temperature. The replacement closure would also shield the fuel loading operator from induced radiation.

A simplified analysis was made to determine the temperatures of the reactor vessel closure and the replacement closure for shutdown conditions. This work indicated that the closure temperatures will permit access for primary sodium temperatures below 260°C (500°F).

It has been suggested that a thermally insulated experimental zone in the reactor may be utilized effectively to isolate reactivity changes due to a controlled change of fuel temperature within this zone during constant power

operation. For this reason a scheme was studied in which the test zone fuel was separated from the clad by a gas annulus. The variation of thermal conductivity of various gases was examined as a function of gas pressure for a constant power experiment. It was initially assumed that a desirable change in fuel temperature may be achieved by changing the thermal conductivity of the gas. However, preliminary results for several gases indicate that considerable pressures will be required (of the order of several hundred atmospheres, depending upon the gas used) to effect a change of conductivity by a factor of four. This work is continuing.

III. GENERAL REACTOR TECHNOLOGY

A. Applied Reactor Physics

1. 3.0 Mev Van de Graaff

a. Elastic Neutron Scattering - Total neutron cross section measurements are being made in conjunction with measurements of elastic neutron scattering to aid in the interpretation of the elastic scattering data. The cross section measurement utilizes the recently developed neutron-gamma ray discriminator for rejection of gamma rays, and rejection of the excited state neutron group from the $\text{Li}(p,n)$ reaction. The excited state group contributes appreciably to the reaction above 650 kev and is approximately 440 kev lower in energy than the ground state group. Thus, unambiguous measurements of structure in the total cross section can be made only if the excited state group is rejected.

Previous "good resolution" measurements of the total neutron cross section of iron do not extend beyond 650 kev. The present measurement has a neutron energy resolution of 2-3 kev and covers the energy range from 600 kev to 1050 kev. The results for iron are shown in Figure 4. The uncertainty in the measured cross section is ± 0.25 barns. It should be mentioned that the same neutron energy range in the "Barn Book" contains only three peaks.

b. $\text{Ni}^{58}(n,p)\text{Co}^{58}$ Reaction - Measurements of the absolute cross section of the $\text{Ni}^{58}(n,p)\text{Co}^{58}$ reaction are being carried out in the region of 1-1.5 Mev neutron energy. The absolute neutron flux is measured with a U^{235} fission chamber. Absolute yields of Co^{58} are determined by counting the 800 kev gamma-ray intensity of Co^{58} in coincidence with the associated annihilation radiation. Preliminary results are:

<u>E_n (Mev)</u>	<u>σ (mb)</u>
1.057	1.16
1.160	3.61
1.263	5.26

c. Instruments - The tritium-deuterium gas target (see Progress Report, November 1961, ANL-6473, p. 41) is in the process of assembly. Preliminary testing with the Van de Graaff generator is planned during the next month in order to determine the maximum allowable beam current and the "struggle" in the nickel foil windows. Initial operation will be with deuterium gas utilizing the d-d reaction. The combination of the deuterium gas target and the Van de Graaff generator will supply monoenergetic neutrons covering the neutron energy region from 4 to 6.8 Mev.

Figure 4. Total Neutron Cross Section of Iron

2. Fast Neutron Spectrum Measurements

In the $\text{Li}^6(n,\alpha)\text{H}^3$ reaction, the energy of the tritium or of the alpha particle is dependent on the angle between it and the incident neutron. With incident isotropic flux, this effect will broaden the energy distribution of tritium or alpha particles due to the interaction of Li^6 with each mono-energetic neutron group. The process of unfolding the neutron spectrum from the pulse height distribution, generated by the incidence of such particles on a junction detector, will consequently be a complicated one.

The electronic addition of the two pulses due to the tritium and the alpha particle formed in one $\text{Li}^6(n,\alpha)\text{H}^3$ reaction will produce a sum-pulse which does not show this angular dependence. The relative advantages of a spectrometer producing such sum-pulses have been investigated. Two ORTEC detectors faced a thick Zapon target covered with a very thin layer of Li^6 in vacuo. Detector-to-target distances were variable and provide some collimation. It was found that the collimation need not be severe, although a minimum amount of collimation was definitely necessary if an acceptable half width of the sum-pulse line at a detector to lithium target distance of 1.6 mm was 3%.

Calculations of the effective acceptance angles for conservative detector to lithium target spacings have shown that the geometric efficiency is sufficient to give count rates of about 10/sec in a fast neutron flux of $10^8 \text{ n}/(\text{cm}^2)(\text{sec})$. The development of the spectrometer has reached the stage where all of the attention may now be directed to miniaturization of the design. Two suppliers of detectors have been contacted regarding special designs of junction detectors having acceptable mechanical and electrical properties and minimum dimensions and moderating materials.

3. High Conversion Critical Experiment

Measurements were completed in the 1.27-cm triangular grid loaded with BORAX-V, 5-wt-% enriched UO_2 fuel. This core was replaced by a 1.27-cm sq lattice loading of the same fuel. Supplementary measurements were made to assist in problem areas from earlier work. Installation of a 1.18-cm triangular grid for 3-wt-% enriched UO_2 (Hi-C) fuel was begun. Atomic ratios of $\text{H}:\text{U}^{238}$ as low as 1:1 may be obtained in this grid.

Yield and traverse measurements were taken with U^{235} -aluminum, plutonium-aluminum, and U^{238} foils in the BORAX-V loadings. In combination with cadmium ratios of various materials and earlier measurements of lattice parameters these will be used in assessment of conversion ratios, ϵ , f , and neutron spectrum.

4. Reactor Physics Data Analysis, ZPR-VII

Work has commenced on a survey of lattice parameters for lattices consisting of $\text{Pu}^{239}\text{O}_2\text{-UO}_2$ fuel rods and light water moderator. Various enrichments of Pu^{239} and various fuel-to-moderator ratios are being considered. This work is being done in connection with the planned future use of plutonium fuel in the ZPR-VII assembly.

5. Theoretical Physics

a. Fuel Cycle Studies (see also Section II-A-1) - Before further proceeding with the fuel cycle parametric survey studies on carbide, oxide, and metal-fueled systems, the cross sections used in the calculations have been reviewed and modified from the previous calculations (see p. 44, ANL-6485, December 1961 Monthly Progress Report).

The ELMOE Code has been used to generate cross sections appropriate to the spectra of the core or blanket regions of each of the systems under examination. The core problems assumed a 15-cm reflector savings and the radii were, for each system, the values corresponding to the 350-Mwe systems. The blanket problems were performed for an infinite medium having the composition of a typical blanket zone for the system in question. The collapsed 16-group spectrum was slightly harder than the fluxes from a corresponding RE-122 problem. The case of the oxide blanket was repeated using a modified source spectrum which forced agreement between RE-122 and a fundamental mode calculation using the RE-122 homogenized cross sections. The resulting ELMOE elastic removal cross sections differed from the original ELMOE derived values by at most 2% or 3%. The transport cross sections were essentially unchanged by this spectrum shifting device.

Owing to the small spectrum errors involved, the cross sections derived from the first ELMOE problems will be used in subsequent CYCLE problems.

The new cross sections were used to repeat the 175-Mwe metal-fueled CYCLE problem. The converged problem agreed closely with the original calculation indicating that the modified cross sections had only a small influence on this system.

b. Mathematical Numerical Methods Analysis - Bauer¹ recently published an Algol version of a numerical integration algorithm due to Romberg.² It requires no stored coefficients, and by repetition of a

¹F. L. Bauer, "Certification of Algorithm-60 Romberg Integration," Comm. ACM, June 1961.

²W. Romberg, "Vereinfachte numerische Integration," Det Kongelige Norske Videnskaber Selskab Forhandlinger, 28, 30-36 (1955).

relatively simple procedure, can be made to give results which are exact (neglecting roundoff) for polynomials of arbitrarily high degree. This algorithm has been tested by programming it for the LGP-30 computer. It was found to be extremely flexible and convenient for integrating functions for which the values are calculable at arbitrary points. A certification describing this experience has been submitted for publication in Communications of the Association for Computing Machinery.

Aitken's³ algorithm has long been popular for interpolating functions the values of which are known only at irregularly spaced points. Hunter⁴ has recently indicated how a similar approach may be used to estimate the derivatives of functions. Unlike the traditional finite difference formulas, Hunter's approach lays no restriction on the spacing of the points, or on the point at which the derivative is to be estimated. An Algol algorithm formalizing Hunter's technique has been written, and checked by translation into the ACT-III language for the LGP-30 computer. The method is far more general than previous ones, but does not, of course, eliminate the inherent dangers of differentiating interpolation polynomials. Submission of the algorithm for publication is being delayed until publication of the redundancy check for Algol programs (Progress Report, December, 1961, ANL-6485 p. 45).

It was observed that with relatively slight modification, Hunter's approach could also be applied to the estimation of integrals, and repeated integrals, such as $\int_0^x \dots \int_0^x f(x) dx \dots dx$. Although study of the algorithm revealed it to be somewhat unstable with respect to roundoff errors, it may be applied to estimate integrals when the function is known only at a set of points for which no conventional quadrature formula exists. It is easily programmed, and has been tested on the LGP-30 computer. A note describing this extension of Hunter's method has been submitted for publication in the (British) Computer Journal.

B. Reactor Fuels Development

1. Corrosion Studies

a. Aluminum Powder Tubing in Corrosion Testing - Additional tubing for corrosion testing has been obtained from Armour Research Foundation and from the Torrance Brass Company. The ARF tubing

³ A. C. Aitken, "On Interpolation by Iteration of Proportional Parts, Without the Use of Differences," Proc. Edinburgh Math. Soc., Series 2, Vol. 3, p. 56 (1932).

⁴ D. B. Hunter, "An Iterative Method of Numerical Differentiation," Computer Journal, Vol. 3, p. 270 (1961)

extruded from A288 (1 w/o Ni-0.5 w/o Fe-0.1 w/o Ti) powder that had been ball milled 72 hours (to improve strength) failed by corroding excessively on the cut ends of the tubing pieces during a three-day test in 290°C water. Tubing extruded from as-atomized A341 alloy (1 w/o Ni-0.5 w/o Fe-0.3 w/o Cr-0.2 w/o Zr-0.1 w/o Ti) survived this short-time corrosion test and has been added to the long-time corrosion test of powder tubing already in progress at 290°C.

No corrosion data are yet available for the finned tubing extruded by the Torrance Brass Company from as-atomized A288 powder billets.

b. Lightweight Alloy for Liquid Mercury - In order to study the effect of heat treatment on the corrosion behavior of titanium alloys, alloy Ti-16 w/o V-2.5 w/o Al was tested in mercury at 371°C in the following pretreated conditions:

- A - Sample annealed at 674°C and slow cooled.
- B - Sample solution treated at 749°C for 20 minutes and water quenched.
- C - Sample solution treated at 749°C for 20 minutes and water quenched, followed by an age at 516°C for four hours.

The results of this test are given in Table VII. Duplicate specimens were used.

Table VII. Alloy Ti-16 w/o V-2.5 w/o Al Exposed to Liquid Mercury at 371°C

<u>Sample No.</u>	<u>Weight Loss mg/cm² for Total Period</u>		
	<u>168 hrs</u>	<u>336 hrs</u>	<u>720 hrs</u>
A	0.34	0.56	1.12
B	0.00	1.32	0.68
C	0.06	0.45	0.61

Cracking developed on Samples B during the first test period. It was accelerated in the following periods of exposure. However, no cracks were found on Samples A and C throughout the test.

The significant effect of aging on the corrosion behavior of titanium alloys has not been experienced previously. Metallographic examination of these samples is under way.

2. Ceramic Fuels

a. UC-PuC Bodies - Preparation and fabrication techniques for UC are being investigated as a complementary program to the work on PuC. Techniques which can be utilized in PuC preparation and fabrication are of most interest.

Work has been continued on precipitated UC produced by the Chemical Engineering Division (See Section III.F.3). Four more lots of material have been prepared. Two are similar to the first lots inasmuch as the carbon used for precipitation was activated charcoal. The remaining two lots were precipitated using -325M spectrographic grade graphite.

To date all sinterings have been done on specimens fabricated from the first lot. The as-received UC was ground dry in a porcelain mill with porcelain balls for four hours. After milling this powder it had an average particle size of from 4-6 μ . Earlier attempts to use Carbowax 4000 as a pressing lubricant and binder were unsuccessful because the binder could not be removed without completely disintegrating the specimen. Since specimens could be pressed with care without binder, several specimens were pressed and sintered in vacuum at 1650°C, 1750°C, 1850°C for two hours. Because of some internal cracking in these specimens, naphthalene was used as a binder in the last pellets pressed and sintered. Naphthalene is not a good pressing lubricant but does serve very well as a binder and it is easily removed from the specimens. Specimens pressed using 1.5% naphthalene as binder were sintered in vacuum at 1750°C and 1800°C for two hours. All specimens were pressed at 30,000 psi in a $\frac{3}{8}$ -in. diameter die. Diameter-to-length ratios varied from 0.75 to 1.0. Following are the linear shrinkages and bulk densities for these specimens.

Table VIII. Shrinkage and Densities of Sintered UC Specimens

	<u>Without Binder</u>		<u>1.5% Naphthalene as Binder</u>	
	<u>% Linear Shrinkage</u>	<u>Bulk Density gm/cc</u>	<u>% Linear Shrinkage</u>	<u>Bulk Density gm/cc</u>
1650°C	14.8	11.25	-	-
1750°C	16.4	12.02	15.9	12.00
1800°C	-	-	16.7	12.06
1850°C	13.3	10.34	-	-

The decrease in density for the specimens sintered at 1850°C cannot yet be explained. Metallographic and X-ray examinations have been made on specimens without binder sintered at 1650°C and 1750°C. The polished specimens revealed the presence of well crystallized UC, globular

pores, both at grain boundaries and intragranular locations, and a light colored, almost unetched phase which was later identified as U_2C_3 . Grain size ratios between the two temperatures was about 5:1. X-ray diffraction done on the polished specimens produced strong sharp reflections for UC and on the specimen sintered at 1650°C an estimated $<5\%$ U_2C_3 . The other specimen had only a trace of the sesquicarbide.

A few trial runs have been made using dextrose as the carbon source in the oxide-carbon reaction. This was done in an effort to find a suitable organic material with a constant carbon content. Though the system shows some promise, the high volatile-to-carbon ratio of the dextrose has caused much difficulty in the coking step.

b. Uranium-Thorium Sulfide - An investigation was made of the oxidation behavior of US powder when the oxygen pressure was varied from 760 mm to 0.5 mm in the DTA apparatus. Both dynamic and static conditions were used. Since US and UO_2 have identical cation lattices, it was hoped that at some reduced pressure a protective UO_2 coating might be formed on the US particles which would prevent further oxidation. This did not occur. Using dynamic flow of O_2 through the sample, the main effects of reducing the pressure were found to be: (1) the elimination of one of the three strong exothermic peaks at 190 mm O_2 and lower, (2) the lowering of the intensities of the peaks from 600-900 μv at 760 mm to 30-50 μv at 14 mm, and (3) the widening of the temperature range over which the oxidation occurred from $368-377^\circ\text{C}$ at 760 mm to $365-532^\circ\text{C}$ at 14 mm. Static conditions lowered the intensity of the peaks and considerably widened the temperature range of oxidation as compared to dynamic runs at the same pressures. At 0.5 mm static O_2 pressure, the first oxidation peak essentially disappeared, leaving only one pronounced oxidation peak occurring at 570°C .

Reassembly of the apparatus for making the low temperature sulfide mixture was completed and two batches of US of about 110 gm each were successfully produced. During the first run it was found that a very small quantity of the finely powdered product was drawn toward the vacuum system from the reaction flask. This was corrected by using baffles in the flask and it is now possible to make enriched sulfide for use in irradiation samples.

3. Fabrication Development

a. Fabrication of Zirconium Alloy Tubing - Defect-free zirconium alloy tubing is a desirable structural and fuel jacketing material for use in light water-cooled or heavy water-moderated thermal reactors. Thin wall tubing over 1 in. in diameter and sound small diameter tubing are not commercially available. For this reason an effort is being made to produce defect-free tubing in these size ranges.

Recent efforts have been directed toward improving the O.D. surface of "core drawn" 0.299 in. O.D. x 0.257 in. I.D. Zircaloy-2 tubing. Considerable surface improvement has been achieved in drawing 8 ft of tubing without a lubricant from ANL-extruded base tube. A red-grey coating is exposed on the as-extruded tube after removing the copper jacket by pickling in 50-50 nitric acid-H₂O. This coating, tentatively identified by X-ray diffraction as containing Zr₂Cu and Cu₃N apparently has unique lubricating properties and is being investigated further.

4. Nondestructive Testing

a. Ultrasonic Techniques - Room temperature measurements of Young's Modulus and Poisson's Ratio have been made on an aluminum specimen. The values calculated as a result of these experiments are $(6.92 \pm 0.04) \times 10^{11}$ and 0.3367 ± 0.0007 dynes/cm², respectively. The uncertainties quoted are due to scatter in measurements of sample parameters only. The results compare favorably with handbook values. Measurements at higher temperatures were also made from which elastic moduli will be calculated. It has been found that the optimum frequency at which each particular measurement should be made is a function of temperature. This seems to result from changes in interference patterns as the lengths and diameters of the specimens change due to thermal expansion.

b. Neutron Techniques - The photographic detection of neutron images is being investigated, with at least one long range objective being to apply neutron radiography as a useful inspection method for nondestructive testing. Because neutrons have little influence on normal photographic emulsions, it is necessary to use intermediate converter materials next to the film. These materials emit photographically detectable radiation when bombarded with neutrons, and thereby improve the photographic speed of the detection process.

Further image sharpness results obtained by averaging at least four different scans of an imaged abrupt absorption change, have shown that best image sharpness qualities are found using gold in thicknesses of 0.005 in. or less, or indium in thicknesses of 0.010 in. or less for transfer neutron radiographs. These values are reasonably consistent with considerations involving the range of the beta emission from these materials. They are also in agreement with our preliminary findings.

One result different from our preliminary findings is that some difference in image sharpness can now be detected between films exposed on the neutron source side of the transfer foil (front films) and those exposed on the opposite side. Intuitively, one would expect better results from front films because of the greater activity for the lower energy components of the emitted radiation. This improved image sharpness for front films used with gold and indium transfers can now be observed.

Attempts to verify the image sharpness results being obtained with our equipment have been made by repeating some measurements on two different Leeds and Northrup recording densitometers. Results of these comparisons were encouraging for the most part although some discrepancies were found. It is believed that some of the inconsistencies observed in the three comparisons were caused by the variations in density between the different radiographs studied.

c. Radiographic Techniques - The aluminum clad aluminum-uranium alloy fuel tubes currently being used in CP-5 have approximately 15 mil thick cladding on the O.D. and I. D. An attempt is being made to measure the cladding thickness by a β backscattering technique. When a beam of β particles impinge on a material, some are transmitted, some are absorbed and others are elastically scattered back out of the material. The intensity of the backscattered beam depends on the thickness and atomic number of the scattering material. When the thickness of the material reaches $\frac{1}{5}$ of the range of the β particles the backscattered beam has maximum intensity. This thickness is called the saturation thickness. The β source being employed is $\text{Sr}^{90}\text{-Y}^{90}$ with a maximum energy of 2.16 Mev. The β range for aluminum is approximately 146 mils. Therefore the saturation thickness is approximately 29 mils or about twice the cladding thickness. Since the cladding thickness is less than the saturation thickness the intensity of the backscattered beam is dependent on the cladding thickness, and because the effective atomic number of the core is higher than that for the cladding, the backscattered intensity is inversely proportional to the cladding thickness.

Using an anthracene crystal as a detector an increase in cladding thickness simulated by aluminum foil (0.7 mil) can be detected at a scanning speed of 40 rpm. At slower scanning speed greater sensitivity is possible. The diameter of the area inspected is $\frac{1}{4}$ in.

d. Eddy-Current Techniques - The pulsed-field reflection system is being used to check the bond quality of pins of plutonium-fissium alloy enclosed in jackets made from a variety of materials and bonded with sodium. The resolution of this test system is good enough to provide a very sensitive test for voids in the bond layer. The bond quality of nine jacketed pins has been checked prior to irradiation of the pins in CP-5. Six of the pins were well bonded. Nonbond areas in the other three ranged from 10% to 30% of the total pin area

C. Reactor Materials Development

1. Radiation Damage in Steel

a. EBWR SA-212B Irradiation - The contents of the third capsule of foils (described on page 49 of ANL-6485, Progress Report for December, 1961) were examined after 168 hours at 600°F. As before, the uranium and

thorium foils were oxidized and the metal wire foils showed alloying with cadmium. Spectrochemical analyses of the surfaces of the metal wires and of the aluminum jackets confirmed the presence of cadmium in high concentrations. Preliminary analyses of the yellow cadmium products found on the various foil wires show the absence of sulfur.

b. Magnetic and Dynamic Properties - Measurements of the damping factor on the SA-212B modified magnet bars with the FM-500 Elastomat (page 49, ANL-6485) revealed that geometry and environment influenced the measurements substantially. The optimum spacing of the magnet bar from the transducers is between 0.040 in. and 0.062 in. At closer spacings damping of the transducers by the magnetic field is appreciable. The number of cycles for a $\frac{1}{3}$ amplitude decay is reduced by 25%. At greater spacings detection sensitivity falls off rapidly. Within the range 0.040 in. to 0.062 in. spacing, skewing of the samples on the supporting wires affects sensitivity roughly $\pm 8\%$.

Measurements were most sensitive when the magnetic field orientations were horizontal, the driver transducer pole shoes vertical and the detector pole shoes horizontal. Vertical magnetic field orientations permitted measurements at spacings closer than 0.040 in. without pulling the samples into the magnetic pole pieces. At these closer spacings, however, the magnetic field damps oscillation of the bar.

Both the fundamental and third harmonic frequencies of the SA-212B steel have a negative frequency-temperature dependence of about 0.5 cps/°F over the temperature range of 80°F to 110°F. The slope of third harmonic frequency characteristic is greater than that of the fundamental. Longitudinal mispositioning of the bars from the prepared nodal grooves had no effect on fundamental and third harmonic frequencies. Amplitude decay variations as great as 7% were found for bars suspended slightly inside or outside the prepared grooves. Identification steel stamping of bars did not affect frequencies or the damping factor of the fundamental frequency.

Frequency standards prepared from a constant modulus tuning fork steel obtained from time-frequency showed a very slight positive frequency-temperature coefficient over the same temperature range. The slopes of the fundamental and third harmonic frequency for this modified Elinvar steel were parallel.

An improved suspension stand including right and left-hand, three-stage micro-positioners is being designed for future use in the caves to solve the problem of the accurate positioning of transducers and the irradiated magnet bars.

D. Reactor Components Development

1. Development of Viewing Systems - Basic Glass Studies

Studies carried out during the past two years have shown that the intensity of the optical absorption induced in glass by exposure to gamma radiation is controlled principally by three effects: (1) changes in glass structure, (2) the addition of two multivalent ions which result in a photochemical reaction, e.g., Bi^{3+} , As^{3+} , and (3) the addition of small concentrations (about 0.01 to 1.0 mole %) of some multivalent elements, e.g., Ce, Fe, Ti, Tl, etc. Recent experimental studies have shown that the induced absorption is influenced by a number of factors related to the glass structure. These include the "polarizing power" and the "coordination number" of the network-forming and modifying ions, the concentration of nonbridging oxygen ions, and the inclusion of multivalent network or modifying ions. The "polarizing power" of an ion is proportional, to a first approximation, to its field strength, Z/r^2 . The "coordination number" refers to the average number of oxygen atoms immediately surrounding the ion.

In order to study the effect of network-forming oxides on the induced absorption, there were melted and irradiated a series of silicate glasses, borate glasses, and phosphate glasses having identical molar compositions of the form $(2 \text{ SiO}_2 \cdot 0.35 \text{ Na}_2\text{O} \cdot 0.3 \text{ CaO})$, where one B_2O_3 , or one P_2O_5 unit was substituted for the 2 SiO_2 units. The positions of the induced absorption bands generally agreed with previous experimental data. In this series the silicate glasses had the highest induced absorption, except for the green to red portion of the spectrum. It was expected that the phosphate glass would have the highest absorption because it has the highest concentration of nonbridging oxygens.

The effect of varying the nonbridging oxygen ion concentration was studied in a series of soda-lime-silicate glasses of the above base composition by varying the soda content. In contrast to previous results in phosphate glasses, it was found that the induced absorption decreased with increasing nonbridging oxygen ion concentration. It is believed that the induced absorption results from an equilibrium between the formation and annihilation of color centers and that this equilibrium can be shifted in opposite directions by the glass structure or the type of color center involved.

E. Heat Engineering and Fluid Flow

1. Condensation of Metal Vapor

A theoretical and experimental study of condensation is being conducted in an attempt to explain the discrepancies between existing condensation theories and published metal-vapor condensation data. Published experimental heat transfer coefficients are lower than theoretical predictions by factors as large as 10 and 20.

Two different condensation theories are currently being studied in conjunction with this program: (1) laminar film condensation which considers the coupled mechanisms of mass transfer through the vapor-phase boundary layer and conduction heat transfer through the thin condensate film; and (2) filmwise condensation in which the transition to turbulent flow is included. The resistance to heat transfer in this model is assumed to be in the condensate film only. Integral momentum and energy balances are employed which require computer aid for their solution.

Design of a rotating, horizontal disc condenser and design of a horizontal plate condenser, both for liquid metal (mercury), have been completed and fabrication will be started as soon as possible.

The fabrication of major components for a horizontal plate condenser to measure transient condensation of non-metal vapors is half completed, while the fabrication of a horizontal plate condenser to measure steady-state or transient condensation of non-metal vapors has been nearly completed and assembly is underway. The non-metal system provides simple experimental methods for studying basic phenomena.

2. Hydrodynamic Instability

The time-dependent behavior of a boiling loop is being examined by determining the frequency response of steam volume fraction to flow and power oscillations. Instrumentation is being designed to make these measurements. A model of a sinusoidal-output gear drive was constructed and tested. Favorable characteristics of the drive were low backlash and good power transmission. Disadvantages were that only fixed strokes are available for valve actuation.

The analog computer model describing a forced or natural circulation system was checked against a series of pressures for one geometry on one test loop. For each pressure, it predicted the proper steady-state void and flow rate as a function of power. The power was sinusoidally oscillated at different frequencies. The resultant void and flow oscillations were similar to experimental measurements including a rather non-linear flow response.

The study of varying the velocity ratio correlation with the power level at which the analog oscillates was partially completed. The model is being compared with experimental results on two test loops of different geometries.

F. Chemical Separations

1. Fluidization and Fluoride Volatility Separations Processes

a. Fluoride Separations - The removal of plutonium hexafluoride from air streams by combined vapor hydrolysis and filtration is being studied in connection with fluoride volatility processes for recovery of uranium and plutonium from nuclear fuels. An electrostatic precipitator has been constructed to collect the particulate matter resulting from the vapor hydrolysis so that particle size and distribution measurements can be made. Examination of electron micrographs prepared from vapor-hydrolyzed uranium hexafluoride samples collected by the electrostatic precipitator, indicate a particle size range of approximately 0.01 micron to 0.2 micron for individual particles. The fiber sizes of AEC filter media have been obtained by electron microscopic examination. The median value of measurements of the diameters of 181 fibers was 0.31 micron with one percent smaller than 0.029 micron and one percent greater than 3.3 microns. The average fiber diameter was calculated as 1.5 microns.

Further work has been done to obtain information about the gamma radiation decomposition of plutonium hexafluoride. A radiation experiment was performed using 1.11 millimoles of plutonium hexafluoride, about four times the amount used in previous experiments. After subjecting the sample to a gamma dose of 1.48×10^8 rads, analysis of the products indicated the formation of 0.293 millimole of fluorine and 0.331 millimole of plutonium tetrafluoride, while 0.819 millimole of plutonium hexafluoride remained. Compared with previous results, the yield of fluorine was in better agreement with the yield of plutonium tetrafluoride. This may be attributed to the larger sample.

Attempts have been made to prepare mixed chlorofluorides of hexavalent uranium by the reaction of sulfur tetrafluoride with uranyl chloride. This would be analogous to the preparation of uranium hexafluoride from uranyl fluoride and sulfur tetrafluoride. The experiments performed with uranyl chloride and sulfur tetrafluoride have so far resulted in the formation of uranium tetrafluoride.

The tetroxides of ruthenium and osmium were reacted in the gas phase with sulfur tetrafluoride at 110°C. Qualitative infrared analysis of the gaseous products indicates that sulfuryl fluoride (SO_2F_2) was the major component, indicating that oxidation of the sulfur compound had occurred. It is believed, but it has not yet been established, that ruthenium tetrafluoride and osmium tetrafluoride are formed in the reactions.

b. Direct Fluorination of Uranium Dioxide Fuel - A direct fluorination volatility process is being applied to oxide fuels. In a current series of runs the fluorination of larger charges (i.e., deeper beds) of uranium dioxide

are being studied in order to optimize processing conditions. In two runs 18 inch-deep batches of uranium dioxide pellets have been fluorinated with promising results. Uranium hexafluoride production rates and heat transfer have been satisfactory during major portions of the run. Control of the concentration of solid fines consisting of uranium intermediates (principally uranyl fluoride) appears to offer means of achieving both higher fluorination rates and fluorine efficiency as well as the prevention of caking. The fines production is in turn controlled by the concentrations of fluorine and oxygen.

Further data available on pellet oxidation runs made previously show that an approximately 30 percent oxygen - 70 percent nitrogen mixture completely reacted a 3-inch deep bed of $\frac{1}{2}$ -inch UO_2 pellets in 4.0 hr at 500°C and 1.3 hr at 600°C . This determination was made on the basis of X-ray diffraction analysis of the oxide residue, which revealed only the presence of $\alpha - \text{U}_3\text{O}_8$.

The solids penetration of the sintered Monel filters on the reactor used for fluorinations and oxidation was measured in four pellet oxidation experiments. These preliminary results showed penetrations of 1 to 100 micrograms per cu ft of gas passed through the filter under run conditions. Previous determination of the particle size of oxide fines indicated a mean size of about 20 microns.

A two-phase air-water fluid cooling system is being evaluated for application to the direct fluorination reactor. Satisfactory control of reactor temperature in the vicinity of 500°C has been demonstrated for various heat loads (up to 10,000 Btu/hr) covering the range encountered in process application. The system consists of a coolant tubing coil bonded to the exterior wall of the reactor with water injection at intervals to give zone control. The heat pickup is chiefly by vaporization of the dispersed water. This method permits a low volumetric flow of coolant to handle a wide range of heat generation rates and also allows for control of temperature for different rates of heat generation at different elevations inside the reactor.

c. Separation of Uranium from Zirconium Alloy Fuels - Sketches of several process vessels for the proposed pilot-scale facility for recovering uranium from zirconium-based fuels have been prepared and submitted for detailed design. A full-scale Lucite mockup of the fluid-bed, up-flow filter reactor (six-inch lower section, twelve-inch upper section) has been fabricated for use in visual studies of operations.

2. Chemical-Metallurgical Process Studies

a. Liquid Metal Solvent Studies - The codistribution coefficients of uranium, plutonium, strontium, and cesium between liquid lead and zinc were measured. The results are given in Table IX.

Table IX. Codistribution Coefficients of Uranium, Plutonium, Strontium, and Cesium between Liquid Lead and Zinc

Solute	<u>Codistribution Coefficient</u>		
	$\left(K = \frac{\text{amount of solute in upper phase}}{\text{amount of solute in lower phase}} \right)$		
	701°C	733°C	761°C
Uranium	68	30	11
Plutonium	18	10	5
Strontium	0.17	0.35	0.20
Cesium	1.3	3	1.3

The coefficients of uranium and plutonium are not significantly different from those measured earlier at approximately the same temperatures (see Progress Report for October, 1961, ANL-6454, page 48).

The solubility of beryllium in liquid zinc has been established with greater accuracy through additional analytical work. The values range from 0.0064 weight percent at 429°C to 0.054 weight percent at 611°C.

The emf data reported earlier (see Progress Report for August, 1961, ANL-6409, page 57) for the uranium-indium cell have been combined with the solubility data for uranium in liquid indium (Progress Report for December, 1961, ANL-6485, page 55) to compute the activity coefficients of uranium in saturated indium solutions. Over the temperature range of 450°C to 675°C, the activity coefficient γ_U (solid uranium reference state) may be calculated by means of the following equation:

$$RT \ln \gamma_U = -731 - 0.710 T + 3.88 \times 10^{-3} T^2$$

Study of the cerium-cadmium system by means of the recording effusion balance has shown the existence of the following phases: CeCd_{11} , $\text{CeCd}_{5.7-6.4}$, Ce_2Cd_9 , and CeCd_2 . The existence of a solid solution range located at about CeCd_3 is also indicated, but further work is required to establish the limits of the range.

3. General Chemistry and Chemical Engineering

a. Conversion of Uranium Hexafluoride to Uranium Dioxide - Two Step Fluid-Bed Process - Additional exploratory studies of the reaction of steam with uranium hexafluoride indicate that higher temperatures (500°C rather than 200°C) reduce the problem of the deposition of solids on reactor

surfaces. Both the reactor walls and the one-inch line connecting the reactor and filter vessels were free of powder. Preliminary information indicates that some conversion to U_2O_8 was achieved. At $200^\circ C$, only uranyl fluoride is produced. The powder produced at $500^\circ C$ also has a lower bulk density, 2.2 g/cc as compared to 3.3 g/cc for uranyl fluoride, and is definitely less hygroscopic than the material produced at $200^\circ C$. An adverse effect of operating at high temperatures is that excessive fines are produced.

b. Preparation of Uranium Monocarbide - Work has continued on the preparation of uranium monocarbide by the addition of carbon to uranium dissolved in magnesium-zinc solutions. Considerable difficulty has been encountered in the past in achieving adequate separations of the unreacted excess carbon from the precipitated carbide product. In recent experiments additives have improved the yield of uranium monocarbide. With these additives it appears that stoichiometric amounts of uranium and carbon will react completely to form uranium monocarbide. The ingot resulting from the experiment was clean and no carbon was visible in the crucible nor could it be seen in the metal ingot. The product appeared to be finely divided uranium monocarbide; however, product analysis is not yet available.

IV. ADVANCED SYSTEMS RESEARCH AND DEVELOPMENT

A. Direct Conversion Studies

The efficiency of the thermionic energy converter has been further analyzed using the LGP-30 computer and following the method of Schock.¹ In Ref. 1 plots of efficiency versus cathode temperature appear for an idealized plasma diode, where cathode work function, or output voltage, and cathode lead wire voltage drop are optimized, and the anode work function and temperature and the effective thermal emissivity are held constant. The parameter, A , from Richardson's equation is given the theoretical value of 120. However, experimental values of A are uncertain.

To allow greater flexibility in optimization of real electron emitters in the actual plasma diode, the same calculation has been done with A equal to 1.2 and 12 as well as 120 amperes/(cm²)(deg²). Figure 5 shows the maximum efficiency, $\eta_{\max}$, plotted against the cathode temperature, T_c , °K for the three values of A , where the effective thermal emissivity is 0.1, the anode work function is 1.6 volts and the anode temperature is 600°K.

Figure 5. Maximum Efficiency, $\eta_{\max}$, versus Cathode Temperature, T_c , for Three Values of A , also Showing the Locus of Points of Equal ϕ_c^*

¹ Alfred Schock, "Optimization of Emission Limited Thermionic Generators," J. Applied Physics 32, 1564 (August 1961).

Also indicated is the optimized or critical cathode work function, ϕ_C^* volts, which is associated with the given maximum efficiency. It can be seen that maximum efficiency is fairly insensitive to reduction in A . Primarily, this is because ϕ_C^* assumes a new reduced value so that the net change in efficiency is small. Output power density, watts/cm², for the optimized converter, shown in Figure 6, is somewhat more sensitive to A .

Figure 6
Power Density, Watts/cm², for the Optimized
Thermionic Converter for Three Values of A

Direct conversion experiments with the potassium plasma diode cell have been continued (see ANL-6485, Monthly Progress Report for December 1961). The high frequency plasma oscillations were investigated. The frequency and amplitude of the oscillations were measured as a function of the positive and negative voltages applied between the emitter (tantalum) and collector (silver oxide), the emitter temperature, and the potassium vapor pressure. Pictures from the oscilloscope recorded the r-f wave shapes. The r-f and d-c components in the output were separated and measured. The cell outputs were then studied with respect to the emitter temperature and potassium vapor pressure. The experiments will be repeated using the cesium plasma diode cell.

V. NUCLEAR SAFETY

A. Thermal Reactor Safety Studies

1. Fuel-Coolant Chemical Reactions

A series of condenser discharge runs with aluminum wires is being made in water at 200°C. Preliminary results indicate that vigorous reaction begins at metal temperatures of about 1500°C and up to 85 percent reaction occurs at initial metal temperatures of the order of 2500°C.

Further studies of the reaction of aluminum-uranium alloys with steam have been made by the pressure-pulse method. Studies have been made with alloys containing 5, 20, and 40 percent uranium at 1200°C. Increasing uranium content increases the reaction rate slightly.

Preliminary runs have been made by a levitation melting method. Aluminum pellets would not ignite in 25 mm water vapor. Samples vaporized smoothly and the vapor reacted quickly to yield Al_2O_3 and H_2 . Installation of a special steam pressure regulator for runs in one atmosphere flowing steam is in progress.

Studies of the kinetics of metal-water reactions under reactor incident conditions are being made in the TREAT reactor. Comparisons were made of three independent methods of determining the fissions (burnup) in fuel specimens irradiated in TREAT. Values of 3.0, 3.4, and 2.8 fissions/(watt second)($\mu\text{g U}^{235}$) were obtained by a fission counter, uranium monitor foils, and by direct radiochemical analysis of the irradiated fuel pin, respectively. This comparison gives a measure of the internal consistency of the energy absorbed by the fuel sample in the CEN autoclave to the total energy of the reactor burst. Post-irradiation examination of the four experiments on graphite fuel, previously conducted in TREAT, indicated that each of the cylindrical fuel specimens had undergone destruction. Sieve-screen analyses of the fragments and particles showed mean particle sizes of 32, 23, and 22 mils for energy inputs of 3.9, 6.5, and 8.9 arbitrary units, respectively. The original diameter of the graphite fuel cylinder was 0.5 in., or 500 mils; therefore, the energetic transients caused an increase in the specific surface area of the fuel by a factor of about 25. Also, there is a trend toward smaller particles as the integrated power of the reactor pulse increases.

2. Metal Oxidation and Ignition Studies

The calculation of burning temperatures of metals in air was continued. Since knowledge of the emissivity of uranium oxide is necessary for such calculations, experiments were conducted to obtain this information. Total and spectral emissivity values were found for surfaces of polished uranium and uranium with various degrees of oxidation. Values of total emissivity of the order of 0.36 were obtained for polished uranium cubes.

The total emissivity of oxidized uranium varied between 0.58 and 0.72. The spectral emissivity at 0.65μ ranged as high as 0.8 for oxidized surfaces.

Isothermal oxidations of pure plutonium have been performed in the alpha, beta, and gamma phase of plutonium. Preliminary data indicate a protective type oxidation in the alpha phase and linear oxidation in the beta and gamma phases. In the beta phase, oxidation was observed to take place in two linear stages. A second stage faster than the first stage was observed.

The effect of the presence of halogenated hydrocarbons on the burning of uranium and zirconium foils in air is being investigated. Flammability of CH_3CHF_2 in air is completely inhibited if mixtures are used in which the $\text{CH}_3\text{CHF}_2:\text{CH}_3\text{Br}$ ratio is 3.9 or smaller. Such mixtures inhibit uranium and zirconium combustion when two percent of the mixture is added to air. However, a decrease of about 100°C is produced in the ignition temperature of the uranium foil.

B. Fast Reactor Safety Studies

1. Experimental Meltdown Program

In-pile meltdown experiments are being performed in the TREAT reactor in order to study fast reactor fuel failure, the resulting molten fuel movement, and relevant mechanisms producing failure and meltdown product movement.

a. EBR-II Element Experiments in the Absence of Sodium - Tests are being run on the behavior of dry EBR-II elements in order to observe sample failure and fuel expulsion without the added complication of sodium coolant.

The four samples of Series XXVI were received and examined. This was the first series of photographic meltdown experiments in which samples were subjected to a shaped axial power profile. The power distribution in the sample was perturbed axially from the usual essentially uniform shape by tantalum neutron absorbers in the top and bottom of the capsules. Radiochemical determinations from the first unfailed samples indicated a maximum-to-minimum power ratio of about 1.4. Post-mortem results from the samples of Series XXVI are given in Table X. Temperatures were measured by fast response chromel-alumel thermocouples spotwelded to the stainless steel cladding.

Thirty-five natural uranium-5 w/o fissium elements were clad and bonded in preparation for the first series of experiments on sample failure and meltdown product movement resulting from exposure of clustered elements. Each test is to be performed using seven natural enrichment elements arranged on an EBR-II pitch with one central element surrounded by a ring of six.

Table X. Results of Meltdown Experiments, Series XXVI

Sample No.	Integrated Power (Mws)	Maximum Cladding Temperature Recorded (°C)	Condition
1	32	765	Slight warpage, but no cladding penetration. Fuel surface eroded and containing areas of uranium-iron eutectic.
2	52	1005	Warpage, but no cladding penetration. Cladding has not been removed.
3	85	1250*	Central section of cladding destroyed. All fuel ejected from cladding.
4	140	1180*	Central section of cladding destroyed. All fuel ejected from cladding.

* Maximum at time thermocouples failed.

b. EBR-II Elements in Stagnant Sodium - Experimentation on samples contained in stagnant sodium capsules is being done to study possible effects on failure and post-failure fuel movement caused by the liquid metal annulus around the element. The last two samples from Series XXVII (see previous Monthly Progress Report, ANL-6485) using an axially-shaped element power were run in TREAT and arrived at Argonne for post-mortem examination at the close of the report period.

c. Dry Opaque Capsule Development for Irradiated Samples - Remote assembly techniques are being developed for encapsulating a previously irradiated fast reactor fuel sample in a dry opaque container. Seven prototype capsules are being built in the shops, and will be used to test assembly procedures using the mockup of Building 301 cave facilities.

The handling coffin for the loaded capsule has not met specifications, and has been returned to the manufacturer for correction. Redelivery of the coffin is expected early in February.

d. Small Sodium Loop - A small sodium loop designed to fit in the 10.2-cm sq by 240 cm long volume of a TREAT fuel element is being developed. A new pump, designed for the small sodium loop, was operated successfully during this period. The new pump develops about 13% higher flow for a given set of input conditions as compared to the original unit. The present short-term maximum flowrate is now 500 cm/sec (16.4 ft/sec) at 500°C.

The design of the instrumentation and control circuitry for the loop has been completed and is being reviewed.

Preparations are complete for laboratory test of one of the loops with the exception of the installation of the pressure transducers. The previously reported inability of the transducers to sense pressure after sodium was introduced into the loop was found to have resulted from mechanical failure caused by excessive pressure or impact applied to the transducer diaphragm during either the loop assembly or filling. Attempts are now underway to correct this difficulty.

e. Large Sodium Loop - A large sodium loop is being developed as a semipermanent installation in TREAT to permit meltdown experiments on failure and fuel movement using clusters of sample elements under full-flow EBR-II conditions.

The final coffin design has been completed. The test section design has been improved with respect to the location of pressure transducers and connection flanges.

Purchase and delivery orders have been issued on parts for the piping, fittings, tanks, joints, stairwell, and equipment monorail systems.

2. TREAT - Operations

The five samples for CEN Fuel-Coolant Chemical Reaction Test Series XVIII were irradiated and returned to Argonne, Illinois, for examination. This series consisted of two graphite-UC samples in a helium environment, two graphite-UC samples in a water environment, and one graphite-UC sample in a vacuum.

The first sample of the GE-APED Test Series on EFCR fuel was subjected to four transient irradiations of successively higher energy input to obtain a correlation between reactor energy release and sample temperature. The final transient on this sample was sized to produce a maximum sample fuel temperature of approximately 4500°F.

Installation of equipment for the BORAX V exponential experiment in TREAT was completed and flux measurements were started in the exponential assembly. This experiment is designed to give information on the cadmium-ratio technique for measuring voids in a boiling water reactor. The exponential assembly is mounted on a track so that it can be withdrawn from the reactor shield during transient experiment. Therefore, this experimental program will not interfere with the transient test program.

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