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PACIFIC NORTHWEST LABORATORY MONTHLY ACTIVITIES REPORT FOR NOVEMBER 1966

DIVISION OF REACTOR DEVELOPMENT AND TECHNOLOGY PROGRAMS

DECEMBER, 1966

BATTELLE-NORTHWEST

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PACIFIC NORTHWEST LABORATORY

RICHLAND, WASHINGTON

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UNITED STATES ATOMIC ENERGY COMMISSION UNDER CONTRACT AT(45-1)-1830

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BNWL-359

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Nuclear Technology
(Special Distribution)

PACIFIC NORTHWEST LABORATORY

MONTHLY ACTIVITIES REPORT

FOR NOVEMBER 1966

AEC DIVISION OF

REACTOR DEVELOPMENT AND TECHNOLOGY PROGRAMS

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December 1966

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SUMMARY

CIVILIAN POWER REACTORS

Nuclear Systems Concepts Analysis

A simulation of the future nuclear economy using a two-reactor description and dividing the next 70 years into 35 two-year periods was presented at the Systems Analysis Task Force meeting in Washington, D.C., early in the month. This simulation showed that the tools for performing the analysis were available and that the method was feasible. The calculations are now being repeated with a five-reactor model. The reactors are (1) the Westinghouse 1000 MW fast reactor, (2) a PWR operating on uranium, (3) a PWR operating on plutonium recycle for four years, (4) a PWR operating on plutonium recycle for eight years, and (5) a PWR operating on plutonium recycle for 12 years. The Phase II effort, which will involve a many-reactor problem, requires some modification of the DAEDALUS code, the linear program matrix builder. The first computer run of Phase II effort will be done in mid-December, using most of the data supplied by other task forces for various reactors. In early January, the entire Phase II problem will be run for presentation at a mid-January meeting of the Systems Analysis Task Force.

Preliminary calculations of the Westinghouse reactor indicate a plutonium value of \$19 compared to U-235 from the cascade. With plutonium at \$10/g and interest at 10%, fuel cycle costs were 1.2 mils versus 1.4 for the Allis-Chalmers reactor.

Code maintenance and development has continued upon both the two and four energy-group ALTHAEA's and their follow on data-handling and economics routines such as FAST, QUICK, and PLOTTER. Modifications also continued in FCC, which inputs data to four-group ALTHAEA and to PACTOLUS, which generates input data for the 1107 linear programming code.

Conceptual Reactor Design Studies

All flow and temperature data available at BNW have been tabulated. The tabulated data cover Pacific Northwest and Southwest watersheds but are incomplete for all areas. Correlation of existing data is proceeding pending receipt of additional data.

Contract negotiations with R. W. Beck and Company, on furnishing engineering cost estimates for heat dissipation systems, are essentially complete. They are expected to start work about December 1.

The MIMIC Program to predict cycle efficiency is nearly completed. Examination of the heat transfer behavior, including the effect of surface roughening, is proceeding.

USAEC-AECL Cooperative Program

The analyses of the experimental data obtained with a test section simulating a flow channel formed by fuel rods in a square pitch array located adjacent to a channel formed by rods on a triangular pitch array were completed. The results show the following effects:

- 1) At a simulated rod spacing of 0.020 inch, the turbulent mixing in two-phase flow was approximately the same as for single-phase flow.
- 2) At a rod spacing of 0.080 inch, turbulent mixing during two-phase flow was somewhat higher than for single-phase flow.
- 3) The amount of turbulent mixing between flow channels does not obey a linear relationship with rod spacing.

Microhardness data have been obtained from Zr-Nb corrosion coupons in the irradiated and unirradiated condition.

The analog computer study of hydraulic and nuclear stability of boiling water cooled reactor systems has been completed. A rough draft copy of the results was written and distributed to members of the TAC Subcommittee on Control and Stability.

An in-reactor test on Zr - 2.5% Nb is continuing at 350 C and 55,000 psi stress.

Specimens of six zirconium alloys were exposed four cycles in the G-7 loop of the Engineering Test Reactor. Flux-induced oxidation was small or absent on etched specimens. Specimens of VALLOY, Zircaloy-2, and Zircaloy-4 prefilmed in steam at 400 C, 1500 psi, underwent marked accelerated oxidation.

APPLIED AND REACTOR PHYSICS

Plutonium Criticality Studies

Criticality experiments were continued for determining critical thicknesses of water reflected "infinite" slabs of plutonium nitrate solutions.

The 12 experiments performed during the month with bare and water reflected assemblies were principally to study the effect of the stainless steel vessel wall on criticality, and to evaluate reflector savings for determination of critical bucklings.

High Temperature Reactor Physics Studies

The gas control valves, sample piping, and thermocouples are being installed.

Installation of the graphite bus bars is 80% complete.

Assembly of the oscillators is 64% complete.

Final assembly of the control rods is scheduled for December.

Assembly of the test core has been delayed by about two weeks.

Planning for the U-233 - Th experiments and evaluation of the unpoisoned k_{∞} measurement technique is continuing.

A test of the control rod worth has been completed.

Assembly of the driver fuel elements was started.

Phoenix Fuel Reactor Program

The experimental program using Phoenix fuel in the Critical Approach Facility has been completed.

All hardware associated with the PRCF-Phoenix experiments is presently being fabricated with delivery promised by December 15, 1966.

Studies of postulated reactor excursions on the PRCF-Phoenix core have been completed.

Possible process variables were identified which should minimize the Pu segregation in the plates. Designs of the fuel element and shim control rod were revised and released for fabrication.

A variety of possible calculational uncertainties in the CAF analysis have been explored. Calculated changes in k_{eff} from the studies made thus far are still less than that needed to bring the total calculation into agreement with the experiments.

The ZODIAC 2 burnup code has been revised to use the HRG slowing down program in place of GAM.

The atom fractions of various materials for a core using burnable poisons similar to the B&W CNSG II, have been determined along with the core volume and dimensions. Physics calculations on this core will form a basis for comparison of Phoenix fueled CNSG cores to be investigated in the near future.

REACTOR FUELS AND MATERIALS

Fast Fuels Oxides and Nitrides

A proposed irradiation testing program in support of mixed nitride fast reactor fuels is being developed.

A literature and user survey of suitable instruments, methods, and approximate costs for microprobe analysis of irradiated materials was initiated.

Installation is nearing completion on an inert-atmosphere hood which will provide the capability for performing operations in a pure (oxygen- and moisture-free) atmosphere. An inert-gas fusion apparatus for analysis of oxygen and nitrogen in refractory compounds was installed in the hood.

The hood atmosphere, of either argon or helium, will be maintained at a purity level of less than 10 ppm oxygen and moisture.

Basic Swelling Studies

Three capsules continue to operate successfully at control temperatures of 450 C (1000 psi), 450 C ($\sim$ 30 psi), and 525 C ($\sim$ 30 psi) toward burnups of 0.75 at.% BU, 0.3 at.% BU, and 0.4 at.% BU, respectively. Two capsules are being constructed to operate at 450 C (5000 psi) and 700 C (1000 psi), respectively.

High purity - high carbon uranium specimens irradiated in the beta phase to 0.2 at.% BU contain larger fission gas pores than do U - 400 Fe, 640 Al, 85 Si, 500 C specimens irradiated under the same conditions.

Nondestructive Testing

New solid state circuitry has been fabricated and placed in operation to provide six channel display capacity for use with the eddy current multiparameter tubing test device and tubing cross section display. This system presents, on an oscilloscope, a graphical representation of both the relative magnitude and position of defects.

A method for plotting vector (phasor) test signals as a function of sensing coil position was developed and demonstrated during this reporting period. This technique was developed to aid in the display, and detailed study, of electromagnetic fields near the sensing coil. Several two-dimensional scans of various flaws have been made with excellent results.

Nuclear Ceramics

A high-temperature, high-pressure filament furnace for use in melting-dissociation studies on UN-PuN compositions was calibrated for temperature measurement using an optical pyrometer.

X-ray line broadening analyses of PuO_2 impacted at a temperature of 1200 C and pressure greater than 20,000 atmospheres indicated minimal residual strain. Annealing of the impacted samples in air at temperatures from 600 to 1650 C showed that the amount of residual strain decreased after one hour at 1200 C and was completely absent after one hour at 1650 C.

The occurrence of recrystallization in annealed samples of impacted UO_2 (-100 +150 mesh; 600, 1000, and 1200 C; and 340,000 psi) was verified. A one-hour vacuum anneal at 1200 C produced no observable recrystallization, but a one-hour anneal at 1400 C produced significant recrystallization of the large unfractured grains. Recrystallization was most evident in the material impacted at 1200 C, which had apparently undergone more plastic deformation.

Effects of natural convection on the thermal profile in horizontally-irradiated, molten-center UO_2 fuel cores were deduced. Upward displacements of the molten region were attributed to heat transfer to the top of the molten zone by natural convection. Quantities analogous to convection coefficients were computed and correlated with diameters of the molten zone.

Oxygen vapor pressure measurements over hyperstoichiometric plutonium-uranium mixed oxides (20% PuO_2) at 1400 C with the chemical potential of oxygen in the gas phase varying between -14.2 and -22.5 kcal resulted in oxygen pressures sufficiently large to cause loss of a metal-bearing species, presumably $\text{UO}_3(\text{g})$.

Mixed UO_2 - 20% PuO_2 was retained stoichiometric at temperatures to 1400 C by heating in one atmosphere of He - 60 mm H_2 passed over 0°C H_2O . Utilizing these conditions, other property measurements can be readily performed on completely stoichiometric mixed oxides.

Eight UO_2 - PuO_2 samples extending from 12 to 100 mole% PuO_2 were melted in an Ar - 8% H_2 atmosphere and quenched to room temperature. X-ray diffraction of these samples indicated an increased lattice constant relative to the pre-melted values, indicating hypostoichiometric solutions.

Uranium dioxides, with O/U ratios between 1.90 and 2.15, were chemically thinned and examined by transmission electron microscopy. Apparent dislocation densities were higher in pneumatically impacted material than in sintered materials.

An evaporator for depositing compositions corresponding to isothermal sections of ternary phase diagrams was designed for use in studying phase equilibria in the Pu-O-N system.

The remainder of the parts necessary to set up the 200 l/sec ion pumping system for high vacuum pneumatic impaction arrived and were assembled.

UO_2 and UO_2 - ThO_2 specimens were prepared, characterized, and distributed among domestic sites for use in basic studies. In addition, the Japan - US Research Newsletter No. 5 was processed and exchanged for the information of the cooperating agencies.

Nuclear Graphite

The high temperature capsule GEH-13-12 was damaged during pre-startup operations at the ETR and had to be discharged. Samples retrieved from the capsule will be incorporated in a reconstructed capsule scheduled for charging December 19.

The first graphite samples irradiated in the EBR-II appeared in excellent condition after successful extraction from the pin assembly.

The GETR is now operating at 50 MW, and the H-3-12 long-term capsule is functioning satisfactorily. The H-3-24 proof-test capsule was charged in October and is currently running within 100 C of design temperatures at all positions.

A sample series graphitized from 2400 to 3000 C shows decreasing contraction rates with higher graphitization temperature after irradiation to 5.5×10^{20} nvt, $E > 0.18$ MeV, in Hanford reactors at about 650 C.

Five percent aluminum, 5% Fe_2O_3 , or 5% Ni_2O_3 additive in the mix of standard needle-coke graphite appears to reduce the contraction rate after 9.3×10^{20} nvt, $E > 0.18$ MeV, at 650 C.

The H-3-13 graphite irradiation capsule is now operating in the GETR at a reactor power of 50 MW. The samples are duplicating their previous temperature history except for three sample positions which are currently about 100 C higher than past temperatures.

The rate of oxidation of boronated graphite has been determined. The oxidation includes two distinct processes: carbon gasification resulting in a weight loss, and oxidation of the boron resulting in a weight gain. Good agreement is observed in mass balance by determining the rate of B₂O₃ buildup by removal of leachable B₂O₃ and determination of the CO₂ product by gas chromatography. The specific response of the chromatograph was shown to vary substantially with varying CO₂ concentration.

Accurate BET surface-area measurements were made on samples with a total surface area of about 1 m².

Irradiation Damage to Reactor Metals

Seven nickel-base alloys will be irradiated in the EBR-II. Tensile and creep-rupture tests will be conducted to determine the effects of a fast reactor environment on this family of alloys.

A creep capsule containing AISI 304 stainless steel is currently accumulating exposure prior to testing at 840 C and 5000 psi stress.

An unidentified fine precipitate has been observed in the as-irradiated buttonhead and gage sections of an in-reactor creep specimen tested to rupture at 750 C and also in the as-irradiated buttonhead of a creep sample tested at 650 C. Electron microscopical examination of the gage section of an un-irradiated control specimen revealed no precipitation of any type.

Optical examination of the irradiated creep specimens has revealed surface pits at the grain boundaries. No such pits have been observed in corresponding unirradiated control samples. The nature of these surface pits is presently being investigated.

Replica and transmission microscopy have been conducted on tensile specimens of Hastelloy X-280 irradiated at 540 F (280 C) to 1×10^{20} nvt, tensile tested at room temperature, and subsequently annealed at 500 C for two hours. The specimen given a standard heat treatment before irradiation showed no change in the as-irradiated defect structure with annealing at 500 C, while the experimentally-treated specimen, which showed no resolvable defects in the as-irradiated condition, exhibited extensive precipitate formation (identified as M₆C) after annealing.

Replica micrographs of stressed sections of Inconel 600, given a special pretreatment prior to irradiation, revealed a "ghost" grain boundary containing small particles. The observation of larger particles

in the grain boundaries of unstrained sections indicates that strain at 1300 F (730 C) might be accompanied with partial resolution of grain boundary precipitate phases.

Results of an out-of-flux oxidation study on three lots of NPR Zircaloy-2 suggest that differences observed in the in-flux oxidation were likely not due to the high extrusion temperature of one of the lots. Comparison of metallography and corrosion rates of annealed and unannealed specimens from one lot suggests that second phase distribution may account for differences in the corrosion behavior.

An intercalibration test has been initiated among several counting laboratories in order to insure that experimenters involved in the Ir-radiation Effects on Reactor Structural Materials Program have comparable fluence determinations.

Comparison of fracture toughness data for 1/4-, 3/8-, and 1/2-inch thick DCB specimens of A302-B steel revealed no variation in toughness for a factor of two increase in thickness. This independence of toughness with specimen size supports the contention that the data represent plane strain conditions.

Warm pre-stressing was found to induce large increases in the toughness of A302-B steel, although aging after pre-stressing tended to return the toughness to the untreated level.

Four irradiation pins containing 28, AISI 304 test specimens have been discharged from the EBR-II after accumulating a dose of 2×10^{22} total fluence. Additional irradiation pins are being irradiated to target fluences of from 3×10^{22} to 1×10^{23} . Two subassemblies containing cladding and structural materials are being readied for elevated temperature EBR-II irradiations in a sodium environment.

Liquid metal capsule GEH-22-3, containing tensile specimens of several austenitic stainless steels, has begun its third irradiation cycle in the ETR. A new (GEH-22-4) sodium-filled capsule also containing austenitic stainless steel specimens is in final assembly. The operating temperature of this new capsule will be 1400 F (760 C).

Work has been started on general laboratory support facilities for the creep and creep-rupture testing facility. Work has also been started on a prototype, shielded, creep-rupture cell. Specific requirements have been determined for an inert gas glove box for handling and loading liquid sodium. The shielded test portion of the liquid sodium loop is now undergoing detail design.

The assembly and testing of the permeation apparatus for the investigation of neutronic hydrogen behavior in fast reactor alloys has been completed, and preliminary permeation studies have been initiated. An investigation has also been started into the effect of a temperature gradient on the concentration

and distribution of carbides and nitrides in these materials, with emphasis on the possible secondary effects on neutronic hydrogen and helium behavior.

Exposure of the refractory metal test assembly in the model gas loop now totals approximately 775 hours at a nominal temperature of 1700 F (925 C). Assembly performance has been satisfactory throughout this exposure.

An ATR specimen assembly containing nickel-base alloy specimens is being fabricated for exposure in the model loop.

Corrosion coupons exposed at a temperature of 2100 F (1150 C) for 59 hours in the ATR model gas loop showed the following weight change behavior: Hastelloy X-280 and Haynes 25 lost weight due to an evaporation mechanism, niobium and tantalum gained weight presumably due to oxidation, and molybdenum and tungsten remained essentially unaffected.

A NiO-MgO fluence monitor is being fabricated for irradiation testing to start in mid-December. It is anticipated that this monitor will have better high-temperature stability than the NiO-Al₂O₃ monitor.

Testing and analysis of Ferrovac E and BMI iron samples irradiated to a goal fluence of 5×10^{19} nvt ($E > 1$ Mev) has been partially completed. The results of activation volume measurements and temperature change tests are very similar to previous results on samples irradiated to 5×10^{18} nvt. The most notable differences are an increased tendency for localized deformation and a greater increase in shear yield strength ($\sigma/2$) of Ferrovac E compared with BMI iron (5.4 Kg/mm² versus 3.2 Kg/mm²) at the higher fluence. The increase in shear yield strengths for the two materials after the lower fluence of 5×10^{18} nvt was about the same at 2.0 Kg/mm².

An internal document, "The Design of Equipment to Generate High Hydrostatic Pressures on Samples Simultaneously Exposed to Nuclear Irradiation," has been written.

The high-pressure, high-temperature, "Belt" apparatus has been successfully tested at the factory to 60 kbar and 1500 C, and has been shipped to BNW.

ATR Gas Loop Operation and Maintenance

The model gas loop was operated 167 hours this month, bringing the total operating time for the new heater to 1733 hours. Seventy-seven percent of this month's operating time was at 2100 F (1150 C).

A meeting was held to discuss the B&W analog study for loop controls, high temperature thermocouples, and other areas of mutual interest. Results of the meeting are summarized.

A purchase order was placed with Calmec Manufacturing Corporation for the large control valves for the ATR gas loop.

Metallic Fuels Development

Tubular fuel elements of Th - 2.5 at.% U - 1.0 wt% Zr clad with Zircaloy-2 exhibit a maximum volume increase of 2.8% after burnups as high as 5.6×10^{20} fissions/cm³ (16,200 MWd/ton) in the ETR hot water loops. The bulk outlet temperature of the loop water was reduced 50 C in order to reduce the surface temperature of the cladding. This should assure adequate corrosion performance of the clad for the duration of the test.

Annealing a specimen of an irradiated Th - 2.5 wt% U - 1.0 wt% Zr element (GEH-10-66, 1.4 at.% BU) at 800 C for 100 hrs resulted in a measured swelling of 22.5%.

Fifteen hollow-core uranium fuel elements, 0.45-inch diameter by 6.25-inch long and clad with Zr-2 have been charged in the ETR M-3 loop facility. Additional rods are being fabricated to increase this charge to 24 fuel elements. An additional replacement charge of 24 elements is also being fabricated.

ENGINEERING DEVELOPMENT

Neutron Flux Monitors

A high flux level in-core test completed at a production reactor will help to further define the operational characteristics of the second generation boron-1 detector. An equation which accounts for beta current generation, competing reactions, energy effects, burnout, and device configuration is being derived to describe the beta generator current.

Upstream Boiling Burnout

Heat transfer experiments to study upstream boiling burnout were performed on an electrically heated test section 0.435-inch ID and 6-ft long. The local pressure drop data obtained suggested that a flow pattern change precedes the occurrence of downstream burnout. This new flow pattern was observed to persist in the unheated length downstream of the test section.

PLUTONIUM UTILIZATION PROGRAM

Fuels Development

Fabrication of five (5) UO_2 (9.54 wt% U-235 enriched) fuel capsules for TREAT experiments was completed.

The damaged pressure sensing devices on two instrumented fuel rods for PRTR were repaired.

An irradiated HPD fuel rod was destructively examined to assess whether changes which may deleteriously affect fuel performance occurred during the past 15-month storage. No such changes were detected.

A FERTF test element of the new design is being fabricated.

Underwater disassembly and manipulation equipment for the examination and maintenance of FERTF experimental test fuels is being designed and will be fabricated locally.

Two Hastelloy baskets for the FERTF fuel elements are being fabricated and are scheduled for completion by mid-January.

The high exposure plutonium studies program is revised. Shippingport and Yankee fuels are onsite and being analyzed for isotopic content and gamma energy spectrum.

Reactor Physics

A complete compilation of burnup data from the plutonium-aluminum fuel which contained initially 16 at.% Pu-240 is being issued as report number BNWL-CC-905. The generalized least square fitting code, program LIKELY, has been modified to facilitate the processing of fuel burnup data.

The reactivity worths of D_2O , H_2O , and air coolants have been measured in PRTR in the central 55 fueled channels.

Papers, "Measurement of the Reactivity Worth of Hafnium-Oxide Rods in a $\text{PuO}_2\text{-UO}_2\text{-H}_2\text{O}$ Lattice," and "Critical Experiments with High Power Density Fuel in the Plutonium Recycle Critical Facility," were presented at the American Nuclear Society Winter Meeting held at Pittsburgh, Pa.

Critical experiments were continued in the PRCF using 2 wt% $\text{PuO}_2\text{-UO}_2$ fuel in an H_2O lattice. Experiments using fuel containing 8 wt% Pu-240 were conducted during the month.

Survey calculations for a $\text{PuO}_2\text{-UO}_2$ fuel in light water are being performed as an extension to a study previously reported in BNWL-305.

A calculation of lattice reaction rates for a 19-rod cluster cell has been made using the RBU Monte Carlo code. Lattice parameters of this cell have been measured in the PCTR, and comparison of calculated and measured values are being made.

An improved calculation is being made for the design of a water tank to be used in the core of the PCTR. These calculations will also be utilized in future water lattice experiment designs for the PCTR core.

The first set of four, 20-inch long fuel rods containing "zero size" PuO_2 (2 wt% $\text{PuO}_2\text{-UO}_2$ solid solution) were completed.

Work continued on the preparation of spherical particles from high energy impacted PuO_2 .

The experiments in the PCTR with Hx mixed oxide rods (2.6% PuO_2 in UO_2 rods - 29% Pu-240) and Lx mixed oxide rods (0.9% PuO_2 in UO_2 - 8% Pu-240) in the 4-inch square graphite lattice have been completed. Analyses of the data are in progress.

A new technique was developed to determine the relative plutonium content in Pu-Al foils with measurable amounts of Pu-241. This technique provides information on relative foil normalizations without the need to activate the foils in a uniform neutron flux.

A new data tape is available for the Hanford Revised GAM (HRG) Computer Code. This data tape includes the latest cross section revisions to 80 isotopes and an extensive rearrangement of nuclide identification numbers.

The computer code UPDATE was modified to provide much faster running time.

The computer code UNICORN from Atomics International has been imported for resonance parameter calculations.

Information from the Evaluated Nuclear Data File "A" (ENDF/A) and from the Sigma Center cross section library tape (SCISRS) is now available for retrieval request on the Univac 1107.

A code has been written which computes Dancoff factors for arrays of cylindrical fuel codes.

Work has continued on the preparation of data for the AEC Evaluated Nuclear Data File/B.

Final processing of previously obtained 3- to 15-MeV total cross section data is in progress.

Reactor Engineering Development

Hydraulic experiments on a full-scale model of the FERTF fuel element showed that 83% of the total tube flow went through the fuel and the remaining 17% was bypassed around the fuel through the annulus between the basket and the pressure tube wall. At the desired coolant flow rate of 65 gpm through the fuel, a pressure drop of 0.6 psid will occur across the fuel, and the maximum coolant temperature at the exit of the fuel will be 121% of the temperature of bulk mixed flow at the pressure tube outlet.

Modification of a linking code called "CHAIN" to include REPP as a link in a set of economic and nuclear burnup codes was completed. A user's manual for operation of REPP is currently being written.

Materials Development

A crack propagation test was performed on a specimen of maximum exposure from tube #5696. A value of 41,000 psi was obtained for a 1.5-inch crack which is consistent with values for unirradiated specimens.

The OPG decontamination process has been applied to the cleanup of FERTF heat exchangers and heaters with apparently excellent results. Preliminary calculations indicate about 50 grams of rupture debris have been removed from the system.

Test Reactor Operation

November was spent in preparing for, and conducting the containment vessel leak rate test and restoring all systems and equipment to normal. The leak rate at 10 psig was 0.43% for 24 hours which was acceptable.

The first phase of the rupture loop decontamination was completed. Approximately 50 grams of fuel material were removed and maximum radiation readings were reduced by a factor of about 40.

The PRTR made miscellaneous startup preparations while awaiting AEC approval to begin batch core power tests. One week will be required after approval is obtained.

EBWR Demonstration Program

Two capsules prepared with short lengths of EBWR, Zircaloy-clad, vibrationally compacted, impacted UO_2 - 1.5 wt% PuO_2 fuel have attained a maximum burnup of 26,900 MWd/ton (6.73×10^{20} fissions/cm³). Two production-

run EBWR rods (burnups are 1650 and 5500 MWd/ton) show no evidence of internal or external hydriding in the lower end cap region (only unauto-claved part of the rods).

The EBWR is presently (November 23) at a power level of 40 MW, and reactivity estimates indicate possibilities of reaching a power level near 70 MW.

A calculational study of the Saxton plutonium experiment has been started. Initial studies concern the theoretical analysis of experiments conducted at the Westinghouse Reactor Evaluation Center.

NUCLEAR SAFETY

Containment Systems Experiment

Construction work was completed on containment vessel penetrations. Installation of leakage rate testing instrumentation was completed, and preparations for planned air leakage rate tests are in progress. The reactor simulator vessel was delivered, and the structural steel for the blowdown test stand was installed. Two runs were made in the Aerosol Development Facility. One run investigated iodine behavior in a stainless steel tank during a pressure decay from initial conditions of 36 psig and 255 F (124 C). The other was an isothermal run at 175 F (69 C) in the painted aerosol tank to obtain data during the tank's initial exposure to steam.

Pressure Vessel Crack Monitoring

Development work at BNW, to produce a system to detect crack growth in reactor pressure piping by sensing the acoustic emission, was compared with similar application work being done at LRL and the NRTS. It was found that the three programs are quite complementary and that no unnecessary duplication of effort exists.

Reactor Safety Analysis and Evaluation

The analysis of the results of the irradiated A212B subsize-vessel burst tests is continuing. Fifteen unirradiated A212B subsize-vessel specimens were destructively tested. A microhardness flow surface technique has been applied to the irradiated A212B subsize-vessel material.

Columbia River Sedimentation Studies

A significantly better correlation between radionuclide concentrations in bottom sediments and distance along the Columbia River is obtained by plotting logarithm of concentration in a particular particle-size fraction rather than that in a sample containing all sizes of mineral particles.

Waste Solidification Condensate Treatment

A two-step scavenging process appears to be feasible for treating glass melter condensate. Phosphotungstate and mixed hydrous oxides are precipitated, centrifuged, and subsequently dissolved for recycle.

Fission Product Aerosol Containment

Testing of equipment associated with the new, larger spray chamber was started.

The reaction between methyl iodide and hydrazine was studied by determining the influence of added chemical agents, temperature, and gas phase volume on the removal of methyl iodide from a static gas volume. A model for the reaction was proposed and tested with the available data.

Radioactive Waste Solidification

A design verification test of the continuous phosphate glass equipment indicated both operating and equipment difficulties using a nonsulfate waste.

The EDL-102 spray calciner has been placed into operation using a PW-1 flowsheet. The first series of runs will further evaluate the comparative performance of air and steam as the atomizing fluid.

A spoolpiece is being fabricated which will permit the testing of batch melting in the receiver pot below the EDL-102 spray calciner. The corrosion rate of 304L stainless steel immersed in a PW-2 melt at 800 C was measured at approximately 135 mils/mo; this is low enough for batch melting.

The first engineering scale USA conversion of highly radioactive liquid waste to solids by pot calcination was successfully completed in WSEP.

Design was completed and fabrication was initiated on test pods for providing controlled environment storage for solidified waste containers.

DIVISION OF REACTOR DEVELOPMENT AND TECHNOLOGY PROGRAMS (F. W. Albaugh)

CIVILIAN POWER REACTORSNuclear Systems Concepts Analysis (E. A. Eschbach)Fuel Utilization Studies

A simulation of the future nuclear economy using a two-reactor description and dividing the next 70 years into 35 two-year periods was presented at the Systems Analysis Task Force meeting in Washington, D.C., early in the month. This simulation showed that the tools for performing the analysis were available and that the method was feasible. The calculations are now being repeated with a five-reactor model. The reactors are (1) the Westinghouse 1000 MW fast reactor, (2) a PWR operating on uranium, (3) a PWR operating on plutonium recycle for four years, (4) a PWR operating on plutonium recycle for eight years, and (5) a PWR operating on plutonium recycle for 12 years. In the last three cases, the reactor finishes its life operating with uranium-enriched cores. The input data for these calculations are now being prepared. The results from the recalculations will be used as a basis for a writeup of the methods and the model being used in the over-all system analysis.

The Phase II effort, which will involve a many-reactor problem, requires some modification of the DAEDALUS code, the linear program matrix builder, because different input data are to be used. Phase I used 30-year burnup data input whereas Phase II will use annual cash and mass flow data as input. The DAEDALUS code has been rewritten to accept this new form of input. Data are being prepared for the Phase II many-reactor problem. Thirty-year cash and mass flow histories are being prepared for several types of light water, advanced converter, and breeder reactors. Thorium cycles are being included. The Phase II model will consider the impact of the conventional power industry as another "reactor" which can be built. It has capital and operating cost and satisfies part of the over-all energy demand. The entire national energy demand is being divided into three regions on a cost basis. It will be assumed that nuclear power must compete with full power independently in each of these three--low, medium, and high--fuel cost regions of the country. Growth curves for each are nearly complete for the 70 years being considered.

The plant factors of the fossil plants, as well as the plant factors of the nuclear plants, must be varied over the lifetime of the plant. The preliminary Phase II calculations will assume that all conventional and nuclear power plants will be integrated into a system with an over-all capacity factor of 67%. If each power plant operates at an 85% plant factor the first five years of its life and the system doubles every 10 years,

plant factor in the last year of each plant's life (if the factor drops linearly) will be 15%.

The plutonium restrictions for the thermal and fast reactors will consider plutonium in three stockpiles: (1) Material which has been discharged from uranium-enriched light water reactors and from cores of fast reactors. This material contains 60% Pu-239, 24% Pu-240, 12% Pu-241, and 4% Pu-242. (2) Plutonium which has been recycled in thermal reactors. This material's composition is 35-32-15-18, respectively. Finally, (3) plutonium discharged from the blanket of fast reactors with a composition of 96-4-0-0.

The first computer run of Phase II effort will be done in mid-December, using most of the data supplied by other task forces for various reactors. In early January, the entire Phase II problem will be run for presentation at a mid-January meeting of the Systems Analysis Task Force.

The coefficient iteration capability, that is, changing the data of the linear program problem and re-optimizing with the linear program codes, has been delayed because we have been unable to arrange mutually satisfactory schedules for consultation with the firm which programmed this capability. Arrangements are under way for this consultation to occur in early December.

Code Maintenance

The last few bugs in a new version of the two-diffusion group ALTHAEA for the 1107 have been corrected. This new version has been adapted so as to create a burnup tape in a binary format identical to that produced by the new four-diffusion group ALTHAEA. Associated problems in PLOTTER have also been corrected.

Code Development

The ALTHAEA code was revised to provide automatic recycle of fuel isotopes in the one-dimensional mode. Recycle control cards may be read at any time during the reactor life to change the recycle mode (for example: startup with enriched uranium, recycle plutonium after some time interval and then return to enriched uranium after another interval). At each change in mode a set of cards may be read defining a schedule of load factors and enrichments for the reactor to use for the remainder of its life. Demonstration output is in preparation for use as input to the linear program model of the electrical industry.

Fast Reactor Economics

Preliminary calculations of the Westinghouse reactor indicate a plutonium value of \$19 compared to U-235 from the cascade. With plutonium at \$10/g and

interest at 10%, fuel cycle costs were 1.2 mils versus 1.4 for the Allis-Chalmers reactor.

Cash Flow

The PACTOLUS code is essentially written, and the code seems to be performing as expected. Debugging of the code will continue to ensure the accuracy of the results.

The code writes an output tape which will be read by DAEDALUS which makes up input for the 1107 LP code. This will be used in the Fuel Recycle Task Force work.

30-Year Burnup Economics - PNL Optimum Cycles

A special version of QUICK is being prepared to give optimum enrichments for varying operating and economic conditions during a 30-year reactor lifetime. Zero-dimensional ALTHAEA burnups will be used with this version of QUICK to provide enrichment values for one-dimensional 30-year burnups where such parameters as load factor, fabrication cost, reprocessing costs, and cost of uranium feed will vary during the reactor lifetime.

30-Year Burnups - Offsite Optimum Cycles

A special version of the FAST code has been prepared to manufacture 30-year binary burnup tapes from cards containing inventories in kilograms. These binary tapes can be read by late versions of QUICK and PACTOLUS.

Code Modifications

Modifications to the "in-house" version of FCC are continuing at a slackening pace. The purpose of the modifications is still directed toward minimizing code input while maximizing simplicity and hence freedom of operator error. Considerable difficulty was encountered in circumventing the many system errors in the current Univac MAP, or memory allocation processor.

Conceptual Reactor Design Studies (J. C. Fox)

Thermal Sink Limitations Study

All U.S. Geological Survey data existing in the BNW Library on river flows and temperatures since 1950 have been tabulated. These data cover USGS Areas 9 through 14, encompassing river basins in the Pacific Northwest and California, the Colorado River Basin, and the Great Basin of Utah and

Nevada. Temperature data are incomplete for all areas, and flow data are complete only for Area 14 (Lower Columbia Basin and Oregon Coastal Streams). Correlations of the tabulated data are proceeding while additional data is awaited.

Reactor Siting Study

Subcontract negotiations with R. W. Beck and Company are essentially complete, and work should begin about December 1. Their work will consist of engineering cost estimates and cost considerations relevant to different types of heat dump and to siting in various sections of the country.

Steam-Cooled Fast Reactor

A MIMIC Program to estimate the cycle efficiency is nearly completed. The analysis includes determination of steam circulator efficiency, an important variable when examining the Loeffler cycle. The analysis also may include effects of regenerative heating by using efficiencies and outlet enthalpies from typical large steam generation plants.

The effect of surface roughening on heat transfer and pressure drop is being considered to permit using surface roughening to improve the heat transfer characteristics of the reactor core.

USAEC-AECL Cooperative Program (J. J. Cadwell)

Cross-Flow Mixing Between Parallel Flow Channels During Boiling

The experimental portion of the program to determine values of cross-channel mixing during two-phase flow in rod bundle fuel elements was continued. The basic purpose of this program is to obtain a method to predict local flow and enthalpy conditions in each subchannel of rod bundle fuel elements. This information will help provide a more accurate means to predict boiling burnout conditions.

Analysis of the experimental data for the test section simulating a flow channel formed by fuel rods in a square pitch array located adjacent to a channel formed by rods on a triangular pitch array (as in a 19-rod bundle) was completed. Two sets of experimental data were taken at a system pressure of 900 psia. In the first set, a simulated rod spacing of 0.080-inch was considered. Data were taken at selected values of heat flux for inlet temperatures of 330 F (165 C) and 510 F (265 C) and average mass velocities of 1.0, 2.0, and 3.0 x 10⁶ lb/hr-ft². In the second set, a simulated rod spacing of 0.020-inch was considered. A similar set of data was taken as in the first set except for the deletion of the test at a mass velocity of 1.0 x 10⁶ lb/hr-ft² with inlet temperature of 510 F (265 C).

The reduced data have revealed some interesting effects:

- 1) At simulated rod spacing of 0.020-inch, the turbulent mixing is very nearly the same for two-phase flow as for single-phase flow. This result was found by comparing the experimental subchannel flow and enthalpy conditions to the predictions of the computer code developed earlier to relate these quantities to turbulent and boiling mixing. By using a single value for turbulent mixing for both the boiling and nonboiling conditions, correspondence was found between calculations and experiments, thus leading to this result.
- 2) At a rod spacing of 0.080-inch, the turbulent mixing during two-phase flow conditions appears to be higher. By comparing the experimental data with the predictions of the computer code using various values of turbulent mixing, it was concluded that the turbulent mixing was about two times higher for two-phase flow as compared to single-phase flow for this spacing.
- 3) The turbulent mixing does not appear to be linear with rod spacing. When the rod spacing was increased from 0.020-inch to 0.080-inch, an increase in the turbulent cross flow by a factor of about two times occurred. If the cross flow is assumed to be proportional to the rod spacing, the cross flow would have increased by about four times. Therefore, a linear relationship is not indicated.

A complete explanation of the differences observed is not readily apparent; however, it would seem that phase distribution, velocity distribution, and the scale of turbulence would be important factors. Analysis of the data is continuing to study the observed phenomena.

Evaluation of Zirconium - 2.5% Nb Pressure Tubing

The plastic anisotropy of Zr - 2.5 Nb was determined for three heat treatments before and after the specimens were irradiated for two cycles in the G-7 loop of the ETR. These were the same specimens as furnished by CRNL for use in the In-Reactor Corrosion Evaluation of Zirconium Alloys program. The heat treatments were as follows:

- 1) Held 15 minutes at 850 C, quenched, 20% cold work, aged 24 hrs at 500 C.
- 2) Same as above with 30% cold work.
- 3) Held at 575 C for 5 hrs, then cold worked 20%.

Each of the above heat treatments caused a distinctly different hardening response to irradiation. The irradiation strengthening can be described as combinations of three models:

- A. Kinematic - a translation of the yield envelope in the direction of a stress deviator without change of shape.
- B. Isotropic - an enlargement of the yield envelope without translation.
- C. Batdorf Budiansky - a bulging of the yield envelope in the direction of a stress deviator.

The specimen heat treated as in 1) above showed the greatest irradiation hardening, 25%. The hardening was type B, isotropic. Specimen number 2) hardened according to models A and C. The translation of the flow surface was as if the specimen had been compressed in the transverse direction, and the bulging was as if the specimen were stressed biaxially as in a tube burst. Specimen number 3) hardened as a combination of models A and B. Again, the translation was as if the specimen had been compressed in the transverse direction.

The shape of the flow stress envelopes was determined by the Knoop hardness method described in previous reports. Specimens representing all three of the above heat treatments with twice the exposure, 4.5×10^{20} nvt greater than 1 MeV, are now available for flow stress envelope measurement.

Hydraulic and Nuclear Stability in Parallel Flow Channel Systems

The analog computer study of hydraulic and nuclear stability of boiling water cooled reactor systems has been completed. A rough draft copy and preliminary analysis of the results have been sent to all members of the TAC Subcommittee on Control and Stability. Additional computer runs were made during the month at the request of AECL personnel. Preparation of the final report for this program is under way.

In-Reactor Measurements of Creep in Zr - 2.5 Nb Alloy

A creep test on Zr - 2.5 Nb is continuing at 55,000 psi and 350 C. A second test on Zr - 2.5 Nb at 36,500 psi and 400 C has ruptured, and the creep capsule has been sent to the radiometallurgy facilities for specimen retrieval and examination.

In-Reactor Corrosion of Zirconium Alloys

Three quadrants (210, 211, 214) containing zirconium alloy specimens were exposed at three flux levels in the G-7 loop of the ETR. The loop

operated at 270-280 C for 77.4 days. The coolant was pH-10 NH_4OH , <0.05 O_2 . Out-of-flux control specimens were exposed in the loop at water transport times ~ 1 sec and ~ 1 min from the flux.

Exposure conditions and preliminary data from the four-cycle test were reported in the October monthly report. Weight gains at the two out-of-flux and three in-flux positions are shown in the table. The weight gains were taken after removal of a black deposit from the samples by wiping with a cotton pad. While chemical cleaning will be done on the specimens to determine if crud removal was complete, the appearance of the specimens and reproducibility of duplicate specimens suggest that the conclusions of the test will not be changed by additional cleaning. However, variations did appear on identical specimens of prefilmed Zircaloy-2, Zircaloy-4, and VALLOY. Separate weight gains for individual specimens are shown in the table of the three prefilmed materials. Otherwise, the weight gains represent averages of in-flux weight gain on specimens in duplicate or triplicate.

The data show:

- Small flux effects for etched specimens, except for VALLOY-S and Zr - 2.5 Nb, which do not show flux effects.
- Considerable flux-induced oxidation for VALLOY, Zircaloy-2, and Zircaloy-4 specimens prefilmed at 400 C, 1500 psi.
- Slightly faster in-flux oxidation rates for heavily cold-worked Zircaloy-2.
- No significant differences in oxidation rate at two G-7 loop out-of-flux positions.

Prefilmed Zircaloy-2 and Zircaloy-4 show general increases in oxidation rate with increasing flux and reproducible behavior for samples in the same quadrant (exception: Zircaloy-2 in Quadrant 210). Prefilmed VALLOY weight gains differed widely for samples in the same quadrant. The weight gains did not correlate with fast neutron exposure.

APPLIED AND REACTOR PHYSICS

Plutonium Criticality Studies (E. D. Clayton)

Basic Criticality Experiments With Plutonium Nitrate Solutions in Slab Geometry

Criticality studies were continued with plutonium nitrate solution in slab geometry. In these experiments the plutonium concentration was ~ 58 g Pu/l

IN-FLUX AND OUT-OF-FLUX WEIGHT GAINS ON ZIRCONIUM ALLOYS AT 270-280°C

Alloy	Condition	mg/dm ² Preautoclave ^a Weight Gain	Out-of-Flux		In-Flux		
			1 sec	1 min	1.8 x 10 ²⁰	4.3 x 10 ²⁰	7.8 x 10 ²⁰
Zircaloy-2	Annealed	E	11.0	9.5	14.1	15.4	16.4
Zircaloy-2	Annealed	17.8-19.9	3.5	3.9	(23.4, 13.1, 14.1)	(27.6, 35.1)	(51.7, 51.3, 44.8)
Zircaloy-2	80% cold work	E	12.8	wt. loss	16.1	17.8	19.5
Zircaloy-4	Annealed	E	12.1	11.0	14.1	15.4	16.2
Zircaloy-4	Annealed	17.2-18.0	1.8	4.0	(7.4, 19.0)	(10.2, 15.9)	(32.1, 26.6)
VALLOY	Annealed	E	-	10.6	13.7	13.0	14.4
VALLOY	Annealed	14.0-14.4	2.2	4.7	(109.7, 70.3)	(198.3, 82.2)	(82.8, 29.0)
VALLOY-S	Annealed	E	29.7	27.9	27.9	29.7	30.3
VALLOY-S	Annealed	19.2-20.5	-	-	-	-	-
Zr-3Nb-1Sn	Annealed, Aged	E	13.9	13.4	-	-	-
Zr-3Nb-1Sn	Annealed, Aged	20.0-21.9	-	-	6.7	6.7	10.5
Zr-3Nb-1Sn	Quenched, Aged	E	12.4	12.2	12.9	15.5	17.1
Zr-2.5 Nb	Quenched 20% CW Aged	E	11.1	14.3	12.2	11.1	11.9
Zr-2.5 Nb	Quenched 20% CW Aged	13.4-14.6	9.0	4.9	5.4	3.7	5.4
Zr-2.5 Nb	Quenched 30% CW Aged	E	13.0	13.6	10.3	11.2	11.9
Zr-2.5 Nb	Quenched 30% CW Aged	12.7-14.0	8.7	8.2	7.0	4.7	9.9
Zr-2.5 Nb	Annealed 20% CW	E	16.0	12.8	13.1	10.1	13.0
Zr-2.5 Nb	Annealed 20% CW	11.9-12.8	9.1	17.6	12.3	10.7	4.0

^a Zr-2.5 Nb specimens prefilmed 400°C, 3 days, 100 psi steam; all other alloys prefilmed 400°C, 3 days, 1500 psi steam. E denotes specimens exposed as-etched.

with the acid molarity being about 5.2. Vessel thicknesses, conveniently obtained with the unique expandable slab tank, that were used were in the range 4.9 - 6.7 inches. The measured critical volumes in the 42-inch slab tank ranged between 51 - 171 liters. The purpose of the experiments is to determine the critical thicknesses of bare and reflected "infinite" slabs and to obtain data on the various factors affecting criticality. The thin steel tank wall, and low density lattice framework which provides structural rigidity, affects criticality differently in the bare and water reflected cases.

During the month experiments were performed to investigate these effects and the effects of reflection generally; twelve experiments were completed. To study the effect of the vessel wall and supporting gridwork on criticality, additional steel in the form of thin plates was added to the assembly in several arrangements. In the water reflected case, the effect of the vessel wall, etc., is to reduce the reflector savings of water by ~ 0.06 inch, the thickness of the vessel wall. In the bare case, the steel has a slightly larger effect on criticality as a reflector (estimated savings of 0.07 inch) than an equivalent thickness of plutonium solution. With these considerations, the true reflector savings for water would be ~ 2.4 cm for dilute plutonium solution in the slab vessel.

The criticality experiments with plutonium solution in slab geometry and analyses of data are continuing.

High Temperature Reactor Lattice Physics Studies (R. E. Heineman)

Reactor Construction

The gas control valves have been received, and installation of these valves and the gas sample piping is in progress.

Installation of reactor thermocouples is in progress. Six of the 80 thermocouples were calibrated with prototypical extension leads for conformance to specifications prior to writing conversion programs for PMACS. It has been necessary to purchase additional thermocouple extension wire and/or splice existing wire runs.

Testing and disassembly for shipping of the neutron generator was witnessed by a Battelle-Northwest representative at the vendor's factory. It is scheduled to be shipped on December 2. Installation is tentatively scheduled for January 1967.

Vendor drawings on the gas drying system have been received for review and approval.

Final approval comments were made on modifications to several systems including the control room layout, the pyrometer, the gas system flow design, and the sampling system.

Comments to the vendor of the independent nuclear channels resulted in several changes deemed necessary for proper operation. The system has been completed, the acceptance tests run, and the certified test data forwarded by the vendor for approval.

Installation of the graphite bus bar system is about 80% complete. An appreciable amount of rework on these parts has delayed progress about two weeks.

Repair of a bus bar section, broken during assembly, was completed using a portion of the EGCR graphite obtained earlier for spare heater material. Sufficient length remains in the log to provide heater spares.

Reactor Equipment

Oscillators. The second main structure subassembly was received, and both units are now installed on their carriages.

The light duty unit has the main backbone, pulleys, cable drum, and driving head installed. Installation of this equipment is under way on the heavy duty unit.

The covers are being modified and strengthened.

The extension bars are completely finished.

Shop fabrication of all components is now complete.

The delay in delivery of hydraulic hose and drive cables has held up preliminary testing of the hydraulic system. Efforts to expedite these items are under way.

The keys were tested and operated satisfactorily prior to final welding and attachment of air lines.

The valves are complete except for attachment of operating air cylinders. Two of the air cylinders are onsite, but the third will not be delivered until the middle of December.

It is estimated that the project is 64% complete.

Control Rods. Pre-installation and adjustment of drive assemblies is continuing. The motor manufacturer sent a field technician to check into the apparent feedback system problem which was reported last month. Following operation and analysis of the system in question, he confirmed the dual pulsing and explained that in the mechanical resonance range (approximately 30 to 60 input pulses per second) it is a normal condition for the stepping

motors to return two pulses in the going direction for one pulse in the opposite direction for each incremental step. Tests with the PMACS drive and logic circuits revealed a tendency of the mechanism to stall at certain drive pulse rates. A test setup has been provided to evaluate these problems.

Assembly of control rod elements is scheduled for the first week in December. The floor area in the southwest section of the 321 Building has been cleaned and will be papered and roped off to provide a control zone for handling and assembly of $\text{UO}_2\text{-C}$ tubes into the rods. RMO preliminary survey has indicated the $\text{UO}_2\text{-C}$ tubes are slightly smearable (100 mr/hr, beta) so extra precautions will be taken during assembly. Radiation Work Permit and Criticality Safety Specifications have been processed. All necessary approvals are expected in time to proceed as scheduled. Assembly of ten rods should be completed by the middle of December. They will then be transferred to the HTLTR.

Programmed Measurement and Control System (PMACS). The gas cycle program has been completed and debugged except for two aspects: the procedure for turning on the gas analyzers has not been determined, and the program has not been checked out with the actual plant since the equipment is not installed.

The heat program is about 90% completed for phase one. It should be noted that this heat program will be called Mark 1 since it does not average the thermocouple readings for the various stack groups. Very good progress was made on the program which calculates the period and the power and checks on high and low level limit trips. Preliminary debugging showed that period calculations were very good using fixed point arithmetic.

In order to save time when the HTLTR is completed, methods of estimating controller settings are being developed. The present effort is aimed at generalizing previous work and evaluating the resulting formulas. The formulas were evaluated for continuous systems and continuous controllers using the 1132 analog computer. The results from the tests showed the formulas to be valuable for estimating optimum controller settings.

A readjustment was made to the scheduling sheets which were originally outlined. The most noticeable change is a more realistic number for phase two and phase three. The total time allotted for 21 programs is now 27 weeks for phase two and 10 weeks for phase three. Not reflected in these manweek figures is the addition of many other programs and rather extensive changes to the now existing programs. Programs which have been added are the engineering routine which will convert all analog counts to meaningful engineering units, several different demand logs, about eight demand alpha numeric display programs, and an input-output routine. Some of the extensive changes are caused by such requirements as: (1) Automatic shutoff of the heaters when various conditions are not met. (2) Demand display of the maximum TC of the heater group. (3) Continuous display of four average stack temperatures.

(4) Continuous display of the absolute pressure. (5) A demand display for the high accuracy periods digitizer 1 and 2, the power on 1 and 2, and the set point for power. (6) The capability of bypassing automatic control and turning the gas blower off or on.

The computer was used for a total of three days testing out the bad tapes. On the third day a Digital Equipment representative delivered 12 new tapes. These tapes were put through a rigorous testing routine, and only one worked on the PDP-7. Upon rewriting the 11 tapes with new timing tracks and retesting them, the tests showed no errors. Two of the tapes were taken to Seattle and tested on the Boeing PDP-7. Their machine found errors on the tape that we wrote a timing track on; hence, it appears that there is something different about the PMACS PDP-7 tape units. No further difficulties were experienced with the tape units or the tapes for the rest of the month.

The alpha-numeric display failed again during the report period. The blue unblanking circuit required a transistor to replace one that had failed. In addition, the circuit breaker on the display unit occasionally will trip; this is apparently due to operating the unit too close to the load trip point of the breaker.

The left hand DECTape unit of the PMACS was balanced by increasing the torque to the weak left hand torque motor. The balancing was necessary because a new torque motor was installed in the left hand DECTape unit.

Reactor Operations

The three Acceptance Test Procedures (ATP's) which require the use of PMACS are being reviewed. These will be revised to use programs that are already written for PMACS where possible. In fact, the revisions will result in a design test to provide a complete system checkout, a component calibration, and a test of program compatibility.

Reactor Physics Program

Startup and Calibration Experiments. The reactor test core was to be mocked up in November. However, it was found that layers one, sixteen, and seventeen were not complete. Additional machining of 135 bars is necessary. An inspection of the blocks for the other layers is being made to minimize later delays due to machining. This additional machining will delay completion by about two weeks. However, due to delays in assembly of the graphite bus bars, noted earlier, the core will still probably be ready to be installed prior to sealing the shell.

The heater thermocouples ordered for the test core would not pass the test requirements of the specification. The test requirements were relaxed

on the high temperature resistance to sheath requirements. The new test requires a minimum of 5000 ohms at one volt dc when a three-foot section of the thermocouple is inserted into a furnace at 1800 F.

Uranium-233 - Thorium Experiments. Work continued on the design of the U-233 - Th experiments to be performed after startup. The startup lattice, 7-1/2" pitch, will be used for the first experiments. In addition, the modifications necessary to the test lattice for the 3-3/4" lattice experiments, which will follow the 7-1/2" ones, are being designed. The oscillator requirements of both of these lattices are being investigated and coordinated with the design group in an attempt to make the couplings simple yet as universally applicable as is feasible. The basic 3-3/4" lattice will also be used as an 11-1/4" lattice merely by changing the fuel and definition of central cell.

Nickel foil is on hand which is sufficient to poison the central cell but not the entire test lattice. Copper foil is also available, and it is expected that a comparison of Cu and Ni will be made at low temperatures.

A test of the unpoisoned technique for measuring k_{∞} is still in progress in the PCTR. Preliminary results indicate excellent agreement between the unpoisoned result and the result obtained by the null reactivity technique for low exposure PuO₂-UO₂ fuel in a 4" lattice, indicating that this technique will be useful in the HTLTR at high temperatures.

An investigation of possible methods of measuring reactivity traverses at high temperatures has begun. The detector possibilities include Monel, copper, and gold as wire pulled by a graphite cable or as foils. The neutron filter will probably be gadolinium instead of cadmium. The oscillator will be used to insert and remove foils.

Control Rod Worth Evaluation. A test in the PCTR to estimate the worth of the HTLTR control rods has been completed.

Preliminary analysis indicates that a control rod will be worth about 60% of a driver and that, if necessary, replacement of the inner graphite cylinders by ones made of the Gd₂O₃-graphite temperature shim material would increase this worth by about 15%.

Driver and Flux Adjusting Fuel. Natural UO₂ pellets were decanned from the former PCTR Al-clad fuel rods. Approximately 350 pounds of the pellets were ground to remove surface contaminants.

Assembly of the driver fuel elements, 5% enriched UO₂ pellets clad in graphite, was started. About 175 10-inch long elements have been assembled. A portion of the threaded 6-inch long graphite tubes and end plugs were returned to the fabricator for rethreading because of imperfect fits. Additional end plugs and the 60-inch and 48-inch long graphite tubes are being machined. Delivery is expected in December.

Phoenix Fuel Reactor Program (D. D. Lanning)

MTR Phoenix Experiment

CAF-Phoenix Experiments. Approach to critical experiments in the Critical Approach Facility were completed this month. The effect of placing beryllium tri-flutes in the interstices of the beryllium columns (30 tri-flutes were used) was measured and found to have a small positive effect on the reactivity of the assembly.

The center fuel can was replaced by a poison can and fuel added, in three steps, until the remaining 18 fuel cans were full. The results, not completely analyzed, indicate the poison can is worth more than 20 inches of fuel in the remaining 18 fuel cans when these 18 cans are surrounded by a layer of beryllium columns.

Noise data from these experiments were recorded.

PRCF-Phoenix Experiment. All structural components for the Plutonium Recycle Critical Facility-Phoenix experiment are presently being fabricated by Minor Construction, with delivery promised by December 15, 1966.

Assembly of the beryllium logs and tri-flutes needed in the PRCF-Phoenix core will begin shortly. About 400 logs and about 800 tri-flutes will be required to mock up the MTR beryllium reflector. Aluminum spacers will be used to simulate the horizontal beam ports through the reflector.

Studies of postulated reactor excursions in the PRCF-Phoenix core have been completed. The results are that the large negative coolant voiding coefficient will limit an excursion before any fuel melting takes place. A detailed report of these studies will be issued.

Fuel Fabrication for PRCF. The present status on Al-clad, Pu-Al fuel plates is as follows:

	<u>Fuel Element</u>	<u>Shim Fuel</u>
Finished fuel plates, acceptable	150	30
Al-clad, Pu-Al fuel plates	44	66
Pu-Al core plates in clad welding process	74	
Pu-Al core plates being fabricated	<u>36</u>	<u> </u>
Total	304	96

Plutonium segregation accounted for 23 rejects this past month. Probably there are more fuel plates which will have to be rejected for this reason.

Two process variables were determined as probable causes. The designs of the fuel element and shim control rod assemblies were revised. Fabrication and/or procurement of the assembly components were initiated. Design of the assembly fixtures was initiated.

The rejection of fuel plates at this time should not cause any delay in meeting the experiment schedule. Fabrication and procurement of the components for assemblies is tight. Therefore, a possible delay of two weeks could occur. All steps are being taken to minimize this possibility.

CAF-Phoenix Calculations. An analysis of some preliminary CAF experimental results is under way. These experiments are designed to provide experimental data in support of the MTR core design effort, but, in addition, they also serve to further refine and extend calculational methods and nuclear data for use in design calculations with plutonium fueled compact thermal reactors. Subcritical reactivities are available for water reflected core and beryllium water reflected PuAl-polyethylene cores. Using 1965 Scher Pu-239 thermal cross sections and a four-broad group diffusion theory calculation with HRG averaged epithermal group cross sections and THERMOS spectrum averaged thermal cross sections, the calculated reactivities for both reflectors are high by about 3.5%.

Uncertainties in plutonium composition and Pu-Al disk thickness are estimated to be capable of producing a maximum 2% spread in multiplication. HRG-THERMOS and 4-group diffusion theory were used in this result.

The use of 16 epithermal broad groups results in a 1.7% decrease in multiplication from the standard 4-group case.

The effect of cylindricizing the 19-can hexagonal core array was investigated using two dimensional diffusion theory. Even considering core edge irregularities due to the large 2.25-inch diameter cans, the effect of cylindricizing is estimated to be less than 3 mk for this system.

The study of this calculation-experiment disagreement is being continued.

Code Modifications. An additional link has been added to the ZODIAC 2 chain which allows the use of the HRG slowing down code in place of GAM. In conjunction with the foregoing change a new CLT (composite library tape) has been drawn from the BNW master library. This new cross section set includes the Westcott evaluation of the Pu cross sections. The total package, HRG plus new cross sections, is in the final stages of debugging.

Due to the gradual shift of codes from the IBM 7090 to the Univac 1107, there is no longer available a code to construct a 7090 HRG library tape. This difficulty has been surmounted by writing a code to convert 1107 HRG libraries to the 7090.

A modification has been made to the sigma 3H subroutine to write out a cross section input tape for the 2-D diffusion theory code EXTERMINATOR.

Phoenix Fuel Application Studies

Investigations have been started on one potential Phoenix fuel application, a Consolidated Nuclear Steam Generator (CNSG). A design similar to the B&W proposed CNSG II, reported in BAW-1280, is being used as a basis for comparison. The atom fractions of various materials for this core have been calculated along with the core volume and dimensions as input to physics calculations to be performed by BNW.

Work is next to be started on the two Phoenix fueled CNSG-type cores to be considered, the first of which will have the poison material ($\text{ZrB}_2 + \text{ZrO}_2$) removed and the metal-to-water ratio increased as far as allowed by acceptable hydraulic limits. The second Phoenix fueled core will also have some fuel material removed, the amount dependent on what is optimum for obtaining the Phoenix affect while still meeting acceptable thermal and hydraulic limits and minimum practical pin sizes. A better definition of these two cores will be made during the next report period.

REACTOR FUELS AND MATERIALS

Fast Fuels Oxides and Nitrides (R. E. Nightingale)

A proposed irradiation testing program in support of mixed nitride fast reactor fuels is being developed. A report, "Proposed Irradiation Testing Program for Developing Mixed Nitrides (UN-PuN) as Fast Reactor Fuels," will be prepared as soon as comments on the program are evaluated.

Survey Microprobe Methods for Irradiated Materials

A literature and user survey of suitable instruments, methods, and approximate costs for microprobe analysis of irradiated materials was initiated. A shielded electron-beam probe for use in determination of most elements above atomic number 4 has been investigated, along with a laser-beam emission spectrograph probe system for sensitive determination of metallic elements. Each instrument offers attractive features, but combination of the two into one dual purpose instrument does not appear desirable.

Mixed Nitride Facilities Development

Installation is nearing completion on an inert-atmosphere hood which will provide the capability for performing operations in a pure (oxygen- and

moisture-free) atmosphere. An inert gas fusion apparatus for analysis of oxygen and nitrogen in refractory compounds was installed in the hood. Initial utilization of the facility includes compositional analysis and powder handling operations on UN-PuN compositions.

The hood atmosphere, of either argon or helium, will be maintained at a purity level of less than 10 ppm oxygen and moisture. A gas recirculation system, using a commercial purification unit was designed and constructed to maintain a purge rate of 20 cfm with a total pressure drop through the system of less than two inches of water. The hood includes an evacuable air lock, which allows passage of material into the hood without contaminating the atmosphere.

Leak checking of the hood and air lock to obtain helium leak-tight conditions is in the final stages. Both the hood and analytical system are expected to be operable by December 1.

Basic Swelling Studies (R. D. Leggett)

The purpose of this program is to characterize and understand the behavior of fissionable materials during irradiation. The theories developed provide a basis for the engineering exploitation of metallic fuel materials in advanced reactor applications. Specimens under study at the present time include uranium with and without dilute alloy additions and thorium-uranium alloys.

Irradiation Program

A tandem assembly of two capsules, P-9 (450 C, 100 psi) and 35 (525 C, 30 psi), continues to operate successfully toward the goal exposures of 0.75 and 0.40 at.% BU, respectively. Another controlled temperature, low pressure (~30 psi) capsule (No. 36) is controlled at 450 C toward a goal exposure of 0.3 at.% BU.

Two controlled temperature-pressure capsules (P-10 and P-15) are currently being constructed. Capsule P-10 is in the final stages of laboratory testing while P-15 is about one-third completed. The several levels of uranium enrichment which are represented in each capsule will provide different burnups within approximately the same environment which will enhance the evaluation of the effects of burnup and burnup rate.

Capsule P-10 will operate at 450 C and 5000 psi while capsule P-15 will operate at 700 C and 1000 psi toward goal exposures of 0.2 at.% BU in the natural uranium specimens. Flux dosimeter wires of Fe, Ni, Ti, and Al-Co are sealed in a stainless steel tube running the length of the specimen chamber inside the tubular uranium specimens. Both the fast and the thermal components of the flux will be monitored, and an effort will be made to determine the enhancement of the fast flux component by the uranium specimens.

Both fast and thermal flux monitors will also be mounted on the exterior surface of the capsules.

These capsules will provide data needed to evaluate the effects of temperature (both alpha and beta phase), pressure, burnup, burnup rate, and minor alloying additives on the irradiation behavior of uranium.

Postirradiation Examination

The electron microscope examination of uranium and uranium alloy specimens irradiated to 0.1 and 0.2 at.% BU, respectively, in capsules controlled at 700 C (beta phase) indicates that high purity - high carbon uranium (500 ppm C) contains larger fission gas pores than do U - 400 Fe, 640 Al, 85 Si, 500 C specimens. In both materials, more pores were present in the higher burnup specimens. A large number of tiny second phase particles were observed in both types of specimens which are suspected to be artifacts. These particles preclude making quantitative measurements on these specimens. Selected specimens are now being annealed at 625 C for 100 hrs to evaluate the influence of postirradiation annealing on material irradiated at these high temperatures.

Nondestructive Testing (J. C. Spanner)

Detection of Irradiation Induced Shift in Nil-Ductility-Transition Temperature

Critical angle ultrasonic methods have been demonstrated capable of distinguishing neutron irradiated carbon steel samples from nonirradiated samples although response anomalies were found to exist on both irradiated and nonirradiated samples. Experimental methods are being developed whereby effects of the anomalies will be minimized.

Equipment and facilities are still being procured. A tank-transducer-specimen manipulating facility is currently being assembled. Assembly work will be completed shortly pending receipt of purchased components. Design of the wide-band gated, power oscillator has been completed, and fabrication is about 20% complete.

Plans are being developed for a more advanced measurements program wherein ultrasonic measurements will be performed both before and after irradiation on two or three materials. An austenitic stainless steel, such as AISI 304, which does not exhibit a shift in NDT temperature will be compared against conventional pressure vessel steels, such as A302B or ASTM A542 Class II. (The latter was chosen because it is the most readily available of the four materials mentioned in the recommendations by the PVRC to the AEC.) Total fast neutron exposure will be the variable with parameters of: a) irradiation temperatures (one above and one well below the self-annealing temperature of ~550 F), b) orientation with respect to rolling direction, and c) location within the donor plate with respect to a surface.

Ultrasonic measurements will be averaged over each specimen before and after irradiation. The effects of previously observed anomalies will thus be minimized.

Notification that funds for a nondestructive testing shielded facility will not be available this fiscal year was recently received, and cell planning has consequently been somewhat de-emphasized.

Fundamental Ultrasonic Studies

Studies to provide an improved understanding of the basic nature of ultrasonic wave propagation continued. The continuing Kautz function analysis showed that no apparent fundamental limitations exist with respect to their application to ultrasonic pulse analysis. During the month experiments were devised which show excellent promise for detecting previously undetectable unbonds.

Prior to this month, it was uncertain whether or not the Kautz transformation from the frequency domain to the time domain would be possible by means of a curve fitting process to the frequency data. This is because the frequency spectra of interest apparently do not have analytical representations. This month, however, it was ascertained that curve fitting using the Kautz spectral functions could be accomplished. Therefore, all the features of the Kautz analysis that are essential for its application to actual ultrasonic data are now available. EDPM programs to utilize this technique are now being prepared.

The detection of unbonds at interfaces between acoustically dissimilar materials has always been a problem when the two materials are held tightly together with mechanical forces. It is believed that an interface can be metallurgically unbonded but can be in tight mechanical contact so that ultrasound is transmitted without a reflected amplitude any greater than that expected due to acoustical mismatch between the two materials. This belief is substantiated by past experience with reactor fuel element claddings. There are fuel element standards that have suspected unbonds of the type mentioned but that have not shown any unbond indications using conventional ultrasonic methods.

One such fuel element was examined using a newly devised ultrasonic method, and the previously undetected unbond was easily detectable. The general test method had been suggested earlier, although previous attempts have not successfully detected the mechanically closed interfaces. This method involves the use of a high power ultrasonic beam to alternately compress and expand the fuel element cladding. During the compression cycle it was expected that the unbonded cladding could be pulled away from the interface. If a higher frequency, lower power beam is made to be simultaneously incident over the suspected unbond area, it would be modulated with the lower frequency since energy would flow through and be reflected at the interface as the cladding is alternately pressed against and pulled away from the substrate.

This method was tried and found to be successful in the detection of the previously undetected unbond. The low frequency beam was made to be incident at approximately the Rayleigh critical angle for maximum cladding vibration efficiency. The higher frequency beam was normally incident as in most conventional bond test applications.

It was also noted in these experiments that ultrasonic signals exhibited many effects that have not been observed in previous applications. These effects are believed to be the result of the nonlinear mechanical behavior of the interface during irradiation with the high power, low frequency energy.

Electromagnetic Testing Research

New solid state circuitry has been fabricated and placed in operation in the laboratory to provide six channel display capacity for use with the eddy current multiparameter tubing test device and tubing cross section display. The present multiparameter tester has four parameter capacity, thus the new display circuitry provides two additional channels which may be used to accommodate the future output of additional channels of the eddy current test device or those of another test device. The new circuitry replaces the former breadboard four channel circuits with which two channels could be displayed simultaneously. A control, and associated circuits, have been included which permit adjustment of the cathode ray beam intensity so that the circles in the display representing the inner and outer tube walls register with equal brilliance.

A new defect standard test piece was fabricated to give a wider range of test conditions for better evaluation of the parameter separating capabilities of the multiparameter tester. The new test standard has two outer wall defects, two inner wall defects, three within-the-wall defects located at different depths and a composite defect region having an outer wall defect, a within-the-wall defect, and an inner wall defect.

The multiparameter equipment and new display circuitry was adjusted to display the defects of the new test standard. The defects were presented as radial deflections in three concentric circular traces, an outer trace showing mainly signals due to outer tubing wall defects, a mid-point trace showing signals due only to the within-the-wall defects, and an inner trace showing mainly signals due to inner wall defects. The outer and inner traces both had some undesired signals caused by the within-the-wall type defects. This effect is due to the large size of the within-the-wall

defects used and is expected to be greatly reduced for smaller defects. In the present arrangement the within-the-wall defect signals are shown as radial deflections of a circular trace positioned midway between the two traces representing, respectively, the outer wall and inner wall of the tubing.

At present, the approximate depth of the large defects within the tubing wall can be judged from the relative size of signals on each trace, but a better means to show the depth on the display is needed. Methods to display these within-the-wall defects at their approximate relative depths within the tubing wall are now being explored.

A method for plotting vector (phasor) test signals as a function of position of a transducer has been developed following the idea and initial tests described in the previous report. This method has been instrumented to aid in the display, and detailed study, of electromagnetic fields near the sensing coils. The final system uses essentially the same technique previously reported. A test coil positioner and oscilloscope are driven by a sweep signal so that each position on a sample corresponds to a point on the oscilloscope face. While the test coil is scanning the sample, the test signal is gated regularly and displayed as a vector at points on the screen corresponding to the test coil position. A camera or storage oscilloscope may be used to record a complete scan for study.

Several two-dimensional scans of various flaws have been made with excellent results. The ability to picture test signals as a function of position gives a novel and useful display of the information available.

Infrared and Thermal Research

Efforts to improve the long-term accuracy of the sinusoidal thermal wave instrument have continued. Although past phase angle measurements (taken in groups of 50 and averaged) on two subsequent days reproduced within 0.01° , measurements recently made over a three-day period differed by as much as 0.27° . A larger number of measurements over a longer period of time will be required to give a reliable picture of the accuracy. High accuracy is required for thermal property measurements on highly conductive materials such as copper or aluminum, since changes in their thermal conductivities have a small effect on the input thermal impedance of the transducer. For samples having lower thermal conductivities, such as stainless steel, phase angle accuracy requirements are not so stringent. For measurements on a copper sample, for example, a 32% reduction in thermal conductivity would be required to cause a 1° change in phase of the thermal input impedance of the transducer, whereas for a stainless steel sample only about 10% reduction in conductivity would be required to give the same phase change. This conclusion is based on a theoretical graphical analysis of the transducer characteristics as a function of sample thermal properties. Experimental results from measurements this month on aluminum and copper samples agree to within 1° of the phase angles predicted theoretically.

One of the main advantages of the thermal transducer method for measuring thermal properties is that no special shaping of the sample, other than having one flat surface, is required. (It should also be possible to develop transducers for cylindrical samples.) Hence, the thermal transducer method should be useful for rapidly measuring the effect of irradiation damage on the

thermal conductivity of fuel materials. The capacity of the method to be automated makes it well suited for remote operation. Although tests on the long-term accuracy of the thermal transducer method are difficult, due to the long time periods over which measurements must be made, it is felt that further measurements are warranted by the accuracy requirements in potential applications of the method. The method is not only applicable to thermal property measurements per se, but also has general applications in nondestructive testing. It also has the capacity for application in measurements on fuel materials from the outside of the cladding.

Investigation of a new transient technique for remotely measuring thermal surface impedance of samples has continued. This technique requires access to only one surface of the sample and is believed applicable to samples without special surface preparation. A high intensity flashtube is used to excite surface temperature transients which are sensed with an infrared radiometer and subsequently transformed to surface thermal impedance values.

The first surface thermal impedances were measured with the remote transient technique this month. A graphical method was used to transform the temperature transients to surface thermal impedance values. The graphical method is somewhat crude and time consuming. An automatic electronic transformation method, which should give greater accuracy, is being investigated.

Theory and thermal transducer measurements have both shown that surface thermal impedances of "semi-infinite" samples of any single material should have a lagging phase angle of 45° . Phase angles of 54° and 53° were experimentally obtained in initial transient measurements of the surface thermal impedance of plate glass and teflon. Measurements on stainless steel and copper yielded surface thermal impedance values having phase angles of approximately 75° . The somewhat poor agreement of the initial transient test results with theory is believed partially due to the black paint with which the sample surfaces were coated to cut down reflections during the initial experiments. In addition, the graphical method of transforming the surface temperature transient is suspected of contributing appreciable errors, especially with the rapid transients obtained on metals.

In spite of errors in the initial transient measurements, the method appears to behave basically in the manner theoretically predicted. Although a number of technical problems must be solved before the method can be considered practical, it appears to have great promise as an advanced technique for making remote thermal measurements.

Nuclear Ceramics (R. E. Nightingale)

Melting Studies on UN - PuN Compositions

A high-temperature, high-pressure filament furnace for use in melting-dissociation studies on UN - PuN compositions was calibrated for temperature measurement using an optical pyrometer. A high accuracy pressure gauge was also installed.

Initial experiments in the furnace will be aimed at determining the congruent melting point of PuN and extending the dissociation pressure curve to a nitrogen pressure maximum of 150 atmospheres.

Recovery and Recrystallization in Pneumatically-Impacted PuO₂

X-ray line-broadening analyses of PuO₂ impacted at 1200 C and pressures greater than 20,000 atmospheres indicated minimal residual strain. Annealing of the impacted samples in air at temperatures from 600 to 1650 C showed that the amount of residual strain decreased after one hour at 1200 C and was completely absent after one hour at 1650 C. Annealing for one hour at 1650 C did not appreciably alter the grain size of the impacted material.

Surface preparation of the samples prior to line broadening measurements markedly affected the final results; the strain produced from mechanical polishing overshadowed residual strain from pneumatic impaction. Chemical etching with a solution of 50 parts HF - 50 parts HNO₃ at 90 C was only partially effective in removing surface strain from polishing, and even after 2-3 minutes of etching, some strain attributed to polishing was still present. Fortunately, many pieces of the impacted material contained flat fracture surfaces, which could be examined in the x-ray diffractometer without any additional surface preparation. In addition, lamellar pieces were formed from fractures normal to the impaction direction; consequently, sample orientation, with respect to impaction direction, was maintained constant from sample to sample.

Recovery and Recrystallization in Pneumatically-Impacted UO₂

The occurrence of recrystallization in annealed samples of impacted UO₂ (-100 +150 mesh; 600, 1000, and 1200 C; and 340,000 psi) was verified. A one-hour vacuum anneal at 1200 C produced no observable recrystallization, but a one-hour anneal at 1400 C produced significant recrystallization of the large unfractured grains. Recrystallization was most evident in the impacted material at 1200 C, which had apparently undergone more plastic deformation.

Three molybdenum cans of ceramic grade UO₂ were impacted at 330,000 - 340,000 psi and 1500, 1600, and 1680 C to study the high temperature impaction behavior. After impaction, the 1500 C can was furnace-cooled at 150 C/hr,

and the other two cans were air cooled. Visual examination of sectioned cans showed no laminations, but a random crack pattern was present which limited the longest dimension of coherent sample to less than 1/2 inch. Metallographic examination revealed that all three materials had oxidized either during pre-impaction heat treatment or subsequent to impaction. Since relatively low pre-impaction pressures were obtained, oxidation probably resulted from fracture of the cans. Material impacted at 1680 C was essentially pore-free but had undergone significant grain growth.

Convection•Heat Transfer in Molten-Center Oxide Fuels

Effects of natural convection on the thermal profile in horizontally-irradiated, molten-center UO_2 fuel cores were deduced. Cross sections of ten, 1.375-inch diameter UO_{2+x} specimens, irradiated horizontally at nominally 30 kw/ft, showed consistent upward displacements of the molten region. Since similar specimens irradiated without center melting showed no structural asymmetries, the effects were attributed to net heat transfer to the top of the molten zone by natural convection. Asymmetries were quantitatively described by measuring the displacements and converting them to incremental integrated thermal-conductivity values. From these, quantities analogous to convection coefficients were computed and correlated with diameters of the molten zone. The resulting relationships permit a prediction of expected thermal-profile asymmetry in a horizontally-irradiated molten-center fuel rod.

Computed virtual convection coefficient values were constant at $0.07 \text{ w/cm}^2\text{-}^\circ\text{C}$ for melt radii greater than about 0.8 cm. The coefficient increased, as expected, as the size of the molten region decreased, to a value of $0.2 \text{ w/cm}^2\text{-}^\circ\text{C}$ for a melt radius of 0.4 cm--the smallest molten zone observed. These coefficients were employed in the conventional way to calculate a temperature differential across the solid-liquid interface. This ΔT (which is not real) must be used in conjunction with thermal conductivity data for the solid fuel to compute an upward displacement of the molten zone. Because of the artificial nature of these computed ΔT 's, coefficients deduced as discussed above are not conventional coefficients and are referred to as "virtual" coefficients.

The results are useful in anticipating thermal asymmetries in molten-center fuels. Such asymmetries are important for design because they can give rise to fuel rod warping which may have catastrophic effects in an operating reactor core.

Actinide-Oxide Phase Studies

Oxygen vapor pressure measurements over hyperstoichiometric plutonium-uranium mixed oxides (20% PuO_2) were continued. Previous results showed that equilibrium in this system was established very slowly in the single-phase MO_{2+x} region at 1100 C. Experiments during the past month at 400 C

with the chemical potential of oxygen in the gas phase varying between -14.2 and -22.5 kcal resulted in oxygen pressures sufficiently large to cause loss of a metal-bearing species, presumably according to a reaction such as

where x varied between 0.05 and 0.10.

In other experiments it was found that mixed UO_2 - 20% PuO_2 was retained stoichiometric at temperatures to 1400 C by heating in one atmosphere of He - 60 mm H_2 passed over $O^\circ C H_2O$. Utilizing these conditions, other property measurements can be readily performed on completely stoichiometric mixed oxides.

Eight UO_2 - PuO_2 samples extending from 12 to 100 mole% PuO_2 were melted in an Ar - 8% H_2 atmosphere and quenched to room temperature. X-ray diffraction of these samples indicated an increased lattice constant relative to the premelted values. The data were in excellent agreement with similar results obtained several years ago and are indicative of hypostoichiometric solutions. The linear lattice constant-composition plot suggests an oxygen loss such that the melted compositions can be considered a solution of $PuO_{1.62}$ in UO_2 . The average plutonium valence in such a solution is 3.24 and may explain why the minimum O/M calculated on the basis of a plutonium valence of +3 is not observed in oxygen deficient (U,Pu) O_2 solutions. For example, one would expect UO_2 - 20% PuO_2 to be reduced to $MO_{1.90}$ if all the plutonium were +3. However, the lowest O/M observed for this composition is 1.92, in good agreement with a plutonium valence of 3.24, though the lattice constant found for a hydrogen-reduced composition of $(U_{0.8}, Pu_{0.2})O_{1.92}$ is larger than observed for the same composition melted. If the minimum O/M is reached during melting, these values should, of course, be identical. The cause of this anomaly is under investigation.

Chemical Thinning of Pneumatically-Impacted UO_2 for Electron Transmission

Uranium dioxides, with O/U ratios between 1.90 and 2.15, were chemically thinned and examined by transmission electron microscopy. Apparent dislocation densities were higher in pneumatically-impacted material than in sintered material.

Pu-O-N Phase Equilibria

An evaporator for depositing compositions corresponding to isothermal sections of ternary phase diagrams was designed for use in studying phase equilibria in the Pu-O-N system. The apparatus consists of a self-resistance heated platinum substrate, a resistance-heated evaporator for the plutonium, and two small metering valves for control of the oxygen and nitrogen flows,

all of which are contained in a 200 l/sec vacuum system. Following evaporation, the platinum substrate will be sectioned and the condensate analyzed by means of microprobe, neutron activation analysis, and gas analysis.

High Vacuum Pneumatic Impaction Process

The remainder of the parts necessary to set up the 200 l/sec ion pumping system for high vacuum pneumatic impaction arrived and were assembled. The system is presently undergoing a high vacuum bakeout which will continue until a pressure of approximately 10^{-10} torr is achieved. A series of trial pinch-offs will be made on a molybdenum tube to verify our ability to hermetically seal the tubing before actually pumping on a sample for pneumatic impaction.

Materials and Information Exchange

UO₂ specimens were prepared by centerless grinding and ultrasonic drilling to form hollow pellets (31.7 x 5.97 mm OD x 3.43 mm ID) for use in basic studies of in-pile thermal conductivity at ORNL. These fine-grained specimens will be used to yield data to be compared with the results of the UO₂ single crystal specimens that were supplied earlier.

UO₂ (4 wt%)-ThO₂ pellets were prepared by pressing and sintering techniques for use in infrared studies by Bell Telephone Laboratories.

To meet local requests for materials, arc fused UO₂, UO₂-ThO₂, and ThO₂ pellets were prepared, characterized, and sent to Chemistry Department for use in dissolution and decontamination studies.

In addition, the Japan/US Research Newsletter No. 5 (about ceramic nuclear fuels) was received, reproduced, and distributed to the operating agencies under terms of the cooperative US/Japan agreement.

Nuclear Graphite (R. E. Nightingale)

High Temperature Irradiations

The high temperature capsule GEH-13-12 was installed in the ETR in Cycle 85. The capsule met disaster, however, even before reactor startup. During routine control rod checks, a rod in the vicinity of the capsule hung up on its surrounding guide tube while being withdrawn and carried the guide tube with it. The guide tube struck the lead tube on the capsule and forced it up, lifting the capsule out of the reactor and kinking the lead tube. When the capsule was pulled out of the reactor to check the extent of the damage, the tube broke at the kink and water entered the

capsule. The capsule was immediately cut open and the graphite samples retrieved and dried. It appears that the samples suffered no damage. Efforts are under way to reconstruct the capsule in time for the next cycle, scheduled to begin December 19. The sample loading will be identical to that of GEH-13-12.

It is not yet known why the control rod jammed in its guide tube. However, precautions will be taken in the future to insure that such an incident will not occur. The tube bending schedule will assure that the lead tubing of the capsule never passes over a control rod position.

EBR-II Irradiations

The first graphite pin BG-1 irradiated in EBR-II was disassembled in a TAN hot cell at NRTS. The operation went smoothly, and the samples were retrieved in apparently good condition. The only questionable samples were those two surrounding the sample spacer containing a Tempil pellet to monitor the temperature. Some oxidation appeared on the sample ends where the pellet material evidently reacted with the graphite. Consequently, any future use of the pellet will be avoided.

The two remaining graphite pins in EBR-II are scheduled to be discharged in approximately six months. Exposure on the graphite at this point will be over 10^{22} nvt.

Irradiation of Nuclear Graphite

GETR has solved the reactor equipment problems which had limited the reactor power level to 45 MW. As a result, the H-3-13 graphite irradiation capsule is now operating at 50 MW. The samples are duplicating their previous temperature history except for three sample positions which are currently about 100 C higher than past temperatures. The H-3-13 capsule is scheduled out of reactor late in December with a maximum accumulated exposure of approximately 2.4×10^{22} nvt ($E > 0.18$ MeV).

Irradiations of "Dimensionally-Stable" Graphite

The H-3-24 capsule was charged in the E-5 position of the GETR during an outage in Cycle 80. The capsule went to temperature late in October. Although the capsule was designed to reach temperature at the previous reactor power level of 45 MW, the gas control system allows adequate temperature control at the current 50 MW. Of a total 13 sample positions, eight are at design temperature, and five are within 100 C of their previous temperature.

One of the nine thermocouples, unfortunately the only 5% W - 26% Rh one, was lost during startup.

Hanford Hot Test Holes

Temperature-of-Graphitization Series. A series of needle-coke graphites having four different graphitization temperatures of 2400, 2600, 2800, and 3000 C is being irradiated. A previous series explored the graphitization range up to 2570 C. Unlike the lower temperature series, this series shows a marked decrease in coefficient of thermal expansion with increasing graphitization temperature. However, the irradiation behavior of both series is consistent, i.e., decreasing contraction rate with increasing graphitization temperature.

The samples were irradiated in a Hanford hot test hole, at a temperature of 650 ± 100 C, to an exposure of 5.5×10^{20} nvt ($E > 0.18$ MeV). The transverse orientations contracted 0.104 to 0.206% for the 3000 C to 2400 C samples, respectively. Parallel samples contracted 0.156 to 0.316% for the 3000 C to 2400 C samples, respectively. The samples will not be recharged in a Hanford hot test hole; however, the two extremes (2400 C and 3000 C) are included in a GETR capsule.

Effects of Additives on Graphite Contraction

One series of additive samples, Speer contract DDR-136, has been discharged after the second irradiation in a Hanford hot test hole at 650 ± 100 C to an accumulative exposure of 9.3×10^{20} nvt ($E > 0.18$ MeV).

In the first set (GLC needle coke and coal-tar-pitch binder) all parallel samples, including the standard, contracted from 0.06 to 0.08%. The transverse samples all show a positive total length change of 0.005 to 0.030% except the standard which contracted 0.016%. However, all transverse samples except those in which 5% aluminum was added, contracted during the second exposure period. Transverse samples with aluminum additive continued to grow, and the contraction rate for the parallel samples was the lowest of any samples during the second exposure period.

A second series of samples manufactured from fluid coke and coal-tar-pitch binder, was not remeasured due to the high contraction during the first exposure.

A third series manufactured from GLC needle coke and Resonox 497 binder, has a total parallel contraction ranging from 0.045 to 0.065% for all samples including the standard. Of the transverse samples, only the standard experienced a contraction rate for the second exposure. The 5% Fe_2O_3 has a continued expansion to +0.048% and the 5% Ni_2O_3 has a continued expansion to +0.019%.

These series of samples will not be recharged in the Hanford test holes. However, the standard for set one, both 5% aluminum groups, the standard for set three, the 5% Fe_2O_3 , and 5% Ni_2O_3 will be irradiated to a higher exposure in GETR as capsule space permits.

Thermal Expansion of Graphite

The thermal expansion of a single-phase polycrystalline aggregate can be described by the general equation:

$$\alpha_x = (1-K_x) \gamma (\alpha_c - \alpha_a) + \alpha_a \quad (1)$$

where α_x is the thermal expansion coefficient of the aggregate in the x direction, K is a constant defined by the orientation function of the crystallites, γ is the c-axis "accommodation factor," and α_c and α_a are the lattice expansion coefficients of a single crystal (see BNW-SA-824).

Analysis of thermal expansion data on nuclear grade graphites has shown that the relationship is obeyed quite well over the temperature range 100 to 1000 C; above 1000 C, this relationship predicts a value for the thermal expansion behavior of other materials, such as lampblack carbon and glassy carbon, which are not polycrystalline single-phase aggregates, obey the single-phase equation. This suggests that the expansion coefficients of non-graphitic carbon could be approximated by a linear combination of the lattice-expansion coefficients of graphite crystallites.

Deformation Mechanisms in Polycrystalline Graphite

If the method used by Buessem [Mechanical Properties of Engineering Ceramics (Interscience New York, 1961) p. 127] to introduce accommodation in anisotropic ceramics is applied to graphite, the large anisotropy of thermal expansion in conjunction with the large temperature changes involved in the cooldown from graphitization imply that, for reasonable values of the assumed critical strain, at least 90% of the supporting material will fail. This suggests that either the means of accounting for accommodation is not realistic or that the shear mode of deformation is dominant. In an effort to derive a more realistic model, the restraint of constant strain on the columns was relaxed so that only the adjacent parts of next nearest neighbors were involved in the accommodation. Such an accommodation mechanism has been introduced in a treatment of the averaging techniques for thermal expansion (see March report). This treatment yields values for the thermal expansion coefficients and the Young's Modulus in very good agreement with the experimental values.

Oxidation of Boronated Graphite

When boronated graphite with no previous oxidation history reacts with 1% O₂ in helium, the rate of carbon loss calculated from the concentration of CO₂ produced is greater than that obtained by direct weighing because of the buildup of B₂O₃ in the sample. However, if the B₂O₃ is then removed by leaching, a general increase in the rate occurs and the weight-loss rate exceeds that calculated from the concentration of effluent CO₂. This anomalous behavior was tentatively related to the measurement of the CO₂ concentration (August 1966 monthly report).

Measurements of the response of the chromatograph to the concentration of CO_2 have disclosed it to be very nonlinear. For example, a He - CO_2 mixture containing 2000 ppm CO_2 when compared to the standard mixture ordinarily employed (530 ppm CO_2) gave a response indicative of only about 1120 ppm CO_2 or only about 56% of that actually present. The nonlinearity of the chromatograph to CO_2 readily explains the anomalous results obtained in the oxidation of leached boronated graphite.

The CO_2 concentrations and oxidation rates of a series of three runs which showed the low CO_2 concentration were corrected and then integrated to obtain the weight of carbon lost by the sample. The results of these calculations are given in the following table.

Run No.	Weight Loss of Sample mg	$\text{W}_{\text{B}_2\text{O}_3}$ mg	Actual ΔW_C mg	Calc. ΔW_C , mg (linear response)	Calc. ΔW_C , mg (nonlinear response)
1	-10.4	4.5	-13.5	-12.8	-13.5
2	-17.8	6.9	-22.6	-17.9	-22.0
3	-24.0	8.9	-30.1	-18.0	-26.0

In the table, $\Delta\text{W}_{\text{B}_2\text{O}_3}$ is the weight of B_2O_3 formed during the run as obtained by subsequent leaching, the actual ΔW_C is the weight of carbon lost by the sample as obtained by correcting the apparent loss for the amount of oxygen in the B_2O_3 , and the calculated values of ΔW_C are obtained by graphical integration of the rate calculated from the concentration of CO_2 in the effluent gas. The calculated ΔW_C of Column 5 is based on the assumption of a linear chromatographic response to CO_2 and is in generally poor agreement with the actual ΔW_C of Column 4. The last column gives the recalculated values of ΔW_C which are seen to be in good agreement with the actual values for the first two runs. The agreement is less satisfactory for the last run where the rate, and hence the CO_2 concentration, were appreciably higher, but it is considerably better than that obtained assuming a linear response of the chromatograph to the CO_2 concentration. The uncertainty in the CO_2 concentration is considerable at the high concentration due to the comparatively low sensitivity of the chromatograph to the higher CO_2 concentrations.

Graphite Surface Area Measurements

To investigate further the accuracy of BET surface area determinations on small samples of graphite, two separate measurements were performed on a standard sample (0.426-inch diameter and 4 inches long) of TSX graphite. The sample was then machined to the size (0.25-inch diameter and 1.75-inch long) to be irradiated in capsule GEH-13-12, and two additional surface area measurements made. The results of these measurements are presented in the following table where it is seen that the agreement among all area values is very good. In addition to values of the surface area, approximate values

for the average heat of adsorption can be obtained from the BET equation. These values are included in the table. The reason for the difference for the two sample sizes is not known.

Run No.	Total Surface Area, m ²	V _m cm ³ STP/g	Specific Surface Area, m ² /g	ΔH _{ads} , kcal/mole
1	7.845	0.1086	0.476	2.41
2	7.918	0.1096	0.480	2.44
3	1.124	0.1075	0.471	2.13
4	1.135	0.1085	0.475	2.17

Irradiation Damage to Reactor Metals (A. L. Bement)

Alloy Selection

The purpose of this program is to study, on a limited basis, materials which are potentially useful for reactor application. From such cursory examinations, the more promising alloys can be selected for study in depth. In addition, program alloys are procured, warehoused at BNW, and dispersed to the various sites participating in the Irradiation Effects on Reactor Structural Materials Program.

Nickel-base alloys are candidate backup materials for the LMFBR Program. A group of alloys, Inconel 600, Inconel X-750, Incoloy 800, Hastelloy X-280, Inconel 625, Inconel 702, and Inconel 718, will be included in the proposed 0.6- and 3-year EBR-II irradiations. Tensile and creep-rupture studies are planned at two irradiation and two test temperatures.

In addition, the alloys will be exposed to the sodium and fast flux environment in both a standard and aged condition. The particular aging treatment for each alloy was selected in light of current thinking, which predicts enhanced postirradiation mechanical properties from tailored pre-irradiation microstructures. Additional EBR-II irradiations of nickel-base, cobalt-base, vanadium-base, and other refractory alloys over a two-year duration are planned. Two heats of Hastelloy-X with varying boron content will be irradiated during the 0.6-year EBR-II irradiation to compare boron effects in a fast (EBR-II) flux with those in a high thermal component (ETR) flux.

An order for experimental heats of FV-548 stainless steel and an Fe-Cr-Al-Y alloy has been placed with the Allegheny Ludlum Research Laboratory. The alloy heats will be poured and fabricated on a best-effort basis, and the chemistry and fabrication history will be documented.

In-Reactor Measurements of Mechanical Properties

The purpose of the in-reactor measurements program is to determine the effects of irradiation on the mechanical properties of reactor structural materials. The effort is currently involved in measuring creep in AISI 304 stainless steel during neutron irradiation.

The last in a series of in-reactor tests was charged in the reactor and is currently accumulating exposure prior to stressing at 840 C and 5000 psi. The micropositioner on this capsule malfunctioned just after reactor startup. However, the heater and thermocouple and stressing systems are intact so that a stress-rupture test may yet be conducted.

The high temperature capsule was sent to the reactor for charging but could not be installed due to a poorly fitting nozzle cap. The capsule was returned for nozzle cap modification and will be returned to the reactor for charging at the earliest opportunity.

Irradiation Effects in Structural Materials

Stainless Steels. Transmission electron microscope examinations have been performed on thin sections from both the buttonhead and gage portions of an in-reactor creep specimen tested to rupture at 750 C. A fine precipitate was apparent in the microstructure of both the as-irradiated gage and buttonhead sections. The buttonhead contained the higher concentration of precipitates. Annealing at 1200 C for one hour eliminated the matrix precipitate, but some larger precipitates were found in the grain boundaries. Examination of a foil annealed for one hour at 1200 C from the gage section of an unirradiated control specimen revealed no precipitation of any type. A positive identification of the precipitates has not been made.

Surface pits at grain boundaries were observed by optical microscopy in irradiated sections that had been annealed at 1200 C for one hour. No such pits were observed in corresponding unirradiated samples. The reason for this observed difference in surface appearance is not known, but further study will be made of irradiated and unirradiated specimens annealed under carefully controlled conditions, e.g., annealing temperature, cooling rate, etc.

An as-irradiated thin section from the buttonhead of an in-reactor creep sample tested at 650 C has also been examined in the electron microscope. Fine precipitates similar to the type seen in the 750 C creep sample as well as large carbides exhibiting moire fringes were observed.

Tensile specimens of annealed AISI 304, 316, and 348 stainless steels are being prepared for assembly in GEH-22-4, the fourth in a series of high temperature, liquid metal capsules being irradiated in the ETR. The capsule is designed to operate at a temperature of about 1400 F (760 C).

A series of Tempil melt pellets, representing 13 different temperatures from 750 to 1250 F (400 to 675 C) (5 pellets at each temperature), has been irradiated at 60 C in the MTR to an exposure of about 1.31×10^{20} fluence. Of the 65 original pellets, 37 have been recovered for experimental testing. These pellets have been individually encapsulated in quartz ampules and are presently being tested to determine the effect of irradiation on their melting points.

Nickel-Base Alloys. The purpose of this program is to determine the effects of modified microstructures on the irradiation stability of nickel-base alloys. Microstructural modifications are made by preirradiation thermal or thermomechanical treatments and are evaluated by tensile tests, stress-rupture tests, and microstructural examinations.

A paper by I. S. Levy, entitled "Postirradiation Mechanical Property Improvements in Hastelloy X-280 and Inconel 600," was presented at the Winter Meeting of the American Nuclear Society in Pittsburgh, Pa., on November 1.

Replica and transmission electron microscopy of tensile specimens of Hastelloy X-280 irradiated at 540 F (280 C) to 1×10^{20} nvt (>1 Mev) and tested at room temperature are continuing. Last month it was reported that no resolvable defects were found in an experimentally-pretreated specimen, whereas a specimen given a standard solution treatment exhibited a cluster-defect structure after irradiation. This month these specimens were annealed at 500 C for two hours, and microscopic examination revealed several features of interest. First, the standard-treated material, which showed the cluster defects in the as-irradiated condition, revealed no appreciable change in size or density of defects after the anneal. Presumably, these defects have been stabilized by impurity atoms such as carbon. Earlier work showed that these defects are removed after about one-half hour of annealing at 735 C. Secondly, the experimentally-treated material, which showed no defects in the as-irradiated condition, revealed extensive precipitation of rod-shaped particles after annealing identified by electron diffraction as M_6C ($a_0 = 10.9 \text{ \AA}$). This precipitate is considered to be the end product of an agglomerate of both impurity clusters produced by the pretreatment and defects produced during irradiation. These agglomerates nucleate as precipitates during the two-hour anneal at 500 C. This precipitation is thought to require strain as well as thermal activation (though this is being checked). Although this form of precipitation was not observed in an unirradiated specimen tested at 732 C, it was observed in the strained section of an irradiated specimen.

Phase analysis of Inconel 600 and Inconel X-750 specimens is continuing. Last month it was reported that a change in carbide phase occurred during tensile testing at 1350 F (730 C) of Inconel 600 and that metallography revealed an apparent reduction in carbide precipitate volume in both the grain boundaries and matrix of the strained section relative to the unstrained section. Further, examination of replica micrographs revealed a "ghost"

grain boundary, one that appeared to be the site of former carbide precipitation. Since this "ghost" grain boundary is in close proximity to a well-defined boundary containing small carbide particles, a concurrent mechanism of boundary migration and diffusion of carbide constituents is indicated.

Stress-rupture tests on 11 Hastelloy X-280 control specimens are nearing completion.

Zirconium Alloys - In-Flux Oxidation. Three lots (AT, CT, and HT) of N-Reactor Zircaloy-2 tensile specimens were exposed in the G-7 loop of the ETR. Accelerated in-flux oxidation occurred on all three lots. However, one lot showed higher weight gains than the other two lots after the in-flux oxidation progressed beyond 150 mg/dm². The differences in in-reactor behavior of the lots did not appear to be related to compositional differences, but differences in fabrication did offer a potential explanation. Lot CT, from which specimens having the highest in-flux weight gains were prepared, was extruded at 840 C, close to the $\alpha + \beta$ transus. This same lot had the lowest in-flux hydrogen pickup. The combination of high weight gain and low hydrogen pickup is characteristic of out-of-flux behavior on Zircaloy-2 heat treated in the $\alpha + \beta$ region as reported by Kass (WAPD-T-1549). If the in-flux behavior resulted from heat treatment effects, differences among the three lots should be observed out-of-reactor in high temperature steam. Accordingly, five tensile specimens from each lot were exposed out-of-flux at 390 C in 1500 psi steam. Coupons from an HT tube section also were exposed in the test. The coupons were prepared by rolling a section of the tube wall, followed by an alpha anneal (8 hrs at 760 C) of the rolled plate.

All three lots of Zircaloy-2 tensile specimens underwent premature breakaway corrosion. After 160 days, weight gains on the tensile specimens were more than double the weight gain on the rolled plate. However, the lot with the highest in-reactor weight gain had the lowest weight gain in the out-of-reactor test in 390 C steam. The range of weight gains for each of the lots in the out-of-reactor steam test are shown in the table.

<u>Lot</u>	<u>Weight Gain</u> <u>mg/dm²</u>
AT	147 - 160
HT ^a	164 - 174
CT	120 - 134
HT ^b	70

^a Machined tensile specimens

^b Rolled plate.

The results offer substantial evidence that the difference in in-flux behavior was not due to heat treatment in the $\alpha + \beta$ region during extrusion. Differences in the microstructures of the tensile specimens and rolled plate from lot HT offer a possible explanation for the differences in corrosion properties. The microstructure of the tensile specimens is characterized by swirls of coarse second phase stringers, while the rolled plate shows the fine distribution of second phase which is characteristic of corrosion resistant Zircaloy-2.

Flux Dosimetry. The Irradiation Standards and Procedures Committee of the Irradiation Effects on Reactor Structural Materials Program has recommended that another intercalibration test be initiated of counting laboratories utilized by program members. The purpose of this intercalibration test is to insure that all participating counting laboratories are consistent in both their determination of monitor activities and conversion of these activities to fluence.

This test will be conducted in a manner similar to the previous intercalibration test (see HW-77052). Two monitors of each type (Fe, Ni, Ti, and Al - 0.1% Co) will be irradiated, counted, and distributed to the participating laboratories by Pacific Northwest Laboratory. Each laboratory will determine the activity of the individual monitors, calculate the fluence, and return the data to PNL for tabulation and comparison.

To date, seven laboratories have agreed to participate, and the irradiated monitor materials are scheduled for shipment to them in January 1967.

Fracture Mechanics

As discussed in the previous monthly progress report, efforts are being directed toward establishing size limitations on the DCB specimen for measuring plane strain fracture toughness of low-strength, high-toughness materials. In connection with these efforts, DCB specimens having a gross thickness, B, of 3/8-inch and made of A302-B pressure vessel steel were tested during the month. The resulting fracture toughness values were in excellent agreement with similar values obtained from 1/4- and 1/2-inch thick specimens of the same material. In addition, gross plastic deformation prior to fracture of the 3/8-inch thick specimens occurred at a β (discussed in previous report) of 0.6 which is also in agreement with results from the 1/4- and 1/2-inch specimens. The lack of dependence of the test results on specimen size lends support to the contention that a relatively small DCB specimen can provide plane strain fracture toughness data. Further testing on 1-inch thick and 3-inch thick specimens remains to be completed.

The effects of warm pre-stressing and strain aging on the toughness of A302-B have also been examined. Warm pre-stressing was accomplished by loading the specimens at -80 C to a high stress intensity level without cracking the specimen. The toughness was then measured at -160 C and was

found to be nearly double the normal value or about equal to the maximum stress intensity attained at the higher temperature. Thus, the plastic blunting of the crack tip which occurred during the pre-stressing resulted in a significant increase in the toughness. Warm pre-stressing of reactor pressure vessels has been advocated as a safety precaution by some USAEA scientists. Unfortunately, aging was found to occur at 50 C which causes a loss of the high toughness induced by the pre-stressing.

Fast Reactor Supporting Studies

Structural Material and Fuel Cladding Studies. The objective of this program is to determine the combined effects of environment and fast reactor irradiation on the mechanical properties of candidate fast reactor structural and cladding materials. The program currently in progress is specifically directed at providing a basis for selection of optimum alloys for FFTF application and at providing a description of material behavior in fast reactor service.

Four MK-A-19 pins have been discharged from the EBR-II and will be available for testing in January 1967. These pins contain 18 AISI 304 stainless steel tensile and 10 creep specimens which have been irradiated at reactor ambient temperatures [800 F (425 C)] to 2×10^{22} total fluence in a helium atmosphere. Testing of these specimens will be directed at determining the effects of test temperature [room temperature to 1500 F (815 C)] and strain rate on tensile and creep properties. The results will be compared to thermal reactor data to aid in establishing the thermal-fast reactor correlations needed to fully utilize the large body of thermal reactor data in a fast reactor technology.

Irradiations currently in progress include 8 MK-A pins containing 50 flat specimens (for correlation with flat specimens being irradiated in the ETR on the structural materials program), 14 round tensile specimens, and 14 round creep specimens. A MK-B-19 pin containing 198 subsize tensile specimens is being irradiated to a target dose of 1×10^{23} . This pin contains a helium environment and is being irradiated at reactor ambient temperatures.

In addition, 24 MK-A pins containing 168 tensile and 48 creep specimens are at the EBR site awaiting insertion on a space-available basis. A set of 12 MK-A pins are also awaiting charging on a space-available basis. Six of these pins are designed to provide contact between the tensile and creep specimens and reactor sodium; the other six provide a helium environment. Subsequent testing of specimens from pins inserted as pairs will allow the combined effects of reactor sodium and neutron irradiation to be evaluated. The 12 pins contain 96 tensile and 36 creep specimens of AISI 304 and 316 in equal numbers as well as a number of wafers for helium and hydrogen analysis.

Two full subassemblies having the MK-B-7 configuration are being prepared for insertion into the EBR-II in January. One subassembly scheduled for

irradiation to 1×10^{23} fluence is to be placed in the reactor core; the other will be placed in Row 7 and is scheduled for 1×10^{22} fluence. Each subassembly contains a series of sodium-filled gamma-heated subcapsules designed to operate at three temperatures; namely, 1400, 1200, and 900 F (760, 650, and 480 C). Both units will contain mostly 304 and 316 tensile, biaxial creep, and uniaxial creep specimens; but backup FFTF alloys such as 348 and 321 are also included, as well as Hastelloy X-280, Inconel 600, Incoloy 800, Inconel 718, Inconel 702, Inconel 625, and Inconel X-750.

Apparatus for biaxial creep testing irradiated specimens and controls in sodium is being evaluated. The proposed biaxial specimen and stressing system was evaluated in part by conducting 10 biaxial creep tests on AISI 304, all at the same stress level (10,000 psi) and temperature [1400 F (760 C)]. The results showed that the average rupture time obtained was consistent with information from other laboratories. Data scatter among the various specimens was about 20%.

Liquid Metal Capsule Irradiations. Capsule GEH-22-3, containing convecting NaK coolant, has begun its third irradiation cycle in the ETR. A temperature record of the capsule is still being maintained by means of one thermocouple. This couple indicates a central nominal sodium temperature of 1110 F (600 C), the equilibrium temperature throughout past cycles at this capsule location. The capsule will be discharged at the end of the cycle during which all communication with the capsule is lost.

A liquid-sodium capsule, described in the previous monthly report, is in final assembly. This capsule, containing stainless steel alloys, will replace GEH-22-3 currently being irradiated. Design modifications are incorporated into the new (GEH-22-4) capsule to permit a sodium temperature to 1400 F (760 C) and to improve over-all capsule life.

Test Facilities. Work has been started on the general laboratory support section of the Creep and Creep-Rupture Testing Facility. Construction has also been started on a prototype, remote cell for creep-rupture studies of irradiated specimens. Both of these activities are expected to be completed by January 1967. Specific requirements have been determined for an inert gas glove box which will be needed for loading sodium into test capsules. Market analysis indicates that a commercial unit may satisfy these requirements. The shielded test portion of the liquid sodium loop is in the detail design stage.

Fast Neutron Mechanisms. A series of hydrogen permeation experiments has been initiated using the recently completed gas permeation apparatus. The preliminary studies, with plain carbon steel as the permeable material, will serve both to characterize the integrated permeation apparatus and to experimentally evaluate the approaches that have been developed to control potential rate-determining phase boundary processes at the hydrogen absorption

surface. The subsequent studies in the series will explore the effect of internal boundary conditions and external desorption environment on hydrogen diffusion and effusion behavior in candidate iron and nickel-base fast reactor alloys. The permeation results obtained thus far are particularly encouraging inasmuch as the concentric tube design of the permeation cell appears to be completely effective in preventing gas leakage into the gas detection system, even with a faulted seal assembly. For certain hydrogen-defect interaction studies, it will be necessary to conduct permeation experiments at or below room temperature. A modification of the permeation cell gasket system has been developed so that electrolytic rather than gas phase hydrogen charging techniques can be used as required for this lower temperature range.

In previous work it was determined that the direct effect of an imposed temperature gradient on neutronic hydrogen concentration profiles in reactor fuel cladding should be quite small. There is, however, a possible indirect effect of a temperature gradient on neutronic hydrogen and helium behavior that merits consideration. Gross changes in the concentration and distribution of carbides, nitrides, and oxides can occur in alloys in response to a severe temperature gradient, and these changes are most pronounced at external surfaces and internal boundaries where the effect on hydrogen and helium behavior is potentially most critical. Unfortunately, there are not sufficient data on which to base an estimate of the possible extent or kinetics of such a solute redistribution; a temperature gradient cell has, therefore, been constructed and some tests started to evaluate the magnitude of this problem for the various alloy systems of interest.

ATR Gas Loop Supporting Studies

ATR and Model Loop Corrosion Surveillance. Hastelloy X-280, Haynes 25, molybdenum, niobium, tantalum, and tungsten corrosion coupons have been exposed to one "startup" of the ATR model gas loop. This test is the first of a series to determine the amount of contamination occurring in loop components and experimental materials which have been exposed to the atmosphere before startup of the loop system. One set of coupons positioned in the 2-inch diameter heater discharge section was at an average temperature of 2100 F (1150 C). A second set of coupons, downstream in the 4-inch diameter section, was at 2025 F (1105 C). The contaminant level of the helium gas was less than 1 ppm during the run. Weight changes and visual inspection of the coupons after 59 hours at temperature showed a loss of weight of the Haynes 25 and Hastelloy X-280 samples due to an evaporation mechanism, a gain of weight for the niobium and tantalum samples, and essentially no weight change for the molybdenum and tungsten coupons.

Comparing the evaporation tendency of Hastelloy X-280 in different environments, a weight loss of about 8.6 mg/cm²/day was observed in a 10⁻⁴ Torr dynamic vacuum at 2100 F (1150 C), a weight loss of 0.52 mg/cm²/day was observed in flowing helium at 2050 F (1120 C) for an average helium velocity of 196 ft/sec

at 300 psi, and finally, a weight loss of $0.32 \text{ mg/cm}^2/\text{day}$ was observed at 2100 F (1150 C) for the corrosion coupons in the model loop. The coupons have been reassembled in the sample holder and placed in the loop for additional cycling.

Test Assembly. Exposure of the refractory metal test assembly in the model loop now totals approximately 775 hours at a nominal temperature of 1700 F (925 C). Dimensional stability of the assembly is still good, and there is no evidence of bonding between specimens and the holder. Almost all specimens now show surface discoloration. Additional experiments in which the temperature distribution within and the pressure drop across the assembly will be measured as a function of helium temperature and flow rate are planned. The assembly will then be ready for dismantling to determine the mechanical properties of the refractory metal specimens.

The creep test of the tapered head Hastelloy X specimen was terminated after 573 hours at 1400 F (760 C) and 15,000 psi stress. The specimen has undergone only a few percent deformation; detailed dimensional measurements of the specimen to evaluate possible deformation in the tapered head are not yet completed. A second creep test at 1400 F (760 C) and 25,000 psi has been started to examine the effect of a higher stress on specimen deformation.

A nickel-base ATR prototype assembly is being fabricated. The container material will be Hastelloy-X. Three tiers will contain high (Inconel 600), intermediate (Hastelloy-X), and low (Incoloy 800) nickel alloys to determine performance of the nickel group to the model gas loop environment.

High-Temperature Fluence Monitors. The high-temperature conditions expected in the ATR gas loop require that fluence monitors be able to withstand temperatures in excess of 1000 C. In order to provide monitors that will not melt, corrode, or become easily contaminated by their environments, a program has been initiated to test available monitor materials and composites. Monitors composed of metallic oxides of iron, nickel, and cobalt dispersed in an aluminum-oxide diluent and presently used by the Dragon Project in England are being studied. Information recently received indicates that these monitors decompose above 1200 C. Consequently, additional emphasis has been placed on the development of a more stable type of composite.

Prior to the receipt of the above information, a program to develop a better quality monitor had been started. The basic concept of a metal oxide in a high-temperature diluent has been followed. The initial investigation employs a NiO-MgO solid solution that should remain stable up to 2400 C. Personnel in the Ceramics and Composites Development Unit will make 100 pellets of the NiO-MgO material. Irradiation and temperature-stability tests are scheduled to start by mid-December. When the irradiation and high-temperature stability of this system has been determined, MgO pellets containing iron and cobalt oxides will be fabricated and tested in a similar manner.

Damage Mechanisms

The objective of this program is to determine how irradiation-induced defects interact with moving dislocations to modify the mechanism of plastic deformation in a metal. Radiation damage and the role of interstitial impurities in alpha iron on this damage are currently being investigated.

During this period, Ferrovac E and BMI iron samples irradiated to a fluence of 5×10^{19} nvt ($E > 1$ Mev) were tested and the data initially evaluated. The following prominent features were noted: The tendency for deformation to be localized, noted in earlier samples irradiated to 5×10^{18} nvt, is accentuated. This is accompanied by a decreased ductility in these samples and a more rapid increase in activation volume with strain. This tendency makes it difficult to determine a value of activation volume which is characteristic of the irradiated sample, especially at lower temperatures and as measured from temperature change tests. The general shape and magnitude of the activation volume curves as a function of strain duplicate the lower exposure results and support the conclusion that defects introduced by radiation damage are not controlling deformation in this material. The most significant feature of the tests was the greater increase in shear yield strength ($\sigma/2$) in the Ferrovac E material (5.4 Kg/mm^2) than in the BMI material (3.2 Kg/mm^2). This compares with an increase in yield strength of about 2.0 Kg/mm^2 for both materials after the lower fluence of 5×10^{18} nvt.

Two capsules containing purified iron single crystals were assembled for irradiation in the ETR. These crystals will be used for length change/bend tests. Equipment setup and development of techniques for measuring dislocation multiplication during tensile testing of iron specimens has progressed.

Calibration of ultrasonic equipment, for use in observing dislocation activity in single crystal molybdenum samples during strain tests, was continued. Almost all of the equipment that was on order has now been received.

Cylindrical samples of fused quartz, having a nominal diameter of 0.3 inch, were tested at an ultrasonic frequency of about 10 MHz to determine if signal amplitude changes due to small differences in sample diameter would be troublesome. No signal changes could be observed for a variation in diameter of as much as 0.002 inch.

Actual diameters of metal crystals to be strained will also be about 0.3 inch, and diameter variations during the strain testing are expected to be much less than 0.002 inch. It is therefore believed that ultrasonic signal variations due to this effect will not be a problem.

Procedures for accurately determining signal amplitude changes using the new ultrasonic equipment were also developed during the month. These procedures are being used to calibrate the ultrasonic transmitter, receiver, and oscilloscope readout system.

High Pressure Studies

An internal document, "The Design of Equipment to Generate High Hydrostatic Pressures on Samples Simultaneously Exposed to Nuclear Irradiation," has been written and distributed.

Testing of the ultra-high-pressure/high-temperature "Belt" apparatus has been witnessed at the factory. Design requirements of 60 kbar pressure at 1500 C were met, and the system has been shipped.

Preirradiation resistivity measurements on the molybdenum samples for the isobaric-isochronal annealing experiment have been made at liquid helium temperatures. The samples have been encapsulated and sent to the K-West reactor for irradiation to 5×10^{18} nvt (fast).

The second model of the In-Reactor Hydrostatic Pressure Cell has been constructed and initially calibrated to 6000 atmospheres at 100 C. The feasibility of using gallium as a pressure transmitter in this cell is being tested. The design capability of this device is 18,000-20,000 atmospheres.

ATR Gas Loop Operation and Maintenance (G. A. Last)

ATR Gas Loop Support

Model Gas Loop. The model gas loop is being operated to determine the effect of the helium atmosphere on materials and specimen holders and to establish the effect of prolonged use on the operating characteristics of the new heater. This loop is a one-tenth size model of the gas loop that is being constructed in the Advanced Test Reactor.

The model gas loop has been operated during the past month for 167 hrs which brings the total operating time to 1733 hrs on the new heater. This includes a total of 40 thermal cycles from room temperature to various heater outlet temperatures. The system operated with an outlet temperature of 2100 F for 77% of the time this month. A new corrosion test assembly was installed within the horizontal test zone and has undergone 129-hr operation to date. This specimen assembly was removed and examined twice during the month. The tensile specimen test assembly in the vertical test zone has been in place during this operating period but has not been removed for examination.

The bearings in the variable speed drive unit of the motor generator set failed again after only three weeks of operation. This failure appeared to be caused by a lack of lubrication in the sealed bearings. Modifications were made to allow manual lubrication of these bearings instead of relying on the initial lubricant installed when they were fabricated.

Evaluation of Struther-Wells Heater. The first high temperature tensile tests of molybdenum from the Struther-Wells heating elements have been completed. Preliminary results indicate a yield strength of about 3600 psi and a tensile strength of 7800 psi at 2500 F (1370 C). These values are somewhat lower than reported data on the high temperature strength of molybdenum, but not seriously so. These tests continue to support the validity of the selection of molybdenum as the heating element material for the ATR gas loop heater.

Heater to In-Pile Tube Stainless Steel Piping. Solar is fabricating the internally insulated 304 stainless steel piping which connects the heater to the in-pile tube of the ATR gas loop. Solar proposed a high temperature brazing operation to join the external water cooling coils to the pipe, but the procedure does not include water quenching from the brazing temperature. This raises the question of possible sensitization of the piping and its effect on pipe serviceability and life. IDOO has requested that PNL review this question and submit recommendations on the acceptability of, or necessary modifications to, Solar brazing procedures. This problem is presently under study; final recommendations will be forwarded to IDOO.

Meetings. Meetings were held on October 24, 25, and 26, 1966, in Richland for the purpose of discussing the B&W analog study for loop control, high temperature thermocouples, and certain other areas of mutual interest. Representatives of AEC-IDO, AEC-RL, INC, B&W, Ebasco, and PNL were present. Results of the meetings are summarized as follows:

- Preliminary results of the analog study on control instruments look promising. Although no optimization was attempted, control settings for the five independent control parameters were determined which cause interactions to be quickly damped out.
- Platinum-molybdenum thermocouple material was selected as the candidate thermocouple material for the in-pile thermocouples. B&W is investigating the required materials, connectors, etc., with potential vendors.
- It was determined that PNL could make beneficial use of the transfer facility at the earliest possible date, without regard to loop completion date, to develop procedures and hardware for test handling.
- Purchase of a fifth circulator casing could permit earlier delivery of the required machine, and substantially improve the GCL completion date.
- Difficulties in design and testing of equipment by the vendor of the analytical and transfer facility instrumentation were discussed.

- INC comments on the PNL heater were discussed. Ebasco was to submit them in the normal manner for disposition.
- Outstanding differences concerning alteration of the shielded filter were resolved.

A meeting was held with Calmec Manufacturing Corporation to clarify the Ebasco specifications prior to awarding a purchase order for the large control valves for the ATR gas loop. The meeting was attended by representatives of AEC-IDO, Ebasco, Fluor, and PNL.

After a complete review of all the specifications, an agreement was reached with Calmec and concurrence by all parties present. Some of the items discussed were seat leakage, pressure drop, back seating requirements, and quality control procedures. It was the consensus that Calmec is well equipped to produce and test the valves in their helium to PNL's strict GCL requirements.

Metallic Fuels Development (G. A. Last)

Irradiation of Thorium-Uranium-Zirconium Fuel Elements

The irradiation of six tubular Zircaloy-2 clad thorium - 2.5 wt% uranium - 1.0 wt% zirconium fuel elements continued successfully in the ETR P-7 and M-3 hot water loops. The current conditions for the six elements are summarized in the table.

Fuel No.	<u>Irradiation Conditions for Th-U-Zr Fuel Elements</u>				
	BU Fissions/cm ³ (MWd/T)	Maximum Core Temp. (°C)	Surface Heat Flux Cal/Sec-cm ² (BTU/hr-ft)	Specific Power Watts/gram (Kw/ft)	Fuel Volume Increase (%)
GEH-10-65	5.6 x 10 ²⁰ (16,200)	440	37 (5.0 x 10 ⁵)	34 (102)	2.8
GEH-10-64	3.8 x 10 ²⁰ (11,100)	400	30 (3.9 x 10 ⁵)	26 (80)	1.6
GEH-10-71	2.4 x 10 ²⁰ (6,600)	460	40 (5.4 x 10 ⁵)	37 (113)	1.0
GEH-10-72	1.5 x 10 ²⁰ (4,300)	490	46 (6.2 x 10 ⁵)	43 (130)	0.1
GEH-10-70	0.9 x 10 ²⁰ (3,900)	470	42 (5.6 x 10 ⁵)	39 (119)	0.5
GEH-10-84	0.6 x 10 ²⁰ (2,500)	440	37 (4.8 x 10 ⁵)	33 (101)	0.1

The volume increase of 2.8% for the element having the highest exposure is about that required to accommodate the solid fission product atoms. The three highest exposure elements will continue to be irradiated in the P-7 loop. Those that were in the M-3 loop are being replaced by the high exposure uranium rods.

The bulk outlet temperature of the P-7 loop water was reduced 50 C (90 F) to an operating temperature of 204 C (400 F) in order to reduce the temperature of the cladding. Examination of fuel element GEH-10-66 (1.4 at.% BU) revealed a 1.3-mil thick oxide on the surface of the clad which possibly increased the clad temperature to 350 C due to the insulating nature of the oxide. There is some concern that this increased clad temperature might result in undue clad corrosion which would restrict achieving the desired exposure in the fuel. Thus, the loop temperature was dropped to reduce the clad surface temperature to 300 C which is in keeping with the original objectives of the test.

Postirradiation Evaluation of a Thorium-Uranium-Zirconium Fuel Element

A discrepancy of about 1% exists between the fuel swelling determined for fuel element GEH-10-66 (1.4 at.% BU) using bulk fuel element density data and fuel sample density data. A correction applied to the bulk fuel element data for the effect of an observed 1.3-mil oxide layer on the Zr-2 clad yields a fuel swelling value of 2.6% which is in excellent agreement with the average swelling determined from two fuel samples. The swelling data are summarized in the following table.

Summary of Swelling Data of Th-U-Zr Fuel Element GEH-10-66

	<u>Density</u>	<u>$\Delta V/V$, %</u>
Bulk Fuel Swelling	-	1.6
Corrected Bulk Fuel Swelling for Oxide	-	2.6
Pre-Fuel Sample	11.70 g/cc	-
Post-Fuel Sample, Ave.	11.40 g/cc	2.6
Post-Anneal Fuel Swelling (800 C - 100 hrs)	9.56 g/cc	22.5

A segment of element GEH-10-66 was vacuum annealed at 800 C for 100 hrs to determine the higher temperature behavior of the highly irradiated Th-U-Zr alloy. After a slow cooling from the annealing conditions, the fuel element sample was observed to have severely cracked in the longitudinal direction (direction of extrusion) and to have separated from its clad by failure of the bond. The specimen incurred 22.5% swelling. Metallography shows that the cracking and additional swelling has resulted from the development of generally elongated gas porosity at grain boundaries. This is much more swelling than has been observed previously in Th or Th-U specimens when annealed under similar conditions.

Chemical burnup and uranium isotopic analyses have been re-run since there was a discrepancy in the U:Th ratio of the first determination. It was concluded that some thorium had precipitated from the solution following sample dissolution. A U:Th ratio of 0.0212 was obtained in the present analysis, and this compares with the preirradiation ratio of 0.0259. The new burnup analysis by the cesium fission-product method is 1.46% which is in good agreement with the calculated burnup from loop data of 1.37%. The analytical data are summarized.

Burnup and Uranium Isotopic Analysis
Fuel Element GEH-10-66

	<u>Preirradiation</u>	<u>Postirradiation</u>	
		<u>1st Anal.</u>	<u>2nd Anal.</u>
U:Th Ratio	0.0259	0.0259	0.0212
Cesium Burnup	-	1.71	1.46
U-232	-	0.04	-
U-233	0.09	35.10	35.12
U-234	1.07	2.73	2.70
U-235	93.20	44.99	45.3
U-236	0.24	10.26	10.09
U-238	5.40	6.93	6.74
	<u>100.00</u>	<u>100.05</u>	<u>99.95</u>

High Exposure Uranium Irradiation Test

Fifteen hollow core uranium fuel elements, 0.45-inch diameter by 6.25-inch long and clad with Zr-2 have been charged in the M-3 hot water loop of the ETR. These fuel elements are part of an irradiation test that has been designed to operate uranium fuel rods at elevated alpha phase temperatures to burnups greater than 10,000 MWd/ton (6×10^{20} fissions/cm³). The variables to be studied in this test include fuel composition, external restraint, and internal void volume. The combined effects of the plastic character of uranium during irradiation and the restraints from the cladding and system pressure are expected to cause the uranium swelling to be accommodated by a central hole. Two uranium compositions are being used: Alloy 1, containing U + 350 ppm Fe - 800 ppm Al; and Alloy 2, containing U + 150 ppm Fe - 100 ppm Si. A fuel enrichment of 4.5% is being used to achieve the desired burnup rate (10,000 MWd/ton in one calendar year) and temperatures. Fuel rods of both components were fabricated by coextrusion to 0.450-inch diameter with 0.025-inch and 0.050-inch thick Zr-2 cladding. Interim measurements and visual examination of each irradiated fuel element will be made in the ETR basin after each reactor cycle to provide a continuous evaluation of their performance.

A full charge in the M-3 loop facility would consist of 24 fuel elements. Additional fuel rods are now being fabricated to replace some of the dummy elements which are currently filling out the unused spaces in the charge basket at the ETR. During the course of brazing these elements, a different brazing

procedure was tried which appears to offer further improvements in expected yield of closures. The new brazing procedures will be further evaluated as more elements are available for brazing. Selected fuel elements from the full charge will be removed at intermediate exposures and returned to PNL for examination. Replacement fuel elements for those removed are being fabricated.

ENGINEERING DEVELOPMENT

Neutron Flux Monitors (W. G. Spear)

Regenerating Detectors (Thermal Flux)

Plans are being formulated for in-core testing at ETR of a set of regenerative detectors in high level neutron flux to an integrated exposure of 10^{22} nvt. The contemplated tests will involve reactor usage charges estimated at \$68,000 to obtain the desired integrated neutron flux.

Modified detector specifications, meeting requirements for testing at ETR, have been submitted to a commercial fabricator. Increased cost of these modified detectors and reactor usage charges for their subsequent test are being reviewed to assure that alternate experimental approaches are not overlooked.

A second 90 milligrams of high purity U-234 was procured for blending into a 90% U-234 and 10% U-235 regenerative mixture for use in two of the in-core detectors presently being prepared. In this material, the U-234 transmutes by thermal neutron capture to fissile U-235 in a manner to retain nearly constant sensitivity for extended in-core exposures, thereby extending the detector lifetime.

Beta Current Generator Detectors (Thermal Flux)

A high flux level in-core test has been completed at a production reactor to further define the operational characteristics of the second generation boron-11 detector. After several hours of exposure, the initial small neutron signal current decayed to a negative output current which subsequently stabilized at 5 microamperes. This reversed signal output, apparently caused by exposure to high level neutron flux, may be due to a complex internal charge distribution that exists within a semiconductor material such as boron. A companion carbon detector element, an integral part of the detector assembly, performed normally, generating a negative current output of 25 nanoamperes.

The boron signal current, in its negative current mode, exhibited overshoot and time constant (about 1-1/2 min) effects which negated precise neutron flux level measurements. Possibly competing nuclear reactions, such as (n, α) reactions with boron-10 generated ion-pairs which, after diffusion throughout the detector volume, created a signal-interfering space charge. Since boron apparently acts as both a current generator and a collection volume, consideration will be given to exploiting it as a solid state ionization chamber. At high neutron flux levels, effects from boron-10 can be significantly reduced (factor of 10) by burnout in 2-3 months of irradiation in a reactor. If need be, interference effects from competing boron-10 reactions will be eliminated by a burnout exposure.

The effects of various parameters and competing reactions upon device operation are being formulated and an equation which accounts for beta current generation, competing reactions, energy effects, burnout, and device configuration is being derived to describe the beta generator current.

Beta Current Generator Detectors (Fast Neutron Flux)

For expected neutron flux levels exceeding 10^{15} nv in fast flux reactors, beta current concepts, similar to those employed in the boron-11 thermal flux experiments, could provide a suitably high level signal current in the order of one microampere. Several candidate materials with an appropriate cross section of 0.1 barn for 3 Mev neutrons are beryllium-9, nitrogen-14, oxygen-16, and fluorine-19. If required for gamma compensation, the twin lead, twin chamber concept could be employed in a manner similar to that used for boron-11 thermal flux work.

Evolved Gas Detector Concept (Fast Neutron Flux)

Preliminary investigations continued regarding the possibility of measuring fast neutron flux by an evolved gas technique. For this detector concept a candidate material is beryllium, which reacts (n, α) with evolution of helium gas at a measurable rate proportional to neutron flux. Helium gas evolves from one gram of beryllium at a rate of 10^{11} atoms/sec at a flux level of 10^{13} nv.

A simple capsule, containing the beryllium would be coupled to a highly evacuated tube which would carry the gas to a detecting instrument. Use of a commercial helium leak detector to measure the evolved helium gas is being considered for an initial experimental test program.

Microwave Detectors (Thermal Neutron Flux)

The neutron flux of a reactor can create a plasma within a gas-filled microwave cavity, where the electron density will be proportional to the neutron flux density. Since the change in the resonant frequency of the

microwave cavity is proportional to the electron density, this change can be used to measure the neutron flux density. The plasma can be created by the following methods: (1) For thermal flux, a gas such as He^3 can be used, where an (n,p) reaction with subsequent ionization of the gas will create the desired plasma, and (2) a material such as U-235 or the U-234 regenerating mixture could be used where the fission fragments cause ionization of a cavity-contained gas.

To avoid sensitivity reduction caused by losses within dielectrics, such as a quartz capsule gas container, a gas tight seal to the cavity is being sought. Eight such seals in waveguide sections have been obtained and fabrication of two cavities to which these waveguide sections will be brazed is complete. Vacuum tight, high temperature brazes will be achieved in a hydrogen atmosphere in facilities being installed. In parallel effort, metalized ceramic discs will be brazed directly to the cavity coupling apertures. If the planned seals are successful, one He^3 filled cavity and one cavity containing U-235 plus a suitable gas will be assembled for in-core experiments.

Microwave Detectors (Fast Neutron Flux)

Continued progress in applying microwave techniques in measuring thermal flux indicates that similar approaches could be extended for fast flux measurements. Ionization of a cavity-contained gas by fission fragments of the neutron-high purity U-238 reaction would cause a measurable shift of the cavity resonant frequency. Compensation for gamma interference effects and temperature-induced frequency shift could be achieved by locking the microwave oscillator frequency to a gamma reference cavity. The difference frequency between the reference and neutron-sensitive cavities would then provide an accurate, sensitive measure of incident fast neutron flux.

Another promising method of measuring fast flux is by relating its effect upon a capsule of metallic, powdered beryllium to an induced frequency shift of a microwave cavity. Activated by the incident flux, the beryllium evolves a gas which would be continuously directed through, and would cause a frequency change of the microwave cavity. Interference effects could also be minimized by including within the instrumentation, a reference cavity to which the microwave oscillator is locked. Theoretical considerations indicate sensitivity of the apparatus would be about 10^{11} nv for a reasonable gas evolution rate of 10^{-2} atoms/sec/nv.

Experience gained during in-core measurements of thermal flux should provide a basis for future experiments and techniques for microwave measurements of fast neutron flux.

Microwave and Infrared Detection of Coolant Impurities and
Measurement of In-Reactor Temperatures (W. G. Spear)

Microwave Detection of Impurities in Coolant Gases

The dielectric constant of a gas mixture is a function of its constituents; thus, the presence and amount of water vapor in a helium gas stream can be determined by measuring the frequency shift of a microwave cavity or the phase change in a section of waveguide through which the gas flows. It may be possible, in the latter case, for the waveguide to be part of the process gas piping, thus becoming a truly in-line device.

Since the calculated frequency shifts will be very small, in the order of 125 Hz per 1 ppm of water vapor, long term sensitivity will be limited by frequency stability of the oscillator, reference cavity, and source cavity. Measurement data of short term frequency stability will be reduced to a form useful for predicting ultimate equipment sensitivity.

Effective use of the change-of-phase of the electromagnetic wave as it propagates in a waveguide section as a measure of water content, will require that extraneous phase changes be minimized. Theoretical calculations indicate that the movement of gas causes a negligible phase velocity change, for gas velocities much less than the velocity of light in vacuum.

High Temperature Measurements

Presently employed techniques of monitoring in-core reactor temperatures are not applicable in the interior of higher temperature reactors which will be operating in the future. To assure that reliable temperature measuring instruments are available for advanced reactors, development work regarding several temperature measuring methods is being pursued.

One temperature monitor, using a temperature-sensitive resonant microwave cavity, should enable temperatures to 1500 C to be measured. Because the microwave cavity is being fabricated from high and linear thermal-expansion metal, the expected frequency shift of 400 kHz/°C (at an operating frequency of 15 GHz) should be easily detectable and have a constant gradient for the temperature range of interest. Laboratory test of the completed assembly should confirm the temperature sensitivity and indicate if problems will be encountered with temperature induced oxidation of the cavity and connecting waveguide.

Radiant power emitted by material bodies is proportional to the temperature of the body, and this fact is the physical principle upon which a second temperature monitor is based. A part of the power, emitted at microwave frequencies, may be measured with a sensitive microwave radiometer. Initial calculations indicate that interfering radiation from the input waveguide, and sources other than the matched load which terminates the waveguide, should be minimized so the recorded temperature is meaningful. Power

emitted at 35 GHz should yield an "apparent" temperature of 583 K at the radiometer for the matched load (and reactor surroundings) at a temperature of 1273 K. Direct temperature readout could be achieved by injecting, within the external instrumentation, a signal corresponding to a fixed compensating temperature.

Infrared Techniques

Previous difficulties with the CO₂ absorption band were eliminated by use of a filter wheel which cut off at slightly shorter wavelengths. A filter wheel, previously fabricated did not work well with the balancing filters (wedge filters). A third filter, added to bring the instrument into balance in the desired sensitivity range, corrected all previous difficulties. Calibration curves were obtained over the range of dew points from 24 F to 56 F. These show that the instrument yields reproducible results with better than ± 1 F sensitivity. Calibrations were not extended below a dew point of 24 F because lower humidities could not be produced in a controlled manner. It appears, however, that the range of dew points between -55 F and 24 F could readily be monitored.

A filter set was fabricated for operation on the 1.83-micron water vapor absorption band. Tests using this filter set demonstrated that the resulting hygrometer is less sensitive with these filters than with those that operate on the 2.6-micron band. Since CO₂ sensitivity has been eliminated, there is no need to operate at 1.83 microns.

Upstream Boiling Burnout (D. R. Dickinson)

Heat transfer experiments to study upstream boiling burnout were performed on an electrically heated test section consisting of an Inconel tube 0.435-inch ID and 6-ft long. Two tests were completed measuring the burnout heat flux at mass velocities of 1 and 2 x 10⁶ lb/hr-ft² with a system pressure of 1500 psig and inlet subcooling of 115 Btu/lb.

These data, together with data from prior tests, show that the burnout heat flux at constant local enthalpy decreases with increasing mass velocity at low mass velocities (below 3 x 10⁶ lb/hr-ft²), whereas at the higher mass velocities, it had increased sharply. This distinction is of interest because it is at the higher mass velocities where upstream burnouts have been observed.

The test section used in these tests had pressure taps at 1-foot intervals to study changes in flow patterns as indicated by local pressure gradients. As the heat flux was increased in steps at constant inlet conditions, the pressure drop over each segment increased as expected, except on the last few small increases before burnout (which occurred at the downstream end), the pressure drop decreased over the downstream 1-foot of heated length and over the unheated section immediately downstream of this. Thus,

it appears that burnout was preceded by a change in flow pattern which persisted in the unheated section downstream. It is planned to obtain pressure drop data of this type over a range of conditions and relate it, if possible, to the occurrence of upstream burnout.

These tests were interrupted by trouble with an injection pump and the cleanup system in the test facility. The delay caused by this equipment failure forced the rescheduling of the remaining tests to later in the fiscal year.

PLUTONIUM UTILIZATION PROGRAM (F. G. Dawson)

Fuels Development

TREAT Capsule Fabrication

Fabrication of five UO_2 (10 wt% U-235 enriched) fuel capsules for irradiation testing in the Transient Reactor Test Facility (TREAT) has been completed. Except for shortened fuel lengths and simple cylindrical end plugs, the capsules are prototypic of PRTR fuel rods. The core of each capsule consists of one depleted UO_2 pellet (12.3 g/pellet) at each end of a vibrationally compacted UO_2 (9.54 wt% U-235 enriched) fuel column (127 g). A vibrationally compacted bulk fuel density of 83-84% TD was achieved.

The capsules were shipped to Argonne National Laboratory on November 15, 1966.

PRTR Fuel Rod Pressure Test

Two pressure sensing lines on instrumented fuel rods which were damaged during fabrication were repaired. One of the two fuel rods was repaired without removing it from the fuel element. The other had to be removed from the element for repair and is currently being reinserted. The repairs that were performed did not change the basic design of the test, and, consequently, a test addendum was not needed. A letter documenting the repairs has been written.

A hand operated regulator has been ordered for the balancing gas circuit as a secondary regulating system. Thus, it will be possible to measure fission gas pressure in the instrumented rods in the event the motorized regulator should not function properly.

Drawings are being prepared to document the pressure sensing instrumentation package.

HPD Fuel Rod

An irradiated HPD fuel rod was destructively examined to assess whether any changes which may deleteriously affect fuel performance occurred during the past fifteen months storage. No such changes were detected.

Examination of the rod from FE-6504, which contained UO_2 - 4 wt% PuO_2 fuel material, revealed a maximum diameter increase of 0.6% over a distance of 28 inches in the central region of the rod due to thermal expansion of the fuel material. This rod operated at a maximum linear power generation of 27 kw/ft with significant fuel melting. Metallographic examination of the Zircaloy cladding did not indicate an abnormally high hydride concentration although a thin, cracked, oxide layer formed on the inside surface. Examination of the fuel material did not reveal any unusual or unexpected observations. The fuel released 92% of the fission gases formed--the highest gas release measured in any PRTR fuel rod to date. There was nothing observed in this rod which could be attributed to its long storage period.

FERTF Test Element

The FERTF test element was redesigned to eliminate the possibility of the basket dropping to the bottom of the process tube and leaving the "bare" fuel element suspended in the core. A complete element of the new design is in the final stages of fabrication.

A prototype FERTF test element is presently being tested in the EDEL-I loop to determine fuel element wear and fretting characteristics under PRTR coolant conditions. Twenty-three days of testing at a coolant flow rate of 82 gpm, coolant temperature of 475 F, and coolant pressure of 1050 psi shows no significant amount of wear and fretting at various points along the assembly.

Project CAH-119 - Underwater Examination Equipment

Underwater disassembly and manipulation equipment for the examination and maintenance of FERTF experimental test fuels is being designed and will be fabricated locally. The equipment to be designed and fabricated locally includes:

1. Fuel element handling tray assembly for installation in the PRTR storage basin.
2. Fuel element support jig.
3. Underwater hood.
4. Scope brackets.
5. Profilometer mounting.
6. Surface water viewing windows.
7. Railing and grating additions.
8. Banding tool.
9. Drilling tool.

Equipment to be purchased offsite includes a viewing scope and a profilometer.

The conceptual design has been completed for the underwater tray, and detailed design will be completed in early December.

FERTF Component Fabrication

FERTF test fuel assemblies will be encased in "baskets" of various materials. One basket material, cast stainless steel that contained boron, was changed to Hastelloy-X in order to obtain the desired physics properties. Fabricators knowledgeable in forming Hastelloy plate into tubing were not readily available. In order to meet the fabrication deadline, a method of successfully forming Hastelloy sheet into tubing was developed at BNW. Two Hastelloy baskets will be available for use with the FERTF fuel element by mid-January.

High Exposure Plutonium Studies

The high exposure plutonium studies program has been realigned to better meet its primary objective of obtaining the necessary data to evaluate radiation exposure from plutonium produced by extended irradiation in civilian power reactors.

Fuel was obtained from Shippingport and Yankee reactors which have been selectively sampled to provide test specimens extending over an exposure range from 10,000 to 30,000 Mwd/Mt. Analysis for isotopic content and gamma energy spectra are complete on approximately half of the Shippingport fuel samples. Complete data are expected by the end of December. Yankee fuel samples have been selected and cut by remote handling procedures and are scheduled for analysis in late December and January.

The sample dissolver solutions are analyzed for fifteen isotopes of plutonium, uranium, americium, and curium and for Nd-148, Cs-137, and Np-237. The purified plutonium is given a beta energy spectra analysis, and when larger samples of plutonium are produced, neutron dosimetry measurements will be made.

In addition to the fuel from the two reactors thus far obtained, fuel is on order from Dresden, and some high exposure plutonium produced by a Hanford reactor will be set aside for the program.

The resulting data will be used for two computer programs, and, finally, experimental fuel elements will be fabricated using the high exposure plutonium as enrichment fuel. One computer program is designed to correlate the isotopic composition with the radiation produced by the resulting plutonium. Radiation hazards, dosimetry rates, and shielding needs can then be predicted from fuels of known isotopic compositions. The second computer program is designed to correlate isotopic buildup to the reactor flux and

fuel exposures and to predict the isotopic composition of reactor fuels as a function of integrated exposure and irradiation history. The information obtained will also be used to better define the quantities of the various transuranic elements produced under various reactor conditions, and define present fission yield and neutron adsorption cross section data.

It has been decided that no additional 5-gram samples will be obtained for this program. A sample of this size is too large for spectrometer studies and self-attenuation is too great for dose rate studies. Instead, sources of approximately 10^7 disintegration per minute ($\sim 5 \mu\text{Ci}$) will be prepared for spectrometer studies. Dose rate studies will be made on much larger samples, up to 1 kgm.

The new neutron spectrometer was received during this period but has not been set up as of this writing. It is planned to calibrate the spectrometer with the positive ion accelerator if we can obtain machine time.

Reactor Physics

D₂O Moderated Systems

Studies of Burnup in Al - 2.6 wt% Pu. A compilation of burnup data from plutonium-aluminum fuel is being prepared and will be published as an informal document, BNWL-CC-905 (12/1/66). The high exposure fuel which contained initially 16.45 at.% Pu-240 was irradiated in the PRTR as part of the Plutonium Recycle Program. The compilation makes the burnup data available to those who are interested and will not present any conclusions concerning the data.

Modification of the Least Squares Fitting Routine LIKELY for Burnup Analysis Problems. The generalized least square fitting code, Program LIKELY, has been modified in order to facilitate the processing of burnup data. These modifications, which leave undisturbed the basic features of the fitting process, have been incorporated in both a single precision version (SINLIK) and a double precision version (DUBLIK) of LIKELY. The double precision version proves valuable in combating the computer truncation effects. The principal modifications are:

1. Subroutine CHANCE has been modified to take advantage of the fact that all isotopes have been expressed as a function of a single isotope (Pu-239 for plutonium systems and U-235 for uranium-plutonium systems). This results in up to a factor of four savings in computer time for burnup problems.
2. The iterative process may be halted after a predetermined number of nonimproved fitting attempts. Usually the iterative process is unable to recover in the direction of a

converged solution after more than two or three non-improved iterations.

3. The problem size has been altered. The maximum number of variable parameters has been cut from 25 to 15, and the maximum number of data points has been increased from 100 to 150. These changes are in accordance with present burnup analysis requirements.
4. Program flexibility has been accomplished without impairing program efficiency by additional labeling for the parameters of the problem. The net effect is to be able to vary any set of successively occurring parameters and hold the remainder fixed without requiring the major effort that would ordinarily be required to rewrite subroutine THEORY.
5. The LAG feature of LIKELY has been expanded. All derivatives of those parameters which are held fixed by the LAG procedure are set to zero. This affects the parameter changes needed for the succeeding iteration. Although this method of holding parameters fixed is more flexible than item 4 above, it is much less efficient and therefore should be used only as needed.
6. Several useful additions to the output are:
 - the parameter variance-covariance correlation matrix,
 - a check on the matrix inversion involving the parameter variance-covariance matrix. (This provides some insight in the area of computer truncation effects.)

Batch Core Experiment. Critical experiments have been conducted in the batch core of the PRTR to determine coolant worths. Reactivity measurements have been made with D₂O, H₂O, and void in the central 55 coolant channels. The worth of D₂O coolant relative to air (for the 55 fueled channels) was measured to be about +0.8 milli-k. The worth of H₂O coolant relative to air (for all 55 channels) was measured to be about -5.6 milli-k. These experiments were conducted with approximately 26 wppm boron-10 in the D₂O moderator.

Analysis of data from the batch core critical experiments is continuing. Periods and reactivities have been calculated from experimental data on doubling times. Analysis of data from the irradiation of lutetium foils is being performed using the computer code LULU.

The paper, "Critical Experiments with High Power Density Fuel in the Plutonium Recycle Critical Facility," by J. W. Kutcher, J. H. Lauby, W. L. Purcell, L. C. Schmid, L. D. Williams, and J. R. Worden (BNWL-SA-743A), was delivered at the 1966 Winter Meeting of the American Nuclear Society

in Pittsburgh, Pa., October 31, 1966. It is anticipated that the material contained in that paper, along with the results of some additional calculations, will appear in a formal report.

Planning is under way for critical tests in the PRTR with the FERTF fuel assembly. Reactivity measurements, flux measurements with gold and lutetium pins, and gamma scanning of fuel rods are currently anticipated.

Experiments in the PRCF with D₂O Moderator. A report entitled "A Poison-Free Critical Configuration in the PRCF with D₂O-Moderator" is being typed.

A rough draft has been written of a report entitled "Reactivity Worth of a Central Safety Rod with and without an Internal Scatterer."

H₂O Moderated Systems

Critical Experiments with UO₂ - 2 wt% PuO₂ Fuel Rods. Critical experiments are being conducted in the PRCF using 1/2-inch diameter UO₂ - 2 wt% PuO₂ fuel rods which contain plutonium with 8 wt% Pu-240. The moderator is H₂O, and the water-to-fuel volume ratio is 1.83. Criticality was achieved with 252.16 fuel rods; several months ago a critical loading with the same fuel in the same configuration was achieved with 252.0 fuel rods. Between measurements the fuel was used in experiments for which boron was added to the moderator. The excellent agreement between measurements indicate that no boron remains on the elements. Measurements have been made to obtain reactivity worth of voids and fuel rods, and dysprosium activation data have been obtained to determine relative flux distributions in unit cells.

PuO₂-UO₂-H₂O Systems. Survey calculations for a PuO₂ - UO₂ fuel in light water are being performed with programs THERMOS, HRG, and HFN as an extension to a previous study. Hexagonal lattice spacings of 0.60, 0.70, and 0.93 inch are being considered. The first two lattices contain rods with 3/8-inch diameter fuel while the last one contains 1/2-inch diameter fuel. The moderator-to-fuel volume ratio is the same for the latter two systems. The fuel is 2.5 wt% PuO₂ in natural UO₂. The plutonium composition is assumed to be typical of recycle plutonium and is 62, 22, 12, and 4 at.% for Pu-239, Pu-240, Pu-241, and Pu-242, respectively. The 0.60 and 0.70 inch lattices are the same as for calculations previously reported (BNWL-305). The choice of rod sizes and moderator-to-fuel volume ratios lie in the range of interest for both pressurized and boiling H₂O reactor systems.

PRTR Water Tank Calculations. The preliminary calculations, from which the previously reported light water lattice results were obtained, are being redone using more realistic geometric and theoretical models. Improved 4-group cross section sets for use in the diffusion theory reactor calculations (HFN) have been obtained from HRG for the fast groups and from THERMOS and

TEMPEST for the thermal group. Only the thermal diffusion coefficients were used from TEMPEST. Included in these completed cross section sets, in addition to those for the normal PCTR material mixes (Pb-UO₂ drivers, U-Al drivers, graphite, etc.) are sets for the mixed oxide lattice cells in the unpoisoned, poisoned, and dry conditions. These lattices were composed of 1/2-inch mixed oxide rods of 2 wt% PuO₂ at a 0.8-inch triangular pitch immersed, respectively, in water, borated water, and air. The mixed oxide contained depleted uranium and 8 wt% Pu-240 plutonium. A start has also been made to obtain similar cross section sets for the 1.05-inch pitch mixed oxide lattice. An outline of a simple experiment, tentatively scheduled for early December, was designed which should solve one of the difficulties in this study encountered to date; that of defining a "correct" equivalent cylindrical boundary to use in the geometric model of the driver cells for the thermal spectra calculations. The results of this experiment will, most likely, necessitate further refinements to the driver cell's cross section sets to bring the reactor calculations in closer agreement with experimental measurements, since a rather crude model was used to obtain the present sets. A calculational technique involving a direct correlation of experimental data was also developed which should alleviate the second difficulty; that of adequately representing in the calculations the effects on the PCTR of the control rods in the open to closed positions.

PCTR Water Tank Fabrication. An experiment is planned for the PCTR which will involve water moderated lattice assemblies. The proposed scheme incorporates a supported water-filled aluminum assembly which will provide the desired volume of fuel, water, and aluminum with appropriate water fill and drain connections.

Comments on drawings were received. These included requests for the following additional set of lattice plates with different lattice spacing for the inner tank and test cells; a temperature sensor accurate to ± 0.05 C for the inner chamber; a method of making a flux traverse of the inner chamber; provision for a plastic model of the tank (to be used for demonstration purposes).

Design of the lattice plates is in progress. Existing temperature sensing hardware at the PCTR is being studied to see if it can be adapted to this core. Estimates have been requested from the Model Shop for the demonstration model. The flux traverse problem can be solved by using foils, but this method is time consuming. Therefore, the use of miniature flux chambers is being studied.

Reactivity Effects of PuO₂ Particle Sizes

Fuel Rod Fabrication. The first set of four 20-inch long fuel rods containing "zero size" PuO₂ (2 wt% PuO₂ - UO₂ solid solution) were completed. The rods consist of cold pressed pellets (80% TD) clad in Zircaloy. Assembly of two 10-inch long rods was started.

PuO₂ Spherical Particle Preparation. Work continued on the preparation of spherical particles from high energy impacted PuO₂. Sufficient particles were made for the 200 microns fraction. Particles for the 100 microns and 340 microns are near completion.

Graphite Moderated Systems

PCTR Experiments on PuO₂-UO₂ Single Rods. The infinite medium neutron multiplication factors (k_{∞}) for two PuO₂-UO₂ systems in a square graphite (PCTR) lattice are being measured in the Physical Constants Testing Reactor. The reactor phase of the experiments has been completed, and foil activation analyses are under way to determine the relative reaction rates in the cell components.

The two oxide systems under study contain 0.9 wt% PuO₂ with 7.3 wt% Pu-240 and 2.6 wt% PuO₂ with 29.3 wt% Pu-240. The uranium in both systems has a depleted U-235 content.

The measurements were obtained for the simplified geometry of a single 0.5 inch diameter fuel rod in a 4.0-inch square graphite cell. The central test sample for these measurements consisted of four cells centered in a 64-cell square array of the PCTR.

The mass of neutron absorber (copper) required to absorb the excess neutrons of the test sample was measured for the two oxide systems. These masses will be combined with the relative reaction rate measurements to calculate the k_{∞} for each system.

High Exposure Pu-Al Foil Mass Normalization. Aluminum clad high exposure Pu-Al foils (0.5-inch diameter) which were used in the 4-inch graphite lattice PuO₂-UO₂ single rod experiments in the PCTR, have been mass normalized by counting the residual Am-241 activity. Since these foils were all from one batch of plutonium and have not been irradiated after their manufacture, the relative Pu-241 content should be a measure of the relative total plutonium mass. The daughter product of the Pu-241 beta decay ($t_{1/2} = 13.0$ years) is Am-241 having a halflife of 461 years and emitting a 60 kev gamma ray. Since the Am-241 activity from an unshielded HX Pu-Al foil was too high to be measured by a scintillation counter, the 60 kev activity was attenuated by the use of a lead foil shield. Lead has a large photoelectric absorption peak (k-edge at 88 kev) with sufficiently low compton effect to make it ideal for this application. The correct lead thickness was determined by counting integrally as a function of pulse threshold bias for shield thicknesses of: (a) 0.0 inch, (b) 0.030 inch, (c) 0.045 inch, and (d) 0.070 inch. Tabulated below are the results of these measurements:

<u>Shield Thickness</u>	<u>Am-241 Activity Counts/min</u>	<u>Attenuation Factor</u>	<u>Activity 7400 kev Counts/min</u>	<u>Attenuation Factor</u>
Unshielded	9,650,000	1.0000	1094	1.0000
0.030" Pb	128,000	75.39	860	1.2721
0.045" Pb	65,000	148.5	816	1.3407
0.070" Pb	25,000	386.0	690	1.5855

The counter gain was increased to expand the detail in the vicinity of the Am-241 peak. Using a standard Am-241 source, the threshold bias was set just below the Am-241 peak. The HX Pu-Al foils were each counted five times (~60,000 counts per measurement) using the 0.045" Pb shield between the foil and the scintillation crystal. Computer code APDAC-III was used to reduce the counting data by a least-squares-fit to obtain relative activities. The resulting relative activities were used as the mass normalizations of these foils when they were irradiated in the PCTR to measure the relative absorption rates in the HX PuO₂-UO₂ fuel of the 4-inch graphite lattice single rod experiment.

Code Development

Offsite Distribution of Computer Program. In response to a request from Phillips Petroleum Company, the latest active version of Battelle-Northwest program Alchemy (Time-Variant Isotopic Transmutation) was shipped to Idaho Falls, for exploratory application to the solution of matrix differential equations.

HRG Data Tape.* A new HRG data tape has been produced which differs in several respects from tapes previously used. The new tape is the first updating of HRG data since the addition of large amounts of new data to the BNW Master Library in the past few months. Data for 80 nuclides have been revised or added. Multiple versions of U-233, U-235, Pu-239, and Pu-241 are present, to provide data compatible with several recommended self-consistent sets of thermal parameters for these nuclides. An extensive rearrangement of nuclide identification numbers has been made, placing the most frequently used nuclides near the front of the tape. Materials--currently H₂O, D₂O, and 304SS--appear as nuclides for the first time. A total of 237 nuclides is present.

UPDATE.* The computer code UPDATE was modified during the past month on the basis of timing runs made of the input and conversion routines for the Univac 1107. The results of the timing study indicated conclusively that the current speed of the I/O operations on the 1107 is restricted by the conversion routines and not by the drum access time. The modifications to UPDATE consisted of uniting two small machine language codes that replace the system

*Partially funded by 02.

routines for this specialized application. With these routines UPDATE runs between two and three times faster.

UNICORN.* Atomic International's computer code UNICORN was imported and converted for use on the Univac 1107 computer. UNICORN calculates unbroadened cross sections at discrete energy points, using resonance parameters for a zero angular momentum Breit-Wigner line shape. The interference between resonance and potential scattering is properly treated. Provision is made for spin-dependent potential scattering and adjustment to 2200 m/s cross section values.

ENDF/A Neutron Cross Section Library.* The latest ENDF/A library tape has been imported and converted for use on the Univac 1107 computer. This version has 1004 records and required almost 10 minutes of computer time for the conversion.

SCISRS.* The current SCISRS library tapes have been received from Brookhaven and are available for retrieval requests on the Univac 1107 computer. The latest tapes have approximately 630,000 data points compared to 300,000 data points for the older version.

Thermos Modifications. A version of THERMOS for cylindrical geometry is now available which contains both the Argonne improved method for calculating the transport kernel elements for thin and/or low cross section regions, and an arbitrary outer albedo option. The status of this version is reasonably debugged and in use.

Thermos Cross Section Library. A thirty-group data tape has been prepared for use with the Univac 1107 version of the THERMOS code. This tape reflects new data from the BNW Master Library and will replace the tape previously used and is recommended for problems involving materials at a physical temperature of 20 C. A listing of the data and the isotopic identification symbols may be obtained by contacting W. L. Purcell.

RBU Code Evaluation. A cell calculation was made using the RBU Monte Carlo code. Lattice parameters for this cell have been previously determined in a PCTR experiment. The cell is a natural UO₂ fueled 19-rod cluster - D₂O cooled and moderated. An error in describing the uranium-238 epithermal cross sections was observed in the first calculation, and the calculation will be repeated after the error is corrected.

Resonance Spatial Shielding Corrections. A computer code, DANCOF, has been developed to calculate the Dancoff correction factor for parallel cylindrical rods. Previously, the Dancoff factors were hand calculated by interpolation and extrapolation in a table published in ANL-5800. The program numerically integrates the expression for the factor C, as given by Dancoff and Ginsburg (CP-2157).

Fast-Neutron Cross Sections

All of the previously obtained total cross-section data have now been prepared for final processing.

Cross Section Evaluation

Work has continued on the preparation of data for the ENDF/B data file. Preliminary data on the 10 isotopes assigned to BNW were completed and presented at a meeting of the CSEWG. A BNW representative was assigned to a subcommittee to study the problem of cross-section standard values.

Industrial Applications

Plutonium Recycle Calculations. Dr. M. A. Robkin, Vallecitos Atomic Laboratory of General Electric Co., visited BNW during the month to perform calculations and discuss problems of mutual interest in the area of plutonium recycle. The calculations were performed utilizing methods developed and in use at BNW for the Plutonium Recycle Program.

Special Studies

Phonon Frequency Spectra. A better understanding of the microscopic structure and modes of motion for H₂O and ice is being obtained from slow neutron inelastic scattering measurements. A dephased rotor background scattering measurement was made on H₂O with an initial neutron energy of $E_0 = 0.608$ eV. The results of this measurement have been corrected to partial differential cross sections and scattering law $S(\alpha, \beta)$ as a function of energy and momentum transfer and combined with a previous phased rotor experiment. The corrected cross-sections and $S(\alpha, \beta)$ values obtained in this way are in better agreement with values obtained from lower initial energy scattering experiments.

A scattering measurement on ice was made with $E_0 = 0.1515$ eV and included a dephased rotor background run. An abstract entitled "High Resolution Neutron Inelastic Scattering Study of Water and Ice" by O. K. Harling has been submitted to the American Crystallographic Association for presentation of a paper at the Atlanta meeting January 25-26, 1967.

Reactor Engineering Development

Hydraulic Studies for the FERTF Fuel Element

Experiments were performed on a full-scale model of the FERTF fuel element to determine the portion of the total flow which goes through the fuel element and the portion which is bypassed around the element through the flow passages exterior to the basket which contains the element. The fuel element model consisted of a ring of six rods 0.564" OD supported by a central support rod 0.905" OD. This element was installed in a basket 2.715" ID and 2.980" OD. The basket had six ribs 0.185" high and 0.250" wide running the full length.

Experiments were performed with cold and hot water to determine the percentage of the total flow going through the fuel element and to find the flow-pressure drop behavior of the fuel element and complete fuel-basket assembly.

It was found that approximately 83% of the flow went through the fuel element and the remaining 17% went through the annulus between the basket and the pressure tube wall. This means that to obtain the desired flow of 65 gpm through the fuel, a total pressure tube flow rate of 79 gpm is required. At this flow rate, a pressure drop of 0.6 psid was measured. The coolant temperature increase across the fuel element will be approximately 121% of the mixed bulk flow at the discharge of the pressure tube.

Reactor Components Development

Fuel Element Testing. The basket type FERTF fuel element has been under test in EDEL-I for 822 hours of which 787 hours have been at the designated test conditions of 480 F, 1050 psig, and 82 gpm. The loop has been shut down weekly and the fuel element removed for inspection. During these inspection periods, the fuel element was disassembled, inspected, and reassembled by representatives of Fuels Development. Inspection of the pressure tube was performed by Equipment Development personnel.

On November 17, the loop was shut down because of a bearing failure in the pump. The pump has now been disassembled, and all bearings and seals will be replaced. Replacement parts are available, but damage to the motor shaft and bearing seats will require additional work to repair. Repair of the pump is under way on a top priority basis.

Rupture Loop Particle Separator. Changes of the installation design, prompted by comments from PRTR personnel, have been resolved and are being incorporated into the drawings. Significant changes are additional shielding

for the filter cask, an improved scheme for handling and removal of the cask, and positive features for locking the cask closed and opening it for filter removal.

Fabrication of the loop in which the axial flow separator will be tested was started during the month. All material has been ordered, and fabrication is estimated 25% complete.

Fabrication of the axial flow separator was continued during the month at Tech Shops and is now estimated at 60% complete. Efforts to complete this fabrication earlier have been unsuccessful due to a severe shortage of machinists at the shops.

UO₂ particles, for use in testing the separator were ordered; one typical of sizes used in PRTR fuel and the other 100 microns or less. Disposal of nonseparated particles using the process sewer in the 314 Building has been approved by the BNW Radiation Engineer.

BWR and PWR with PuO₂ Fuel Study

Modification of a linking code called "CHAIN" to include REPP as a link in a set of economic and nuclear burnup codes was completed. A user's manual for operation of REPP is currently being written.

Materials Development

PRTR Pressure Tube Evaluation

The PRTR reactor safeguards program includes study by destructive examination of the effect of reactor environment upon the Zircaloy-2 pressure tubes. Work in progress is composed of measurement of elastic strain on un-irradiated tube specimens while under different loading conditions and crack propagation tests at various temperatures. The respective objectives are to describe elastic behavior of an anisotropic tube and to provide crack propagation data. This work is continuously improving the basis for judgment of irradiated specimens.

The following has been done on the evaluation of pressure tube #5696. A 20" crack propagation specimen was taken from the area of maximum flux and was measured for outside diameter and wall thickness at Radiometallurgy. A 1-1/2" slot was then milled 80% through the wall. The specimen failed at 3650 psi and had a wall thickness of 0.153" at the flaw and an average diameter of 3.578". Using Lamé's equation a hoop stress of 41,000 psi was obtained. This value is very close to the values obtained in the past and shows that neutron irradiation up to 2×10^{21} nvt-fast has not shown much of an effect on crack propagation characteristics of the tubes.

Six 20" crack propagation specimens have been measured and slotted with flaws milled 80% through their minimum wall thickness. Three of these specimens with slot lengths of 3/4", 1-1/2", and 3" have been broken at 300 F. The other three tubes with identical slot lengths are going to be broken at 570 F. When the three specimens are broken at 570 F, plots will be made of strength vs temperature for the different size crack lengths to determine if they all have a common slope as previously found in tests that were run up to 300 F.

Three notch tensile specimens and three regular tensile specimens were cut out of 6" length of tubing. The notched specimens had two 60° notches with a radius of 0.001" coming in from the sides. The notches cut the cross section of the specimens in half. The average strength of the notched specimens was 92,300 psi, and the average strength of the unnotched specimens was 79,400.

Two 20" tubes have been measured, vapor blasted, and are now being hydrided with 150 and 275 ppm hydrogen. When the tubes are hydrided, they will have the case etched off and then will be slotted with 1-1/2" slots milled 80% through the wall.

Ceramic Fuel Dissolution Studies

The primary emphasis in the ceramic fuel dissolution program during the past month has been placed on applying the OPG process to the decontamination of the PRTR Fuel Element Rupture Test Facility (FERTF). The heat exchangers and heaters have been cleaned with apparently excellent results. Rough calculations using β - γ measurements indicate that about 50 grams of 9/29/65 incident rupture debris have been removed from the system. Currently, preparations are being made to complete the FERTF decontamination. The entire system will be treated with the OPG solution; the AP-RDW-3 process will follow to remove adsorbed radioactivities from system surfaces and pipe walls.

Cycle Analysis

Code Development

A few remaining problem areas are being removed in the addition of a thermal hydraulics code link to the 1107 Fuel Cycle Analysis Chain. This link is to have a capability of handling both PRTs and BWRs.

A major revision is under way for the JASON link of the chain. Results of this revision are expected to allow use of up to five isotopes/region, improve output formats, expand output capability, expand the number of regions available to 30, and provide a documentation of the end result.

Water Reactors

Final revisions are currently under way for inclusion to BNWL-SA-776, "Some Nuclear Characteristics of PWRs Recycling Plutonium at Minimum Fuel Cycle Costs." This report was delivered at the winter meeting of the American Nuclear Society in Pittsburgh.

ANS Paper

The ANS paper, "Economically Optimum Plutonium Lattices for Water Reactors," by J. H. Nail and D. E. Deonigi presented at the Pittsburgh meeting has been cleaned up and will be made available for distribution on request.

Transient Behavior Studies

About one-quarter of the graphs for the paper, "Dynamic Effects of Plutonium Recycle," have been completed. Further calculations are being done at this time.

Fuels Reprocessing Development

The terminal reports of the Chemical Development contribution to the Salt Cycle Process have been turned into Technical Files for final approval. The assigned report numbers are:

BNWL-354, Engineering Development of the Salt Cycle Process,
G. Jansen, Jr., K. K. Mudge, and P. W. Smith

BNWL-355, Demonstration of the Salt Cycle Process in a High-Level Radiochemical Facility, W. R. Bond, G. Jansen, Jr.,
and L. K. Mudge.

Work has been started on Chemical Development's portion of a chapter in a book called Process Chemistry to be edited by C. R. Stevenson, Argonne National Laboratory. The chapter is to be entitled, "The Salt Cycle Process," by K. M. Harmon and G. Jansen, Jr. The deadline for getting this submission in final form and completely approved is January 1, 1967.

Test Reactor Operation

Operating Experience

All preparations for the containment vessel leak rate test were completed early in the month, and on November 12 and 13, the vessel was pressurized to

10 psig. The 24-hour leak rate was measured by the reference vessel method at 0.43% by weight. This compares favorably to the allowable operating leak rate of 0.678%.

Following the successful completion of the containment vessel pressure test, the remainder of the month was spent returning systems and equipment to normal. At month end, the facility is capable of startup on a week's notice.

The primary system D₂O was drained to the three north tunnel storage tanks for the duration of the containment vessel pressure test and has not yet been returned to the piping.

Piping and containment alterations to the rupture loop were established and phase I of the rupture loop decontamination was completed. Approximately 50 grams of fuel element debris were removed by dissolution and transported to special cribs in 200 East Area. This reduced the radiation readings from a maximum of 20 R down to a maximum of 600 mr.

Indicated helium loss for the month was 28,584 scf. D₂O loss was 64 lbs, based on stack monitoring data. (No physical inventory was taken.)

Process Technology

Critical Test No. 14 was written to measure the power generation rate of the FERTF six-rod fuel element assembly in channel 1550 with H₂O coolant relative to the power generation in an adjacent 19-rod cluster.

A supplement to Batch Core Power Test No. 8 (oscillator measurement) was issued covering the short period problems which were experienced during Critical Test No. 10.

Batch Core Power Test No. 9 (reactor loading sequence for power operation) was drafted and is being circulated for comments.

A set of single rod Thermos calculations was performed for the Fuel Element Design Unit for comparison of the relative power generation in an H₂O-cooled, 1.785 wt%, 95% TD fuel rod, with that in an identical rod cooled by D₂O. The results from these calculations were reported by letter.

Final reports were issued for the following PRTR tests:

- No. 32 - Shim Worth Measurement
- No. 75 - Correlation of Automatic Controller Data
- No. 77 - Operational Testing of Automatic Controller
- No. 94 - Corrosion Study in Calandria Atmosphere.

An interim report was issued for PRTR Test No. 61 (irradiation of six instrumented mixed oxide fuel elements). Irradiation of five elements has been completed. The sixth element will be recharged for continued irradiation.

Analyses of fine solids filtered from the moderator show very small amounts of fuel and fission products, although the majority of radioactive material is Co-60, as reported earlier. Ratios of other activity to that of Co-60 are:

$\text{Zr-Nb}^{95} = \sqrt{\frac{1}{15}}$, $\text{Ce-Pr}^{144} = \sqrt{\frac{1}{10}}$, $\text{Pu}^{239} = \frac{1}{350}$, $\text{Pu}^{238} = \sqrt{\frac{1}{150,000}}$, $\text{Cm}^{242} = \sqrt{\frac{1}{40,000}}$
and $\text{U} = \frac{1}{2,000,000}$. The measured weight percent of total Pu (in the uranium oxide fuel) was 3.2 to 4.8% (about as expected).

Improvement Work Status

Work Completed. The H-79 valve, an auxiliary helium bleed from the pressurizer, was found to be unnecessary and expensive to maintain and operate, and was therefore removed from service.

A reactor hall crane work platform was installed to safely work on the fog spray system and to change reactor hall lights.

The gas blanket regeneration blower speed was increased to reduce tower regeneration time and increase drying tower capacity.

The primary system O₂ analyzer, previously unused, was removed.

One corrosion monitoring probe was installed in the discharge line from the core blanket blower and three probes were installed in the thermal and biological shield system--one at the inlet and one at each shield outlet.

Work Partially Completed.

New Instrument Power Supply
Effluent Monitor System Calibration Modifications
Instrumented Fuel Element Installation
Helium Compressor Compensating Pump
Remote D₂O Addition Station
M&M Wing Reheat Coil
Gamma Scanner Modifications
R. L. Bypass
R. L. Particle Removal Equipment
Dump Valve Testing
Safety Circuit Modifications
Remote Readout for Primary System
Unsoftened Water Control Valve
Deaerator Overflow Modification
TW Tank Bypass
Lobby Modifications for Personnel Accountability

Design Work Completed.

Safety Circuit Modifications
New A- and B-cell Sump Pumps
TW Tank Bypass
Unsoftened Water Control Valve

Design Work Partially Completed

Analysis of HX-1 and Primary Pump Supports
Improvement of pH Monitor
Containment Valve Testing Connections
New Storage Basin Tray
Reflector Sweep System Modifications
Exhaust Air Monitor Installation
Fire Protection Improvements.

Projects - Technical Guidance

CBB-001 - Increased Power Level. Scheduled phase I items and the chemical shim system are complete except for deaerator modifications; the new deaerator distribution system has been received and will be scheduled for installation in the near future.

BCP-007 - PRTR Waste Handling. This project is to provide improved waste handling capability and control of reactor systems during accident situations. The work on MH-2 pump and TW tank bypass by Minor Construction is 90% complete. NPC's and design changes for seven items not presently covered by such documents were prepared during the month. Work on the deaerator overflow modifications (DC-279) was started during the month. Work on the rupture loop bypass (DC-RL-22) is 70% complete. Valves for the rupture loop change are not expected until January. The D₂O addition station is 60% complete.

BCP-013 - PRTR Contamination and Waste Control. This project is to provide improved contamination and waste control during accident situations at PRTR. A directive and work authority for design only were received from the AEC during the month. Design was started on all eight items during the month.

AEC-193 - Fire Protection Improvements. This project is to provide sprinkler protection in the 309 Building in all areas except the control room and containment vessel. Drawings, specifications, ATP's and special conditions for the sprinkler risers, detection system and alarms were received from the AEC for comment and comments returned during the month. The sprinkler system proper is to be designed by a sprinkler contractor.

EBWR Demonstration Program

Irradiation Testing of EBWR Prototype Fuel Rods

Two capsules containing short pieces of EBWR, Zircaloy-clad, vibrationally compacted $\text{UO}_2 - 1.5 \text{ wt\% PuO}_2$ fuel rods (1.07 cm OD) continue to be irradiated in the MTR. The maximum burnup achieved to date is $\sim 26,900$ MWd/ton ($\sim 6.73 \times 10^{20}$ fissions/cm³). The two capsules are the last of a group of 32. One of the discharged capsules (GEH-14-529) is currently being sectioned for examination. The burnup attained with this capsule was 22,100 MWd/ton (5.5×10^{20} fissions/cm³). The capsule had no plenum, originally contained 52.7 grams of fuel ($\sim 84.3\%$ TD), and operated with an initial linear power of ~ 16.3 kw/ft. When drilled, the capsule released 6.7 ml of fission plus other (e.g., initial helium) gases. The double-clad capsule showed no diameter change on the outer aluminum (2.9 cm OD) sheath.

Two production-run EBWR fuel rods (burnups are 1650 and 5500 MWd/ton) are being examined. The lower end cap region (only unautoclaved portion of the rods) showed no evidence of internal or external hydriding. Of the total of 58-64 full-size rods to be irradiation tested, 33 are in reactors and 25 have been discharged.

EBWR Experiment

The EBWR Plutonium Demonstration Experiment is in the approach-to-power phase. During this phase oscillator stability measurements are being made at various power levels. The power is ~ 40 MW, and reactivity estimates indicate the possibility of reaching a power level near 70 MW. Approvals are needed to operate the reactor at the higher power level for the plutonium experiment.

Analysis of Saxton Experiments

A calculational study of the Saxton plutonium experiment has begun. Analysis of the $\text{UO}_2\text{-PuO}_2\text{-UO}_2$ and $\text{UO}_2 + \text{PuO}_2\text{-UO}_2$ loadings in H_2O moderated lattices are being studied initially.

NUCLEAR SAFETY

Containment Systems Experiment (J. M. Batch)

Experimental Facilities

The containment vessel with completed penetrations was returned to operations from construction forces on November 14.

Construction work priority was assigned to the blowdown facilities after work on the containment vessel was completed. Installation of the structural steel for supporting the reactor simulator vessel was completed, and the cage for holding the vessel was delivered. Utilities and the high pressure loop piping were extended to the building. The reactor simulator vessel was delivered in mid-November.

Aerosol Studies

Two tests were made in the Aerosol Development Facility. In one, the containment behavior of iodine in a stainless steel vessel during a pressure decay from 36 psig and 255 F (124 C) was studied. In the other, the iodine behavior in a painted tank during its first exposure to steam was studied. The pressure decay test proceeded as expected on the basis of earlier isothermal tests, with the half-time of containment gas phase removal decreasing as the steam condensation rate decreased with time. After nearly all the steam had condensed, about 65% of the iodine remained on the internal surfaces; 35% was in the condensate and 0.1% remained airborne.

The second test was made in the painted aerosol tank to obtain data on the interaction of iodine with a painted containment surface which had not been previously exposed to heat or steam. The test was made at the end of the report period, and results are not yet available. The tank interior had been sandblasted and sealed by a primer coat and three coats of Pheno-line 302. The last coat was applied February 28, and the paint has air dried since then with no exposure to heat or steam. The run was made under isothermal conditions at approximately 175 F (69 C) -2" Hg gage with an atmosphere of about 50% steam-50% air by volume. Iodine was injected as aerosol, essentially instantaneously, in an amount calculated to provide one milligram in the 50 ft³ tank volume.

A preliminary theoretical investigation of the effect of a liquid steam condensate film holdup showed that a knowledge of holdup volume is important to understanding material balances as a function of time. The holdup in the stainless steel aerosol tank was measured as 470 cm³ at steady state conditions of 80 C, 0 psig.

Aerosol Generation

Labeled hydrogen iodide was air swept through an 0.87" ID stainless steel delivery line into Maypack and scrubber sampling systems. Fifty milligram release tests at delivery tube temperatures of 20 and 100 C and an air sweep flow of 0.5 fm showed greater than 92% conversion of HI into elemental iodine. Residence time of the HI in the 8-ft length delivery tube was 1.3 sec which shows the oxidation of HI is rapid for the above conditions. Retention on the 100 C delivery line was less than 0.5% of the input inventory; similar retention was observed for elemental iodine in the same apparatus. Hydrogen iodide prepared by the same method but swept by nitrogen directly into a CCl₄-H₂O partitioning scrubber showed greater than 90% of the activity persisted as HI.

Leakage Rate Testing

Instrumentation for leakage rate tests, including 24 platinum RTDs, 7 lithium chloride humidity sensors, and a 400-ft piece of nickel wire have been installed in the containment vessel. The reference volume tubing is being tested with a helium leak detector sensitive to $<2 \times 10^{-8}$ atm cc/sec. A halogen gas detector is being used for locating the leakage points on the containment vessel.

Instrumentation

The heated thermopile anemometer for measuring containment atmosphere velocity was tested further on the stainless steel ADF tank, using the rotary arm previously used to test the performance of the probe in a steam atmosphere. The probe performed within a 10% accuracy band to 85 C, 6 psig steam/air, but during tests at 123 C (253 F), the accuracy was not within a 10% error band. Convection currents are generated by steam injection, rotation, and temperature gradients, making calibration difficult, especially at the higher temperature.

Development continued on the neutron densitometer by the Instrumentation Services Unit. Calibration tests were made with new BF_3 counter tubes which have the advantage of being insensitive to vibration. The calibration curves matched those made previously with the ionization detector. The dynamic range of the setup was between six and seven. That is, when the water fraction was changed by a factor of 10, the current output changed by a factor of 6. X-ray densitometers have a typical range of only 1 or 2.

Pressure Vessel Crack Monitoring (J. C. Spanner)

Detection of Metal Overstress by Acoustic Emission

Development work at BNW, to produce a system to detect crack growth in reactor pressure piping by sensing the attendant acoustic emission, was compared with work in acoustic emission application being done at two other locations--Lawrence Radiation Laboratory (LRL) and the National Reactor Test Site (NRTS). Both locations were visited for purposes of exchanging information and assessing any duplication of effort among the three programs. There does not appear to be any significant duplication. The LRL program is directed toward a very specific, classified application and is concerned with a unique material which is not included in either of the other programs. The program at NRTS, being conducted by the Phillips Petroleum Co., is aimed at producing a proof test monitor for the LOFT pressure vessels. Although this has some similarity to the BNW application goal, PP Co. personnel are investigating an approach distinctly different from that being pursued at BNW. In view of this, the two programs complement each other, rather than duplicate, because both approaches show individual merit making them worthy of further development.

Hardware development of a prototype instrumentation system has been limited to the analyzer portion of the system. A count-rate meter is being used in conjunction with a multivibrator circuit to determine the occurrence rate of the acoustic emission. The output is plotted automatically with an X-Y recorder. The count-rate meter provides a continuous indication of the instantaneous event rate, and the multivibrator circuit allows compensation for the fact that many of the acoustic emission events reflect the resonant "ring-out" of the transducers.

Another group of double cantilever beam specimens has been tested to study emission from crack formation and growth in 304-L stainless steel, A-212-B carbon steel, and Zircaloy-2. Analysis of these data, plus earlier similar data, with the newly assembled analyzer system shows evidence of frequencies not previously observed. In addition to the previously observed single pulse transient effect, there is also an indication of a high frequency (100-150 kHz), continuous type emission of varying amplitude. An explanation of the profile of this emission vs the test sequence has not yet evolved but could be quite significant in the development of useful "acoustic signatures."

The frequency response characteristics of the acoustic emission monitor system now in use have been defined in detail as an aid to evaluating authenticity of acoustic emission data.

Development work continued on a self-aligning electrostatic transducer for detection of acoustic emission. The high sensitivity required for detecting acoustic emission has been demonstrated with the electrostatic transducer; however, breakdown of the dielectric films has been experienced both from bias voltage and from shock pressures. An attempt is being made to strengthen the dielectric film by vacuum deposition techniques. It now appears that the next step in optimizing the electrostatic transducer will be to improve the dielectric deposition techniques to obtain better bonding plus the use of better electrode materials.

Metallurgical consultation in support of the acoustic emission program has been arranged. A series of selective tests are being initiated to provide a more positive insight into the correlation of the metallurgy of crack formation and growth with accompanying acoustic emission.

A verbal summary of program effort planned for the balance of the fiscal year was provided to the program sponsor, R. R. Newton, DRD&T, USAEC, during a telephone conversation on 11/4/66.

Reactor Safety Analysis and Evaluation (R. G. Wheeler)

Subsize Vessel Failure Analysis

This portion of the program is concerned with evaluating the failure behavior of irradiated low alloy steel pressure vessels from tests on subsize vessel specimens.

During this period 15 unirradiated A212B subsize-vessel specimens were destructively tested. Each of the specimens had a through thickness longitudinal crack. The results of these tests will provide the control data appraising the effect of irradiation on A212B subsize vessel tests. In addition, the microhardness flow surface technique was used to obtain room temperature yield strength values of the irradiated and unirradiated A212B for multiaxial loading conditions. The results of these tests are being analyzed and will be prepared for presentation in a formal report during the next reporting period.

The unirradiated subsize vessel tests indicate a fracture mode transition, fracture surface appearance of ductile to brittle, at -17.8°C . This temperature is somewhat higher than the NDT temperature (-30°C) predicted by a modified drop weight test. The fracture appearance transition temperature of unirradiated subsize vessels representing two different specimen sizes was determined in order to investigate size effects. One size was identical to the irradiated specimens (0.25" wall thickness, 2.00" inside diameter, and 18" long). The other size was the same length and inside diameter as the irradiated specimens but had a wall thickness of 0.20". Although there is a significant difference in wall thickness between these two sizes, they both indicated the same fracture mode transition temperature. It was previously reported that the irradiated subsize vessels indicated NDTT shifts of 103°C for 5×10^{18} nvt and 54°C for 2.5×10^{18} nvt. This NDTT information was based on the assumption that the NDTT coincided with the fracture mode transition temperature. In view of the recently obtained information, the NDTT shifts should be corrected to 90°C for 5×10^{18} nvt and 42°C for 2.5×10^{18} nvt. These corrected values fall near the center of the scatter band of NDTT shift values reported in the literature.

The room temperature hoop stress at the yield point for an unirradiated A212B subsize vessel specimen was measured (from a strain gauged test) to be 58,200 psi. The predicted value, from a stress calibrated microhardness flow surface, was 58,500 psi. The microhardness flow surface was calibrated, in units of stress, from the uniaxial tensile tests. The uniaxial yield stress of this material was measured as 46,800 psi for the longitudinal direction of the subsize vessels. This hardness number to stress calibration of the unirradiated material was used to calibrate the microhardness flow surfaces of the irradiated material. The irradiated subsize vessels were predicted to have yield strengths of 82,000 psi for the hoop stress in a subsize vessel pressurization and 77,000 psi for a uniaxial tensile test in the longitudinal direction. Although these predictions are in reasonable agreement with values in the literature, they might be improved by calibrating the irradiated flow surfaces with the results of a tensile test of the irradiated A212B subsize vessels. There was no significant difference between the flow surfaces for 2.5×10^{18} nvt exposure and for 5×10^{18} nvt exposure. The irradiation did reduce the effect of biaxial strengthening of these subsize vessels from 24.5% to 6.5%. For an ideal isotropic material the biaxial strengthening of a subsize vessel would be 15.5%. In addition, the radiation damage indicated by the change in the multiaxial loading yield surface was not three dimensionally uniform, isotropic.

Columbia River Sedimentation Studies (D. R. Kalkwarf)

A significantly better correlation between radionuclide concentration in bottom sediments and distance along the Columbia River was obtained by analyzing particle-size fractions of these sediments. A plot of the logarithm of radionuclide concentration in a particular particle-size fraction was linear with distance over a 240-mile reach of the river involving seven sampling locations, whereas corresponding plots for whole sediment reflected the wide variation in the particle distribution of the samples. The separated fractions have also been submitted to the U.S. Geological Survey laboratory at Denver for identification of the minerals present.

Waste Solidification Condensate Treatment (C. J. Touhill)

A two-step scavenging process appears to be a feasible method for treating glass melter condensate. Optimum treatment is effected by the addition of 5×10^{-4} M phosphotungstic acid for precipitation of a mixed phosphotungstate in the first step, and by the addition of 0.03 M Mn^{+2} for scavenging by mixed hydrous oxides in the second step. The centrifuged residues from both steps can be readily dissolved for return to the head end of the solidification process, thus the mixed phosphotungstate residue from 50 ml condensate was dissolved in 0.5 ml of 9 M NaOH. The mixed hydrous oxide formed at pH = 12 from a final aqueous volume of 112 ml was dissolved in 1.5 ml of concentrated HNO_3 . In neither case did any reprecipitation occur upon standing for 24 hrs.

Fission Product Aerosol Containment

Removal of Organic Iodides with Hydrazine (L. C. Schwendiman)

Testing of equipment associated with the new, larger spray chamber was begun. After minor modifications, pumps and electrical services appear to function as expected. A chamber temperature difference of more than 40 F was observed when the chamber was heated by injected steam. The original stirring propeller was ineffective in mixing the tank as evidenced by the existence of the large temperature gradient. This stirring device subsequently failed, and other means have been sought to provide the desired mixing.

Physical Chemistry of Hydrazine-Methyl Iodide Reaction (L. L. Burger)

The reaction between methyl iodide and hydrazine was studied by determining the influence of added chemical agents, temperature, and gas phase volume on the removal of methyl iodide from a static gas volume. Also, a model for the reaction was proposed and tested with available data.

No increase in the rate of removal of methyl iodide in the static system was observed when either octanoic acid or butylamine were added to the alkaline hydrazine solution. Addition of ammonium hydroxide to a 13 wt% hydrazine solution (5 M NH_4OH , 0.05 M NaOH , at 50.1 C), increased the rate of removal, $d(\ln\text{CH}_3\text{I})/dt$, from 0.0224 min^{-1} to 0.0385 min^{-1} . In the absence of hydrazine, the rate was found to be 0.007 min^{-1} which is explained by reaction of the hydroxyl ion with methyl iodide.

The rate of removal of methyl iodide from the gas phase over a 50 wt% hydrazine solution as a function of temperature is shown in the table following.

Rate of CH_3I Removal as a Function of Temperature

50 wt% N_2H_4	
0.0504 N NaOH	
3.6 cm^2 surface area of liquid pool	
54.5 ml volume of gas phase	
<u>Temp., °C</u>	<u>$d(\ln\text{CH}_3\text{I}) \text{ min}^{-1}$</u>
50.1	0.11 ± 0.01
40.0	0.082 ± 0.005
35.0	0.077
32.4	0.066
21.2	0.059

The effect of gas phase volume on the rate of removal of methyl iodide was determined. The first order rate constant for the disappearance of methyl iodide was found to be inversely proportional to the volume of the gas phase over a volume range of from 12.5 to 45 cm^3 . The model which is proposed correctly predicts the volume dependence. The model also predicts a dependence upon the surface area of the liquid, and this is consistent with results reported earlier.

The proposed model for the removal of methyl iodide from the gas phase over an alkaline hydrazine solution results in the equation:

$$-\frac{d \ln n}{dt} = \frac{K_f A}{V} \frac{kT}{2\pi m}$$

where n is the gas phase concentration of CH_3I , K_f is the fraction of molecules which react with the surface after collision with it, A is the surface area (cm^2) of the hydrazine solution, V is the volume of the gas phase (cm^3), T is the temperature ($^\circ\text{K}$), k is the Boltzman constant ($1.38 \times 10^{-16} \text{ erg/molecule } ^\circ\text{K}$), and m is the mass of the CH_3I molecule (g/molecule).

It was determined experimentally that K_f was proportional to the mole fraction of hydrazine hydrate in the solution. At 50.1 C the ratio of K_f for ethyl iodide to K_f for methyl iodide was found to be about 0.5.

Disposal of Reactor Off-Gas Into Soil Systems (W. A. Haney)

Work is progressing on findings ways for economizing the solution by finite differences of the gas injection problems for the heterogeneous soils at NRTS. Ways of choosing better acceleration factors and thus decreasing required computer time are under study.

The solution for the diffusion problem progressed satisfactorily. The results will be used to analyze field data from NRTS to assess the significance of diffusion as a natural driving force for moving gas through the ground once pumping has ceased.

Radioactive Waste Solidification (A. M. Platt)

Process Chemistry

Assistance to Run WSEP Run 1 (PW-1, Pot-Cal Process). Blending of Purex 1WW and inert feed solution to form the desired PW-2 plus additive composition was demonstrated with simulated solutions. The resulting solution concentrated without serious foaming. Sulfate evolution from the calcine did not begin until a temperature of 1000 C was reached; between 1000 C and 1200 C, only 1.7% of the sulfate initially present was volatilized.

Assistance to Run CPG-4 (PW-1, Phosphate Glass Process). The feed to this run was very high in phosphate (M/P = 0.68). The melt was found to be unstable in a thermobalance run. Weight was lost rapidly until a stable composition corresponding to an M/P ratio of 0.914 was reached.

Assistance to WSEP Run 2 (PW-1, Spray Solidifier). Twelve new melt compositions were examined in a compositional area that is of interest for processing PW-1 with sodium and phosphate as additives. The stick points, melting points and slump points fitted quite well into the pattern that has been previously derived for this region of the $\text{PW-1:Na}_2\text{O:P}_2\text{O}_5$ system. It now seems quite unlikely that there exists in this area any combination of components which will yield a significantly reduced melting point.

WSEP Run 2 is scheduled to be operated with the off-gas and condensate from the spray solidifier routed back to the feed concentrator. An Othmer still was used to obtain HNO_3 vapor-liquid equilibria for simulated WSEP Run 2 feeds at several concentrations and acidities. These data, plus density measurements, were correlated to assist in defining operating conditions for the feed concentrator.

Continuous Phosphate Glass Design Verification Tests. The second phosphate glass DVT experienced flowsheet and equipment problems. Extensive operating difficulty was encountered because of excess phosphoric acid. An error in communications with BNL resulted in the use of excess phosphoric acid. An M⁺/P ratio of 0.7 instead of 1.0 was used. Predominate difficulties encountered during the run were:

- a. Evaporator was difficult to control and operate.
- b. Air lift feeding to the melter was difficult to control. The rate was too low because the melter capacity (5-7 liters/day glass) was limited by heat transfer and the air lift eventually plugged with solids.
- c. Severe distortion of the melter lid occurred, and the off-gas flange cracked.

Development Spray Calciner. The EDL-102 spray calciner was placed into operation. The first run involved a PW-1 flowsheet and used air atomization. The equipment operated smoothly throughout the run. Inspection of the internal part of the calciner after the run showed the calciner barrel, spray nozzle, filters, and filter chamber to be relatively clean. The calciner cone surfaces were clean with the exception of the portion directly under the calciner barrel. At this point a calcine stalagmite approximately 15 inches high and containing approximately 3 kg of calcine had formed. Several unsuccessful attempts were then made to resume operation with steam as the atomizing fluid. The nozzle was removed from the unit, and it was determined that the nozzle had an internal leak caused by a missing internal weld. This resulted in leakage of liquid into the gas side of the nozzle. This could account for the poor atomization experienced during the air atomization portion of the run.

Alternative Melter Development. A spoolpiece is being fabricated which will permit the testing of batch melting in the receiver pot below the EDL-102 spray calciner. The corrosion rate of 304L stainless steel immersed in a PW-2 melt at 800 C was measured at approximately 135 mils/mo; this is low enough for a batch melting.

Waste Solidification Engineering Prototypes (WSEP)

The first USA conversion of highly radioactive liquid waste to solids by pot calcination was performed in the Waste Solidification Engineering Prototypes (WSEP). A total of 850 liters of sulfate-bearing PW-2 waste containing approximately 85,000 curies of radioactivity (0.7 watts/liter), was fed to the pot calciner in 43.7 hours. This radioactivity included about 9,200 curies of ruthenium-106 (Ru-106). The waste was chemically adjusted by

adding 0.3 M Na⁺ to the feed and by adding 0.25 M Ca⁺² in 88 liters of solution as a separate stream to the pot. These chemical adjustments were made to tie up sulfate and minimize its volatilization from the calciner during the 19-hour pot drying period.

After termination of the pot feeding and drying steps, the pot was cooled by forced air to 250 C at the pot centerline over a 17-hour period and removed from the furnace. A leak test cap was then welded on and further tests are now being performed. These include pot leak testing, ultrasonic pot wall thickness testing, gamma scanning at various points on the pot, and calorimetry testing. The maximum pot temperature with the pot hanging in air is presently 140 C at the pot centerline.

From the PW-2 waste, 151 kg of calcine (approx. 105 liters) containing approximately 83,300 curies of radioactivity was obtained. This should produce an internal heat generation of about 5.7 watts/liter in the calcine. The 1700 curies of activity that were collected in the two pot condensate streams consisted primarily of ruthenium and accounted for approximately 15% of the radio-ruthenium in the feed. The final condensate stream from the nitric acid fractionator contained approximately 10⁻⁴ microcuries/cc of ruthenium activity and was 0.04 M nitric acid. This level of radioactivity meets maximum permissible concentration limits for drinking water.

GE-412 Computer Studies

Run PC-1 was monitored and data logged throughout the four-day run. Several program changes were made while the run was in progress to correct minor calculational errors; all but four of the sixty values logged were valid numbers. Following this run, the data log format was reviewed, and an improved output format for the next pot calcination run was established.

The spray calcination data logging program is now being modified to conform with current program requirements. This work is targeted for completion prior to the spray calcination run, SC-1, scheduled for December 12.

The cause of the "Core Parity" problem which was described last month has not been positively identified, but certain physical operating procedures have successfully kept the computer operating without a "Core Parity" failure for the last three weeks. The cause of the previous failures appears to be that the sense amplifier biasing voltages are temperature sensitive, and careful control of the van and computer temperature environment is required.

Pot Leak Testing, PC-1 Pot

Initial bubble tests on the PC-1 Pot (15 psig internal pot pressure with nitrogen) indicated severe weld porosity at two points in the cap weld. The joint was cleaned by motor-driven stainless steel wire brush and rewelded (12 volts, 167 amps, 12 inch/min). The second bubble tests, as well as

pressure drop-out tests (pot pressure holding at 15+ psig for 16 hours at this writing), indicate that the reweld was successful. Results indicate that more efficient methods are required for cleaning the weld joint after processing.

Vacuum leak testing on the pot will proceed following checkout of the vacuum leak test system.

Solids Storage Environmental Testing

Approval for the CPFF contractor to initiate fabrication of environmental test pods was received. Field letters of instruction have been transmitted to commit about \$35,000 in fabrication funds. This initial effort is expected to provide two pods for B-Cell installation by March 1, 1967, plus certain auxiliary components.

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