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**PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
FOR MAY 1967**

**DIVISION OF PRODUCTION
AND
HANFORD PLANT ASSISTANCE
PROGRAMS**

JUNE 1967

MASTER

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C-65, Plutonium Production
(Special Distribution)

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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
MAY 1967

Division of Production
and
Hanford Plant Assistance
Programs

by the
Staff of Battelle-Northwest
S. L. Fawcett, Director

June, 1967

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PACIFIC NORTHWEST LABORATORY

MONTHLY ACTIVITIES REPORT

DIVISION OF PRODUCTION AND HANFORD PLANT ASSISTANCE PROGRAMS

R. S. PAUL

DIRECT AEC SPONSORED PROGRAMS

Summary

Columbia River Studies

The analysis of the physical factors covering the size and temperature gradients in effluent plumes has produced an appropriate correlating equation.

Mechanisms of Environmental Exposure

Children in grades six through two were measured in the whole-body counter stationed at the seventh and final Richland elementary school during May. The measurements of children in grade one during the first week of June will complete this phase of the study.

Work continued on the statistical survey of fishing license holders in the Tri-City area.

U235 Analyzer

The AEC Division of International Affairs continued field tests in Europe and South America on the portable analyzer and spectrometer that was developed for measuring the U²³⁵ content of unirradiated reactor fuel elements.

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DIRECT AEC SPONSORED PROGRAMS

Columbia River Studies

Effects of Reactor Effluent on the Quality of the Columbia River Water

(R. T. Jaske, Environmental and Radiological Sciences Department)

A report summarizing field measurements and the development of a mathematical model capable of predicting size and temperature gradients of the Hanford effluent plumes is in draft form. While the correlation equation contains several exponential factors, the use of average values within the data range produces a simple linear relationship of downstream difference as a function of distance. This is in substantial agreement with independent studies and conclusions reached by Tibby in his work for the State of California using marine discharges. In the summary report of this latter work, it was concluded that the simple expression obtained was sufficiently flexible to be used as a model for describing the concentration distribution of a quasi-steady state dye plume. The equivalent form of the Hanford equation is:

$$\frac{\text{Maximum } \Delta T \text{ in River}}{T_{\text{effluent}} - T_{\text{river}}} = \frac{150}{x} \text{ for } x = 1200 \text{ to } 6000'$$

Mechanisms of Environmental Exposure

(J. F. Honstead, Environmental Studies Section)

During May, measurements of radioactivity in school children were begun at the seventh and final Richland elementary school using the new mobile whole-body counter. The measurements have proceeded on schedule in spite of several instrument malfunctions. The measurements are scheduled for completion on June 5. The problems with the high-speed printer, which arose in April, have been resolved and the counting results are currently being printed instead of punched on paper tape. The results previously punched on tape will be run through the printer after the counting is completed in June.

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Several attempts at repairing the multichannel analyzer to print automatic subtraction of a pre-measured background during the whole-body counting of each child have been only partially successful. It is anticipated that this problem can be corrected in June after the mobile counter is returned to the 300 Area Laboratory from the school. Excellent cooperation has been obtained from school personnel in modifying counting schedules to compensate for periods of equipment malfunction.

Work continued on the personal interviews of fishing license holders in the Tri-City area by employees of the State Game Department. These interviews are a follow-up of the statistical survey of fishermen along the Columbia River shore, completed this spring by the Game Department.

U²³⁵ Analyzer

(W. G. Spear, Instrumentation Section)

Field tests on the portable gamma spectrometer continued with inspection trips in both Europe and South America by the AEC Division of International Affairs. In early June, the instrument will be returned for future laboratory review regarding noise interference problems. The instrument was developed to monitor the U²³⁵ enrichment of unirradiated fuel elements and for general gamma spectroscopy.

ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Summary

Mission 1

A test plan is being prepared for the study of anisotropic growth phenomena of hot-die-sized aluminum-canned fuel.

Uranium specimens electroplated with from 6 to 16 μ of nickel showed many pores when heated in air, even with prior formation of a uranium-nickel diffusion bond.

Attempts to occlude silicon in nickel electrodeposits by plating onto a vertical surface from a bath containing suspended silicon particles have had little success. Extensive silicon occlusion on a horizontal surface was achieved.

Conversion of a UT-2 tester to a uranium billet tester continued. Prototype circuitry for crack detection in such billets has been successfully operated and final fabrication will be accomplished upon arrival of vendor supplied components.

Fabrication of twenty high-resolution, high-frequency transducers for testing the internal cladding on co-extruded fuel elements is in process.

A method was chosen to integrate the hydrodynamic equations describing the coolant boilout following a generalized loss-of-coolant incident. This method has been programmed for digital computer solution and the debugging of the code is under way.

Mission 4

Equipment is being assembled for development of techniques for the impregnation of graphite rods with NpO_2 .

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Mission 5

Aluminum clad bismuth slugs and process tube sections were fabricated for PCTR tests. Aluminum alloy 1100 cans 3.75 in. OD were fabricated by back extrusion. "Buffer zone" elements were prepared by casting bismuth directly into cans, machining to length, and welding end caps. "Center zone" elements were prepared by machining bismuth castings and shrink fitting in aluminum cans. Process tube sections were prepared by drawing 6061 aluminum alloy tubing and welding end plates.

Mission 6

Fifty neptunium target elements, including five quick-turn-around targets, were fabricated for irradiation in a K Reactor in support of the Pu²³⁹ demonstration program.

Mission 7

The in-reactor phase of experiments on both a 5x5 array and a 6x6 array of test cells in the PCTR, with 2.1% enriched uranium fuel and 2.93 wt% lithium in aluminum target in a 3-to-1 ratio, was completed both wet (water coolant in process tubes) and dry (no water coolant). End effects of center cell buffers were measured as a function of test cell length. The reactivity effect of the exposed surface difference between removal of a square center cell (6x6 array) and a T-shaped center cell (5x5 array) was also measured.

The bismuth uranium supercell test using a 3.66 in. diameter bismuth target element and standard KVE (0.947 wt% enriched) fuel element will commence in the PCTR next month.

Preliminary calculations to statically simulate melting and dispersion of the driver fuel after water loss were completed for the K-lattice "black target," or alloy-aluminum driver, supercell over a wide range of driver enrichments for both 3-to-1 and 2-to-1 driver-to-target ratios.

Radiometallurgical study of selected cracked and uncracked high power density fuel alloy elements continued. Some macro-cracks detected by nondestructive testing methods in reactor basin were concluded to have existed in reactor but detailed metallography has not yielded causes for the cracks.

Melting was completed for fifteen of twenty-one alloys that are being prepared for evaluation of aluminum alloys resistant to ledge and groove corrosion. The cast ingots will be extruded to outer and inner tubing for KSE fuel cladding and evaluation.

A bibliography of Al-Ni-U bonding was prepared from an extensive literature search. Ingots of Al-10.5 wt% Si alloy and X-8001 alloy were made in preparation for fabrication of duplex cladding tube.

Wire of eutectic AlSi composition was fabricated to provide material for spray coating. Additional aluminum components were coated with zirconium by electron beam evaporation.

Formation of solid materials in the plating bath has been observed in studies of the electroplating of zirconium from fluoride salt melts at low current densities.

Mission 8

The test disposal of reactor effluent to ground at 100-D Area continued during May.

Mission 10

Removal of parent isotopes by water treatment at pH 6.6 and +10 mv zeta potential was more nearly complete than was obtained under other experimental conditions employing the sulfate system of water treatment.

The charge density on a positive iron colloid added to process water was insufficient to maintain a positive potential after adsorption of negative species during passage through a reactor. This resulted in an increased film deposition and

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increased the concentration of radioisotopes in the reactor effluent water.

Further work on in-reactor arsenic adsorption and activation using an analog computer has shown that for arsenate ions, the ratio of the concentration in the coolant to that on the surface film is 0.00456 cm^{-1} . This is in reasonable agreement with independent experiments.

The infrared scanner to map thermal differences in the Columbia River model at Washington State University is now being fabricated. Its use on the model is scheduled for mid-June. A project display for the scanner data has been developed to aid in its evaluation.

Process Technology

Examination of Zircaloy process tubes used with low quality anodized aluminum spacers did not show any hydrogen pickup in eight months of service. Examination of a C-Reactor Zircaloy process tube in service for 55 months showed a moderate amount of hydrogen pickup with a maximum of 512 ppm at 24 in.

Analysis of coupons associated with the second of a series of tests to investigate the effect of continued reactor exposure on the hydride distribution of K-Reactor process tubes was completed. Results indicate that eight months of operating exposure under conditions which prohibit the introduction of hydrogen by cathodic charging resulted in no obvious significant change in coupon hydrogen distribution or concentration. Exposure under such conditions results in no apparent migration of hydrogen from the dense case on the inner surface of the tubes into the underlying material.

Fuel elements with various experimental nickel plate coatings were charged into an out-of-reactor loop for corrosion testing.

Electrically-monitored corrosion probes in the B-Reactor have not shown large differences between the two different dichromate concentrations under test.

Stored-energy measurements were completed on core samples from a graphite tube block in F Reactor. The maximum stored energy found was 466 cal/g. A linear increase in stored energy with increasing c spacing was not observed.

Eight manometers which will allow headloss measurements to be made throughout filter beds have been added to the laboratory pilot plant. An alternate method of determining backwash efficiency is being evaluated.

Field evaluation studies were initiated on the experimental miniature dose rate instrument, which provides ranges from 10 to 300 mR/h with selectable signal alarm points.

Maintenance of Facility Capabilities

Transport theory was used in place of diffusion theory to check calculations regarding flux peaks in holes in the old reactors. Diffusion overestimates by 50% in most cases. Correction of an earlier diffusion coefficient error requires increasing enrichment by 40-50% and doubling the water jacket or screen thickness to obtain the previously-reported peak fluxes.

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ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Mission 1 - Basic Production

Fundamental Study of Growth Behavior in Aluminum-Canned Uranium Fuel

(J. H. Hoage, Metallurgy Design Unit)

Work continued on the study to develop a better fundamental understanding of anisotropic growth phenomena in aluminum-canned fuel. This study will correlate preirradiation measurements of uranium structural parameters with irradiated behavior for possible use in future process changes. A detailed test plan describing sampling techniques and test procedures is being prepared. The preparation of the production test and procurement of test fuel elements has been started by Douglas United Nuclear.

Fuels Development, Uranium Nickel-Plating Studies

(J. R. Lundquist, Chemical Separations Unit)

Nickel plating of uranium is being investigated as an alternate method of obtaining a thermal bond between uranium and an aluminum can. The plated uranium piece will be canned in aluminum before heating for the hot die sizing process. Uranium specimens plated with from 6 to 16 μ of nickel showed many pores when heated in air, either with or without prior formation of a diffusion bond. Uranium oxide formed in the pores and the thinner plates disintegrated. Similar specimens showed no evidence of uranium oxide formation in the pores when they were placed in a very close fitting aluminum jacket and capped before heating. Therefore, in the hot die sizing process the limiting factor does not seem to be porosity, but only the amount of nickel which diffuses into the uranium during the life of the fuel element in the reactor.

Fuels Development, Occlusion of Silicon in Nickel Deposits

(J. R. Lundquist, Chemical Separations Unit)

Preliminary experiments were carried out for occluding

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silicon in nickel deposits. A Watts bromide plating solution was used and 0.2 g/ml of silicon (325 mesh) was added. The silicon was kept in suspension by a magnetic stirrer. Several mils of nickel were deposited with the cathode in a vertical position but very little silicon was occluded in the deposit, even when the cathode was stationary. Ultrasonic agitation provided no substantial improvement. The cathode was then placed in a horizontal position and the silicon particles suspended by rapid stirring. The stirring was then stopped and the particles allowed to fall on the surface while plating. As expected, this technique caused large quantities of silicon to occlude in the plate. It is hoped that this technique can be modified so as to adapt it to cylindrical surfaces.

Fuels - NDT Test Development Support

(T. J. Davis, NDT Systems Engineering Unit)

Work continued on UT-2 modifications for crack detection in uranium billets. Prototype circuitry was assembled and evaluated and final fabrication of electronics and transducers is awaiting the arrival of components on order.

Assistance was provided in the testing and correctional modifications of new circuit boards for the UT-2C crack tester.

Electrolytic test containers for DUN fuel configurations were received, and compilation of electrolytic characteristics for a selection of fuels is expected to begin immediately.

Transducer Development*

(N. E. Dixon, NDT Methods Development Unit)

Fabrication of 20 high-resolution, high-frequency transducers for testing the internal Zircaloy cladding on co-extruded fuel elements is nearing completion. One transducer, which has been completed and evaluated, met the sensitivity

* This work supported equally by N-Fuels Section, General Electric Company.

and resolution requirements. The other 19 are being mounted in preparation for final in-service evaluation. Four of the initial 24 assemblies were rejected due to hot leadwire unbonds, shorting of electrodes, or fracture of the crystals in milling. Evaluation of other potential applications for these transducers is planned.

Reactor Boilout Study

(D. S. Rowe, Thermal Hydraulics Analysis Unit)

In this study, the transient flow and the temperature and pressure behavior in a fuel column during coolant boilout is being investigated analytically and experimentally. Two specific possibilities leading to this coolant boilout are being considered--a complete plugging of the process tube inlet and a front header break. Initially, the front plugging possibility is being examined.

In the mathematical analysis of this case, progress is being made on the computer solution of the differential equations describing the hydrodynamic behavior of a process channel and downstream hardware. A method of numerical integration to solve these differential equations was selected and the formulation of the computer code was completed. Debugging of this code is under way.

In this debugging, an inlet break-type incident is being considered, because a large amount of unclassified experimental data is available regarding this type of incident. The boundary and initial conditions imposed on the solution were as follows:

Boundary Conditions:

inlet P	=	600 psia
rupture pressure	=	14.7 psia
inlet H	=	38 Btu/lb

Initial Conditions:

mass velocity	=	833 lb/ft ² -sec
ΔT in water	=	250°F - 70°F

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It was assumed that the heat generated within the constant area flow passage remained constant during the transient following the break.

The program predicted acceleration of the water to cover 5 times its original velocity in 0.0157 sec. It appears to handle the transition from a single phase region to a two phase region satisfactorily. There are some inconsistencies in the results, however. The sonic velocity calculation appears to be in error and a proper mass conservation does not seem to be made. These inconsistencies are being examined, and appropriate corrections in the computer code will be made to eliminate them.

Mission 4 - Plutonium-238

Neptunium Oxide - Graphite Target Elements (H. T. Fullam, Fission Product Chemistry Unit)

Work has been undertaken to prepare eight neptunium target elements, each consisting of a graphite rod impregnated with ca. 40 grams of neptunium (as oxide). The object is to minimize the production of Pu^{236} via the $\text{Al}(\gamma, n)$ route. Requisite equipment is being assembled and the graphite cores are being fabricated. The work will be conducted in a glove box.

Mission 5 - Other Isotopes

Bismuth Target Element Fabrication (W. E. Gurwell, Fabrication Metallurgy Section)

Aluminum-clad bismuth target elements and associated process tubing for reactivity measurements in the PCTR were in preparation. The 54 bismuth elements are 3.75 in. in diameter with a 0.047 in.-thick 1100 aluminum cladding. Ten $4\frac{1}{2}$ in. long "center cell" elements were prepared from cast and machined bismuth slugs, shrink fitted into the aluminum cans. Forty-four $6\frac{1}{2}$ in. long "buffer" elements were fabricated by casting the bismuth directly into the aluminum cans

using directional solidification. The bismuth was then machined to a length of 6.000 ± 0.010 in. and the closure end cap was welded in place. Process tubing was drawn to 3.99 in. outside diameter from commercially available 4 in. diameter by 0.083 wall 6061-T6 tubing. End caps of 0.090 or 0.270 in. thickness are being welded on one end of each of the 14 sections of process tubing. Some difficulty was experienced in welding the thin 0.090 in. thick caps into the tubing while maintaining length tolerances of ± 0.003 in. The 1100 alloy aluminum cans for the target elements were fabricated by back extrusion which produced clad tubing with one integral end cap. The integral end cap was machined to thickness before the bismuth was cast or shrink-fitted into the can. Some difficulty was experienced in maintaining uniform can wall thickness.

Each of the above fabrication processes was chosen based on the following criteria: (1) Stringent dimension control is necessary to assure the validity of the experimental results in the PCTR, (2) Cost, (3) An early completion date. Fabrication of the components was completed with the exception of support weldings.

Mission 6 - Pu²³⁸

Preparation of Neptunium Target Elements

(C. H. Bloomster, Fuel Process Development Unit)

Fifty neptunium target elements, containing two kilograms of Np, were fabricated using the casting process. The process has the high yield and short dependable fabrication cycle required for the quick-turn-around concept. The two major target fabrication problems associated with the quick-turn-around were the short time available for fabrication and the high radiation levels.

Five neptunium target elements were fabricated for irradiation in KE reactor, demonstrating the quick-turn-around concept. The neptunium was obtained from the March discharge of neptunium target elements from N reactor. Contact

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radiation levels from the 300 gram batch of quick-turn-around neptunium when available for target fabrication were ~ 20 R (beta plus gamma) and ~ 5 R (gamma).

The total processing time, from discharge to completed target elements, was 44.5 days, which was less than the original target of fifty days. The N reactor neptunium target elements were originally fabricated by the Materials Department last year. After irradiation in N reactor, the targets were disassembled by Radiometallurgy, chemically reprocessed by BNW Chemistry Department, and converted into neptunium oxide by Isochem. The five quick-turn-around elements were fabricated in two days, which was considerably less than the originally-scheduled seven days.

Mission 7 - Increased Reactor Irradiation Space

2.1% Enriched Uranium Separate Target PCTR Experiments (D. F. Newman, Heavy Moderator Reactor Physics Unit)

The in-reactor phase of experimental measurements on both a 5x5 array and a 6x6 array of test cells with 2.1% enriched uranium fuel and 2.93 wt% lithium in aluminum target in a 3-to-1 ratio were completed both wet (water coolant in process tubes) and dry (no water coolant).

The effect of exposing the ends of the front and rear buffer elements when the cell is removed was measured as a function of length using a short center cell (7.072 in. long) and the standard center cell (21.875 in. long). The correct mass of copper to achieve $k_{\infty} = 1$ was measured in the 5x5 array and the 6x6 array both wet and dry. Results of these measurements are tabulated below:

Test Array	Condition	Center Cell Length, in.	Mo ^{Cu} , g Cu	Mo ^{Cu} /l, g Cu/in.
6x6	Wet	7.072	86 ± 8	+12.1 ± 1.1
		21.875	65.0 ± 15	3.0 ± 0.6
5x5	Wet	7.072	58 ± 15	8.2 ± 2.0
		21.875	2.0 ± $\frac{60}{37}$	0.1 ± $\frac{2.7}{1.7}$
6x6	Dry	7.072	+65 ± 15	+9.2 ± 2.1
		21.875	-26 ± 28	-1.2 ± 1.3
5x5	Dry	7.072	-11 ± 11	-1.6 ± 1.6
		21.875	-237 ± 13	-10.5 ± 0.6

A consistent loss of approximately 9 grams of copper per inch was measured when the center cell length was increased from 7.072 to 21.875 in. Based on the approximate equivalence of 1.8 grams of copper per inch of center cell length = 1 mk, a reactivity decrease of approximately 5 mk is observed. As the length of the center cell is increased, the solid angle of neutrons in the void seen by the ends of the front and rear buffer elements decreases. A theoretical calculation (normalized to these experimental results) will be required to determine if the 21.875 in. long center cell is sufficiently long to make the end effect correction negligible.

Differences in the mass of copper that would be required to achieve $k_{\infty} = 1$ for a 5x5 array and a 6x6 array of test cells are shown in the preceding table. In matching the spectrum incident on the test lattice so that the distribution of neutrons incident on the central cells is typical of the distribution that would be present in an infinite array of such cells, the following matched gold cadmium ratios were measured.

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<u>Test Array</u>	<u>Condition</u>	<u>Gold Cadmium Ratio</u>
6x6	Wet	4.44 $\pm$ 0.06
5x5	Wet	4.47 $\pm$ 0.06
6x6	Dry	3.31 $\pm$ 0.03
5x5	Dry	3.30 $\pm$ 0.03

These results indicate no measurable difference in the neutron spectrum between the 6x6 and the 5x5 array. When the cell was removed from the test lattice the type and number of surfaces exposed to the void changed. In the 6x6 array eight surfaces were exposed, six uranium surfaces and two target surfaces, in a 3-to-1 ratio. The 5x5 array used a T-shaped center cell that exposed ten surfaces, nine uranium and one target. Since the exposed surface ratio with the cell out of the 5x5 array was not a 3-to-1 ratio, neutron streaming across the void to other uranium cells could have caused the cell-out reactivity to be too high. Such an effect would decrease the cell worth and hence the correct mass of copper to achieve $k_{\infty} = 1$. Preliminary investigation of the cell boundary fluxes measured both with the cell in and the cell out indicates that neutron streaming across the void in the 5x5 array will account for the observed differences. Based on the value of 1.8 grams of copper per inch of center cell = 1 mk, the decrease in k_{∞} from the 6x6 array is approximately 1.6 mk wet and 5.2 mk dry. In all cases, the change in reactivity on loss of water coolant was measured to be negative and small, as shown by comparison of wet to dry values in the preceding table.

Separate Large Bismuth Target PCTR Experiment

(A. D. Vaughn, Heavy Moderator Reactor Physics Unit)

A supercell experiment using uranium fuel and separate bismuth target elements (3-to-2 fuel-to-target ratio) in a graphite-moderated lattice with an 8-in. pitch has been scheduled for the PCTR. The fuel and target material plus the moderator, process tubes, and foil holders, were designed and ordered so that the experimental measurements can begin approximately June 1.

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The supercell is designed to approximate the effect of removing a 4-in. graphite hole bar from the two columns adjacent to a single coolant header in order to facilitate the insertion of large quantities of bismuth into the reactor.

Battelle Metallurgy has manufactured the target elements and the associated process tubes. Douglas United Nuclear has provided the fuel charges which are standard KVE cores. The placement of supports on the fuel and target material is under way so that the experimental measurements can begin as scheduled.

Highly Enriched Study Survey Calculations

(C. L. Bennett, Heavy Moderator Reactor Physics Unit)

Preliminary calculations to statically simulate melting and dispersion of the driver fuel after water loss were completed for the K-lattice "black target," or alloy-aluminum driver, supercell over a wide range of driver enrichments for both 3-1 and 2-1 driver-to-target ratios. The results for no coolant loss in the target cells are summarized in the following table:

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Static Driver Fuel Melt Simulation

<u>Case Description*</u>	<u>7 wt%</u>		<u>11 wt%</u>		<u>15 wt%</u>	
	<u>k_x</u>	<u>$\Delta k_x/k_x$</u>	<u>k_x</u>	<u>$\Delta k_x/k_x$</u>	<u>k_x</u>	<u>$\Delta k_x/k_x$</u>
Wet Driver	1.0487		1.1771		1.2469	
Dry Driver	1.0261	-2.2%	1.1551	-1.9%	1.2273	-1.6%
Driver plus Clad Melt	1.0246	-2.3%	1.1581	-1.6%	1.2278	-1.5%
Slump of Melt to Process Tube	1.0355	-1.3%	1.1695	-0.7%	1.2423	-0.3%
Uniform dispersion of melt into 1st 1/3 of cell graphite volume	1.0479	-0.1%	1.2177	+3.5%	1.3172	+5.6%
Uniform dispersion of melt into 1st 2/3 of cell graphite volume	1.1122	+5.7%	1.2798	+8.7%	1.3783	+10.5%

* Target cells were wet for all cases

150% Normal Power Density -3:1 Supercells - 0 Exposure

<u>Case Description*</u>	<u>11 wt%</u>		<u>15 wt%</u>		<u>20 wt%</u>	
	<u>k_{∞}</u>	<u>$\Delta k_{\infty}/k_{\infty}$</u>	<u>k_{∞}</u>	<u>$\Delta k_{\infty}/k_{\infty}$</u>	<u>k_{∞}</u>	<u>$\Delta k_{\infty}/k_{\infty}$</u>
Wet Driver	1.0019		1.0781		1.1341	
Dry Driver	0.9738	-2.8%	1.0500	-2.8%	1.1108	-2.3%
Driver plus Clad Melt	0.9766	-2.5%			1.1145	-1.7%
Slump of Melt to Process Tube	0.9892	-1.3%	1.0741	-0.4%	1.1359	+0.2%
Uniform dispersion of melt into 1st 1/3 of cell graphite volume	1.0376	+3.6%	1.1496	+6.6%	1.2426	+9.6%
Uniform dispersion of melt into 1st 2/3 of cell graphite volume	1.1019	+10.0%	1.2160	+12.8%	1.3086	+15.4%

* Target cells were wet for all cases

A region of interest appears around the 7 wt% or alloy drivers where, even if the dispersion of fuel throughout 2/3 of the graphite occurs, the control system may be able to keep the reactor shut down. The next studies to be made involve the effectiveness of the present control system in such a lattice with "black" targets.

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High Power Density Alloy Fuel Evaluation

(J. W. Goffard, Metallurgy Design and Fuel Materials Units)

Radiometallurgical study of five fuel alloys being evaluated for high power density fuel elements continued. Two selected elements of 16 which were determined by nondestructive test equipment to have cracked cores after tube exposures to 1700 MWd/T (KW Reactor) are being examined. Some of the cracks in these elements are believed to have existed in-reactor, but metallographic study, including electron microscopy, has not revealed swelling voids or other features which might be related to the cracks.

Seven elements including all five alloy compositions which NDT equipment indicated were uncracked were examined for macro-cracks. Elements representing two of the alloys have shown cracks; however, these cracks are believed to be artifacts from sample preparation and not preirradiation or irradiation-induced. The search for macro and micro cracks in these 12 selected alloy elements will continue.

Aluminum Alloys

(R. G. Nelson, Primary Fabrication Unit)

A development program was started which will lead to the evaluation of several aluminum alloys as can stock materials resistant to ledge and groove corrosion. Three alloy types will be investigated: Al-Ni-Fe alloys, the age-hardening alloys and the powdered-metal alloys. The Al-Ni-Fe alloys will be made with the X8001 base and with high purity low-silicon material to determine the effect of silicon on the corrosion resistance.

A total of 21 alloy compositions will be melted into 4½ in. diameter by 10 in. long ingots. Fifteen of these have been melted. Chemical analyses for alloy content and impurities have been performed on samples taken from the top and bottom ingot locations.

Alloys to be melted are as follows:

High purity, low silicon aluminum
X8001

Al + 0.64Ni + 2.1Fe + 0.1Ti (low Si base)

Al + 0.64Ni + 2.1Fe + 0.1Ti (X8001 base)

Al + 0.93Ni + 0.53Fe + 0.1Ti (low Si base)

Al + 0.93Ni + 0.53Fe + 0.1Ti (X8001 base)

Al + 4.8Fe + 7.0Ni (Powder Met)

Alloys to be remade because of "off chemistry" are as follows:

Al + 1.0Ni + 0.15Fe + 11.5Si + 0.84 Mg (Low Mg plus Cr contamination)

Al + 1.2Ni + 0.3Fe + 12.0Si + 0.66Mg (Low Mg plus Cr contamination)

Al + 0.64Ni + 2.1Fe + 0.1Zr, X8001 Base; Low Fe

The remaining eleven alloys are within 10% of the desired composition with normal impurity levels. The silicon content of the alloys made from high purity aluminum is 100 ppm compared to 600 ppm in the alloys made with the X8001 base metal.

Drawings of extrusion tooling for fabrication of K5E tube sizes were submitted to Technical Shops. Ledge and groove corrosion specimens will be made from this tubing.

Ni-U Bonding

(W. E. Gurwell, Fabrication Development Unit)

A bibliography of Al-Ni-U bonding neared completion; a critical analysis of the bonding system is being prepared. Both are the result of an extensive literature review.

Ten Al-10.5 wt% Si, and X8001 Al castings were made. These castings will serve as billet components for coextrusion of duplex tubing. The tubing that will be evaluated in the hot-die-size bonding process will consist of approximately 40 mils of AlSi and 15 mils of X8001 Al on the exterior, water

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cooled side. Theoretically, the AlSi layer in the clad element will be cathodic to the X8001 outer layer (the X8001 will corrode preferentially) and thus provide a secondary corrosion barrier.

Development of Coatings for AlSi and HDS Fuels

(R. V. Bowersock, Fabrication Development Unit)

A study to investigate the corrosion protection afforded to the external surfaces of aluminum clad fuel by various coating techniques is in progress. Coating methods under investigation are spray coatings and diffusion bonded Zircaloy-2 sleeving. One-eighth-inch diameter wire was fabricated from eutectic AlSi and will be used to spray coat X8001 aluminum dummies. Coating adhesion and corrosion resistance will be evaluated by autoclave testing.

Further attempts to provide a thin coating of pure zirconium on the OD of standard aluminum components by electron beam vapor deposition have had limited success. More uniform bonding has been achieved by improved surface preparation but the as-deposited material has a dark slate grey coloration presumably from oxidation of the extremely reactive surface.

Installation of the AIRCO Heliweld seam welder relocated from 325 Building was started. This welder will be used to fabricate sleeve material from 5 mil Zircaloy-2 sheet.

Zirconium Plating from Molten Salts

(R. W. Stromatt, Chemical Separations Unit)

Zirconium plates of excellent quality were applied to a copper substrate using a previously reported procedure. This month, two deviations from the procedure have been tested.

First, after noting that a peculiar and localized corrosion on the cell appeared to be near or in contact with the control thermocouple for the furnace, the location of this thermocouple was changed. In subsequent runs the corrosion has not been observed and the previously reported peculiar

square-wave-type current and voltage behavior has, with one exception, disappeared.

The second deviation was in the pre-electrolysis current density. In previous experiments, a current density of 200 mA/cm² was used. Upon reducing the current density to 100 mA/cm², a solid material was produced in the melt. Subsequent electrolyses increased the quantity of the solid. The solid was not dispersed in the melt and in one electrolysis a crust of solid formed on the surface of the melt extending radially from the cathode to the cell body, hence short circuiting the cell. The crust is not soluble in water but does react with acid and dissolve. Its identification will be attempted.

Other plates were obtained following the usual procedure, with excellent results. The current is very unsteady at 10 mA/cm² but not at 30 mA/cm².

Mission 8 - Nuclear Safety

Reactor Effluent Disposal

(J. R. Eliason, Geochemical and Geophysical Research Unit)

A trench near the 100-D Area retention basins received a flow rate of 20,000 gal/min of reactor effluent for a 20-day period during the month. Riverbank springs were sampled regularly, water levels in wells were determined, and temperatures of the ground waters were measured to determine the effect of disposal of large volumes of water to ground.

Mission 10 - Columbia River

Water Treatment Variables

(W. B. Silker, Water Reactor Corrosion and Chemistry Unit)

Removal of parent isotopes by water treatment at pH 6.6 and +10 mv zeta potential was more nearly complete than under any of the experimental conditions evaluated with the sulfate treatment system. The effluent radionuclide concentrations were relatively low but were still somewhat higher than existed

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during treatment at pH 6.6 and zero mv zeta potential. This parallels the results obtained with the nitrate system of water treatment.

Evaluation of activated silica has been in process for too short a period for any meaningful results to be obtained.

Deionized water of 1.5-2.0 megohm cm resistivity is now being supplied as coolant for two process tubes through a stainless steel transport system. This condition will continue through one fuel cycle to determine steady-state radioisotope concentrations attainable with this system.

Iron Colloid Addition

(W. B. Silker, Water Reactor Corrosion and Chemistry Unit)

A positively-charged iron colloid was added to the cooling water for two single-pass process tubes in an attempt to provide sites for nucleation of negative colloids formed from impurities in the coolant during its passage through the reactor. The colloid, added at a concentration of 0.5 ppm, imparted to the residual colloids a zeta-potential of +10 mv, which is a polarity and magnitude that should suppress deposition onto the positively charged fuel and tube surfaces. Upon passage through the reactor, however, a charge reversal occurred, with a resultant zeta-potential of -10 mv exhibited by the colloids in the reactor effluent. There was a significant increase in pressure drop across the two tubes throughout the test, resulting from deposition of these negative colloids. The ΔP across the zirconium tube increased 24 pounds during the seven days of addition, which is untenable from an operational standpoint. Effluent concentrations of the various radioisotopes increased somewhat over the values obtainable with optimized coolant pretreatment, reflecting increased radioisotope production due to the larger film inventory of parent isotopes.

The reversal of colloid charge was probably due to mutual coagulation with negatively charged components, just as was intended. The concentration of negative colloids, however, was

too large to permit a net positive charge on the combined species. In attempt to provide a more positive matrix upon which the nucleation of negative species can proceed, a cationic polyelectrolyte will be added to the cooling water after authorization to do so is obtained.

Effluent Control Program

(J. R. Divine, Advanced Systems Corrosion Unit)

In our recent reports we have been discussing the use of the analog computer to solve our model of the kinetics of activity buildup on the reactor process tubes, fuel elements and in the coolant. Two of the parameters used in the model are the coefficient (A) which describes the release of nuclides from film into the coolant and the coefficient (B) which describes the rate of transfer of nuclides from the coolant onto the film.

It may be shown that

$$B = aA + b$$

where a and b are constants at constant temperature, radiation flux and heat flux. Furthermore, a is related to the equilibrium coefficient, k, which defines the ratio of nuclide concentration in the coolant to that in the film. The term b is also composed of more basic terms, some of which are not presently obtainable. From the analog computer study, we have found that

$$B = 1750 A + 48$$

This value of a is equivalent to

$$k = 0.00456 \text{ cm}^{-1}$$

which is an average value for an in-reactor surface using arsenate as the test ion. An independent experiment at 20°C gave a value of k approximately equal to 0.0003 cm⁻¹. Thus, 0.00456 cm⁻¹ is not unreasonable inasmuch as the average temperature will be greater than 20°C.

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Further work is being done to break b into its component parts.

Thermal Mapping

(J. R. Eliason, Geochemical and Geophysical Research Unit)

The infrared scanner to map thermal differences in the Columbia River model at Washington State University is now being fabricated. Its use on the model is scheduled for mid-June. A project display for the scanner data has been developed to aid in its evaluation.

Process Technology

Low Temperature Zircaloy Hydriding. Process Tube Evaluation (B. Griggs, Water Reactor Corrosion and Chemistry Unit)

The hydriding of the Zircaloy-2 process tubes in the K-Reactors is currently being controlled by the use of anodized aluminum spacers. The anodizing prevents the electrical contact of the spacer with the process tube and mitigates against the corrosion process which furnishes the current for the hydriding. Process tube 4372 KE was loaded with low quality (reused) anodized spacers for eight months. Examination of the tube between 0 and 60 in. from the rear face showed no hydrogen pickup.

Process tube 4475-C, in service for 55 months in C-Reactor with aluminum spacers, was examined for hydrogen content. The pattern of hydride distributions was similar to that in the K process tubes but more spotty. Maximum found was 512 ppm at 24 in., which gradually declined to pre-exposure levels at 120 in. and beyond. Base metal hydrides ranged around 60 ppm.

Low Temperature Zircaloy Hydriding. In-Reactor Tests (W. K. Winegardner, Advanced Systems Corrosion Unit)

Analysis of coupons associated with the second of a series of tests to investigate the effect of continued reactor exposure on the hydride distribution in K-Reactor process tubes was completed. Results indicate

that eight months of operating exposure under conditions which prohibit the introduction of hydrogen by cathodic charging resulted in no obvious significant change in coupon hydrogen distribution or concentration. Exposure under such conditions results in no apparent migration of hydrogen from the dense case on the inner surface of the tubes into the underlying material (base metal).

Tests to investigate the effect of continued exposure on existing hydride distributions consist of the in-reactor exposure of pre-hydrided Zircaloy-2 coupons in either aluminum or stainless steel holders. Process tube hydriding has been attributed to galvanic contact between tubes and aluminum spacers; coupons are being exposed in contact with materials both anodic and cathodic to Zr-2 (aluminum and SS holders, respectively) so that the effects of exposure can be investigated under conditions conducive as well as those not conducive to hydriding.

The second set of holders discharged from KW-Reactor earlier consisted of one aluminum and one SS holder and attained 247 days of operating exposure prior to discharge. Each holder contained 12 case-hydrided coupons machined from two previously installed reactor process tubes. Material immediately adjacent to locations from which coupons were machined from tubes, termed side strips, was retained. The effects of exposure are investigated by comparing the hydrogen content of side strips to that found in appropriate coupons. In addition to hydrided coupons, each holder contained two control coupons machined from tubes with no in-reactor service.

Analyses of the hydrogen content of both control and hydrided coupons indicated that exposure in the SS holder resulted in no change in hydrogen concentration or distribution. Two of the 24 analyses suggested an increase in base metal concentration of previously hydrided coupons but this was attributed to factors such as sample nonuniformity or contamination. Analyses of hydrided coupons exposed in the aluminum holder revealed that, in most instances, the 247-day exposure resulted in an increase in base metal concentration.

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The average difference between base metal content of these coupons and hydrided coupons exposed in the SS holder was approximately 12 ppm. This corresponds to a rate of increase of only about 1.5 ppm per operating month.

Base metal concentrations of control coupons exposed in the aluminum holder was 51-55 ppm. Analyses of similar coupons from the first set of holders had previously revealed that 106 operating days exposure increased base metal content from an original 5 ppm to about 32 ppm.

Low Temperature Zircaloy Hydriding. Out-of-Reactor Tests
(W. K. Winegardner, Advanced Systems Corrosion Unit)

Out-of-reactor tests were continued to investigate cathodic charging of hydrogen in Zircaloy. Electrolytic cells of the test facility consist of platinum anodes surrounded by tubular Zircaloy-2 cathodes. The electrolyte currently being used is reactor process water at about 90°C. After 328 hours, current densities of approximately 12, 0.8, and 0.08 ma/cm² resulted in cathode hydrogen concentrations of 958, 664, and 328 ppm, respectively. No hydrogen pickup was observed after 53 hours exposure to a c.d. of 0.005 ma/cm². Cathode hydrogen content prior to exposure was 10-13 ppm. The hydrogen content of cathode material underlying any surface case formed during the above exposures will also be determined and exposure with a c.d. of 0.005 ma/cm² is continuing.

Fuel Element Cladding Corrosion
(R. D. Weed, Water Reactor Corrosion and Chemistry Unit)

An out-of-reactor fuel element corrosion facility (TF-18) was charged with 24 fuel elements with various surface coatings. The test is operating on once-through reactor process water at 130°C and 33 gpm. Elements will be discharged in June for visual examination.

Half Plant Dichromate Concentration Test

(R. D. Weed, Water Reactor Corrosion and Chemistry Unit)

Corrosion probes installed in B-Reactor to measure the corrosion of 8001 and 1100 aluminum alloys and 1020 carbon steel have operated satisfactorily. To date, the probes have indicated only small differences between the side operated at one-half ppm dichromate and the side operated at 1 ppm dichromate.

Stored Energy in F Reactor Core Samples

(D. E. Baker, Materials Compatibility Unit)

Stored-energy measurements were completed on graphite moderator core samples from a tube block in Channel 2296-F. The maximum stored energy was 466 cal/g within 0.060 in. of the tube bore. At increasing distances from the tube bore, the stored energy then decreased to 423 cal/g at 0.18 in. from the bore, 375 cal/g at 0.70 in., and 286 cal/g at 1.20 in. (outer surface of bar). The $\bar{c}$ spacing at the tube bore was 7.64 Å and decreased to 7.14 at the outer surface of the bar. The stored energy in the tube block did not increase linearly with increasing $\bar{c}$ spacing. In a central-zone tube block sample from Channel 2284-F, the stored energy was 225 cal/g within 0.16 in. of the tube bore and 77 cal/g at the outer surface of the bar.

A quantitative thermal analysis of graphite from the tube bore of 2296-F was performed with a differential scanning calorimeter. Measurements of the stored energy release were obtained as the sample was heated to 500°C at a rate of temperature rise of 20°C/minute. The energy release between 200 and 500°C was 60.5 cal/g or about 13% of the total stored energy in the sample.

Stored-energy measurements were also completed on NC-7 and NC-8 graphite samples. The NC-7 sample had expanded 19.8% upon irradiation at 800°C to 2.4×10^{22} nvt. The total stored energy in the NC-7 sample was 115 cal/g. The NC-8 sample was irradiated at 750°C to 1.97×10^{22} nvt. The stored

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energy in the NC-8 sample, which had a radiation-induced expansion of 2.8%, was 76 cal/g.

Water Filtration Mechanisms

(D. E. Olesen, Water and Wastewater Research Unit)

Eight manometers were added to the laboratory water filtration pilot plant to allow the measurement of headloss throughout the depth of the filter beds. Work is in progress to provide the capability to backwash at a range of constant water temperatures.

The procedure for measuring the efficiency of backwash was tested in the laboratory and found to be inadequate for its purpose, as no correlation could be established between this measure of efficiency and the filtration system performance. An alternate method of selecting the proper backwash efficiency is under test. This method employs a turbidimeter to continuously monitor the effluent backwash water turbidity. The object of the testing is to produce a set of curves that, for a given length of backwash, will give the wash rate which will produce the cleanest filter for the least amount of wash water. The effect of temperature on the selection of a wash rate will also be included.

To date, we have received or have notification of shipment of forty-one different polyelectrolytes for aid in coagulation and filtration. We will shortly begin a study to evaluate these products for application to water treatment.

Grit Suspension Studies

(A. J. Shuckrow, Water and Wastewater Research Unit)

Studies were conducted to characterize the aqueous waste suspension from the 100 KE grit blast tube experiments. Particle size analyses were performed on the Black Diamond Grit and on the Black Beauty Grit as well as on suspensions of the effluents from two of the tubes. It was found that the particle size of the suspended material was considerably smaller than that of the Black Diamond Grit used in the test.

Gamma spectra obtained with samples of effluent suspensions from the first and fifth tubes which were grit cleaned were quite different. The only gamma emitters found in the suspension from the fifth tube were Zr^{95} - Nb^{95} . In addition to Zr^{95} - Nb^{95} , Sc^{46} and possibly Zn^{65} were present in the effluent suspension from the first tube which was grit blasted.

In both cases, most of the activity was found to be associated with the solid phase. Filtration through 0.1μ membrane filters removed > 99% of the activity. However, the greatest portion of the activity was associated with particles in the $1-50\mu$ size range. The presence of colloids less than 0.1μ in size was detected by high speed centrifugation of effluent collected from the 0.1μ filter. The supernatant after centrifugation showed a further reduction in activity to one-fourth. Filtration of the grit suspension through 40 cm of soil reduced gamma activity in 10 column volumes to background levels.

Radiation Detection and Measurement Instrumentation
(E. M. Sheen, Instrumentation Section)

Field evaluation testing was started on the experimental miniature dose rate instrument, which incorporates an aural signal alarm. The instrument provides selectable ranges of 10, 30, 100, and 300 mR/h. The field evaluation studies will aid in determining future requirements for radiation protection instrumentation of this type.

Maintenance of Facility Capabilities
(E. A. Eschbach, Advanced Concepts and Analysis Section)

Certain of the calculations regarding methods to obtain flux peaks in holes in the old Hanford reactors were checked using transport theory in place of diffusion theory. The comparisons indicate that diffusion overestimates flux peaks by 50% in most cases. The comparison also revealed an error in the diffusion coefficients used to obtain the results reported earlier. In general terms, correction of this error requires increasing enrichments by 40% or 50% and doubling the water jacket or screen thickness to obtain the reported peak fluxes.

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On the other hand, the size of the flux trap and associated target may be increased by the same factors.

Diffusion calculations indicate that flux peaks of either fast or thermal flux to values of 10^{15} can be achieved in systems less than three feet diameter provided the heat produced can be dissipated. However, the device is essentially a critical reactor by itself and requires its own control system since interaction with the surrounding graphite reactor is only one or two percent. On the other hand, minimum control problems arise to produce flux levels of 10^{14} . In the thermal case, the target must be of low cross section and additional source neutrons may be produced in Pu-Al or highly enriched U-Al elements matched to the flux level and target requirements. In the fast flux case, this modest increase could be obtained by removing 50% to 75% of the graphite from the channels within two feet of the target (i.e., a minimum of 37 channels--a 7x7 array excluding three in each corner). Enrichment levels of 0.9% to 2% would be required depending on the target configuration.

Isotopic Data Tape

A minor discrepancy in spacing on the printout from the DECAY program was corrected. At the same time, an internal shift was provided to permit handling longer decay chains without exceeding dimensioned limits.

The decay information on approximately 1800 isotopes is ready for processing with the Battelle-Northwest nuclear data tape. This will then complete the basic information included on this tape about each isotope except for nuclear reactions. The information on nuclear reactions is quite limited in scope and it is hoped that some good theoretical approximations that will calculate the cross sections for different isotopes can be used. This would then allow the development of a simple burnup code that could include all of the isotopes and a large number of different nuclear reactions.

Input/Output Analysis

The preliminary steps to undertaking the I/O project were planned. The first step will be to set up a restricted matrix and fill in with the information now on hand. This preliminary work will define the form of the I/O matrix and the additional information desired.

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ASSISTANCE TO GENERAL ELECTRIC N-REACTOR DEPARTMENT

Summary

Mission 1

The recovered end closure of Failure #19 (end associated Mark I inner) is being metallographically examined for the cause of failure. Six radial cracks in the inner weld have persisted through about 60% of the end closure thickness.

Fuel swelling in a high exposure Mark I fuel element was determined to be 9.2% for the outer component and 5.2% for the inner component. Buckling occurred in the inner bores of both components with indications of circumferential flow of the fuel during irradiation to permit both thickening and thinning of fuel walls. A high density of uniformly-small grain boundary tears was seen in the central (hot) zones of the fuel.

Modification of the clad-to-core tester by the addition of RF gating circuitry continued and very successful results have been achieved to date.

Results of the development of a tester to determine support flexure for 12-in. fuel assemblies are very encouraging since the test results are repeatable. Application of the tester to 24-in. fuelelements as yet fails to produce repeatable results.

The effort to measure the stresses in the N-fuel extrusion press liner was successfully completed.

Simulation runs on the response of N-Reactor main header pressure to turbine trips showed that further analysis is needed to bring the model into agreement with plant behavior. These are the first N-Reactor runs to use the hybrid computer configuration in the model.

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Mission 2

An electrically heated model of a coproduct fuel column was completed and installed in the high pressure flow loop in the 189-D Heat Transfer Laboratory. A total of 25 sets of measurements was taken during zero heat generation in the test section completing the isothermal phase of the program. Experiments with heat generation in the test section were undertaken late in the current report period. Initially, a simulated tube power of 5650 KW was considered and six sets of measurements of pressure drop and flow rate were made at this power level. A maximum outlet quality of 3% was attained during these tests.

Aluminum end caps of the inner aluminum target cans have been found to be detached after the removal of the outer Zr-2 cans of the target assembly. Examination of some loose caps indicates that they may be detached by the mechanical operation of removing the outer Zr-2 can.

Seventy-two fuel elements were measured. The modified NEM Program is in use.

Relaxation tests run for 1000 hours on zirconium-2% niobium W-springs indicate no appreciable difference in load force compared to Zr-2 W-springs at comparable times.

Metallographic study of coproduct drivers with Fe and Si alloy additions and Fe and Al alloy additions continued. About two-thirds of the 3.6% ΔV in the Fe-Si fuel should exist as porosity, but optical metallography has not yielded evidence of porosity. Electron microscopy is being pursued to resolve the microstructure of both alloys.

Studies are being conducted to determine the effect of welding sequence on pinhole rejects on outer welds of driver fuel elements.

Mission 5

Recent changes in BARNS-II to permit numerical integration of resonance region contributions to group cross sections are still being debugged.

Programs received from Brookhaven recently have been compiled and are now on tapes. Program ETOM has been successfully executed for the isotope lithium-6.

Four exponential experiments were completed in the NPR exponential assembly. One, without control rods, was analyzed.

Two different methods of approach are being utilized in the gamma-heating study for the NPR Mark-II assembly. Both ANISN and QAD-B computer codes are being compared. Results are lacking at this time due to computer hardware problems.

Mission 8

A scope of work to study the detailed heat transfer effect from a MK II fuel element following a loss of coolant was finalized.

Electron micrography was employed to identify particulates in soil column effluent. Particulates in the simulated rupture effluent were clay particles $< 0.5\mu$ from the soil, smooth spherical particles 0.1 to 0.3μ dia (ruptured fuel element debris), somewhat larger irregular shaped particles of river-water and soil origin and introduced polystyrene balls (0.08μ dia).

Process Technology

Work was started to prepare high quality Be-Zr braze rings for evaluation. The process chosen is double vacuum consumable-arc melting with the second melt in a skull furnace to permit casting. The primary objective of the program is to produce sound material free of contamination by atmosphere, crucible and molds.

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To determine whether or not competing homogeneous reactions affect the graphite-SO₂ system, the reactions of CO and SO₂ and of CO₂ and SO₂ were investigated in the absence of graphite.

Preliminary work indicated that the high weight gains on an N-Reactor Zr-2 reactor pressure tube may be due to surface condition during autoclaving.

Samples exposed to a simulated decontamination cycle employing BNW Citrox solution with added iron showed less corrosion but similar surface deposition to similar tests employing Turco 4521 solution with added iron.

The necessary planning, training and mechanical preparations to conduct rupture tests in the IRP loop were completed.

Indications are that a trench or trenches may intercept much of the oil at 100-N Area moving with ground waters toward the river and permit its burning or recovery.

Work has started on the development of a convenient standard (a salt hydrate as a source of water vapor at a fixed pressure) for checking hygrometer calibrations.

ASSISTANCE TO GENERAL ELECTRIC N-REACTOR DEPARTMENT

Mission 1 - Basic Production Mission

N-Reactor Fuel Failure Analysis

(J. W. Goffard, Metallurgy Design Unit)

Metallographic examination of the closure of inner "end associated" Failure #19 is being pursued. This end cap had been missing for about a year and was recently discovered and recovered from N basin. Previous visual examination of the failed component yielded no clues to the cause of failure. The first metallographic section of the closure (0.01 in. below the outside end surface) showed seven radial cracks in the inside Be-Zr eutectic weld bead extending from the Zr cap to the surface of the weld bead. These cracks were oxide-filled and several of them appeared to show two distinct zones of oxides. Six of these cracks have persisted through the weld bead and braze remelt zone to a depth of 0.13 in. The inner and outer clads and the cap itself have shown no manufacturing or material defects, and there have been no deficiencies noted in the outer braze-weld zone. Examination is continuing through the remaining thickness (about 40%) of the closure.

Examination of High Exposure Mark I Assembly

(J. W. Goffard, Metallurgy Design Unit)

Study continued of an initial load production test Mark I fuel element assembly from a tube that attained 5300 MWd/T average exposure.

A fuel sample from the center of the outer component yielded a density of 17.32 g/cc (preirradiation density, 18.92). This postirradiation density value corresponds to 9.2% fuel swelling. A fuel sample from the center of the inner component yielded a density of 17.99 g/cc which corresponds to 5.2% fuel swelling. Metallographic study of transverse sections from the center of both components indicates that the clads are well bonded to the fuel and the bonds have not been altered or damaged by the fuel and clad deformation

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that occurred during irradiation. Uranium metallography (optical) on both inner and outer components revealed a dense population of uniformly-sized grain boundary tears in the center or hotter zones. This type of porosity existed throughout the fuel section but in a diminishing amount in regions near the fuel surfaces. There is no aligned porosity evident, presumably because the maximum fuel temperature in these components was less than 450°C.

All four clad surfaces of the two-component assembly were evaluated for corrosion oxide accumulation and hydrogen content. Performance has been excellent in light of the fact that this assembly was exposed to the reactor environment since initial start-up. Oxide thicknesses were about 4μ on the outer surface of the inner clad and 2μ on the other three surfaces. Hydrogen analyses of all four clad samples were in the 40-56 ppm range.

Transverse rings of both tubular components were prepared for study of the buckling behavior of the inner bores. In a given cross-section of the outer component it could be seen that the inner clad was buckled at perhaps five sites with buckling being both inward (toward the tube axis) and outward (toward the fuel). In the inner component buckling was simpler with the development of an egg-shaped or oval inner bore. Where the buckling was inward, the fuel section was thickened and where buckling was outward, the fuel section was thinned. These local changes in section thickness in the fuel of necessity were accompanied by circumferential plastic flow of the fuel. In the outer component a maximum thickening of the fuel wall of 32% or 0.085 in. and a maximum thinning of 6% or 0.017 in. was measured. In the inner component a maximum thickening of the fuel wall of 22% or 0.074 in. and a maximum thinning of 11% or 0.037 in. was measured. Evaluation of these high exposure Mark I fuels is continuing.

NDT Routine Tester Development Support
(T. J. Davis, NDT Systems Engineering Unit)

Installation of RF gates in the clad-to-core tester chassis continued. Two 20-volt threshold gates were installed in the

through-transmission testers and the units are functioning properly. Four adjustable level gates are being fabricated for installation in the clad-to-core testers.

A chevron end test for inner fuels was set up and its performance has been satisfactory.

Fuel standards with the new, thinner cladding thicknesses are being manufactured and should be ready for test evaluation soon. Testing of a 30 mHz transducer indicated that the unit will be capable of resolving voids beneath 15 mils of cladding.

Vibration Test

(L. T. Lamb, NDT Systems Engineering Unit)

Development efforts to establish a vibration test for N-Reactor fuel assemblies continued. Three 24-in. fuels and three 12-in. fuels were assembled with cracked supports. The cracked supports gave test indications similar to those resulting from loosened supports. Weak (loosened or cracked) support indications were reliably obtained for the 12-in. assemblies; however, for some orientations of the 24-in. assemblies, the assemblies tested "good", i.e., no weak support indications. The degree of support cracking was not determined but cracked supports were not reliably detectable.

The mechanical fixture for the production tester was designed and is being fabricated. The electronic portion of the tester is now complete.

Extrusion Press Liner Stress Evaluation

(P. H. Hutton, J. F. Dawson, NDT Methods Development Unit)

Stresses in the N-Fuels extrusion press liner were measured during three separate series of extrusions using special length billets. This work is in support of an evaluation of the capability of the press to accommodate a billet somewhat longer than the standard. Although the high temperature environment (800°F) created problems in obtaining strain measurements during the extrusion operation, a significant

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number of consistent measurements was obtained. They showed the stress levels to be quite nominal--less than 5000 psi--at the unsupported outer end of the liner.

N-Reactor Simulation

(R. D. Benham, Systems Analysis and Simulation Unit)

Analog computer runs were made with the "overall N-Reactor" to study the effects of certain parameters on the main header pressure response to turbine trips. These studies were a continuation of earlier work last reported in March. In this particular series most of the parameters adjusted were concerned with the dump condensers and included: dump condenser outlet friction coefficient, dump condenser condensation rate and the pressure differential across the dump condenser. Detailed analysis of the main header pressure transient studies will be required to bring the pressure response properly in line with plant results.

Some hybrid computing was accomplished on the N-Reactor model. The generation of non-linear functions was performed by the PDP-7 digital computer, replacing all the analog diode function generators in the model.

Mission 2 - Coproduct

Hydraulic Characteristics of a Coproduct Fuel Column

(J. K. Anderson, Thermal Hydraulics Analysis Unit)

The purpose of this study is to experimentally determine the hydraulic characteristics of a coproduct (MK II) fuel column under both liquid and two-phase flow condition. This information is important in the analytic determination of the flow stability of reactor coolant channels under a variety of standard and non-standard operating conditions.

An electrically heated model of the downstream two-thirds of a coproduct fuel column was completed and installed in the high pressure flow loop in the Heat Transfer Laboratory. Detailed experimental procedures (run plans) were formulated

and experimentation was started.

The run plans divide the experimental program into two phases. In the first phase, flow, pressure drop, and temperature measurements will be made with no heat generation in the simulated fuel. Coolant temperatures ranging from 150 to 550°F (65 to 288°C) and flow rates from 25 to 250 gallons per minute will be considered. The purpose of this phase is as follows:

- Provide a thorough check-out of instrumentation and equipment.
- Establish the single phase flow characteristics of the experimental model for comparison with the actual reactor configuration.
- Determine heat losses from the model, information to be used to correct data obtained later during boiling conditions.

In the second phase, heat will be generated in the simulated fuel by electrical resistance heating. Pressure drops across the two coolant subchannels in the fuel column model, the support charge, outlet nozzle and the outlet jumper will be measured at constant heat generation as the coolant flow-rate is reduced in steps. The experiment will be terminated before conditions are reached which can be expected to produce boiling burnout.

Twenty-five sets of experimental measurements were taken during zero heat generation completing Phase 1 of the investigation. Phase 2 was started by considering a reactor tube power of 5650 KW. Because of the shorter length of the electrically heated model, this reactor tube power corresponds to a model power of 3860 KW. A total of six sets of measurements was made at this power. Outlet steam qualities up to 3% by weight were obtained. Following these measurements, it was necessary to interrupt the experimentation because of the unavailability of electric power. When power is again available, the experiments will be resumed.

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Examination of Coproduct Drivers
(J. W. Goffard, Metallurgy Design Unit)

The examination of two 1.96% enriched coproduct driver elements from PT-NR-13 continued with the purpose of comparing the irradiation performance of fuel containing 150 Fe and 100 Si with the British alloy (350 Fe - 800 Al). The end closure (chevron cap) and braze heat-affected zone of the driver containing Fe and Si additions (exposure of 2946 MWd/T and fuel swelling of 3.5%) was examined. The closure appears to have performed well without any indications of inadequacies and there are no noticeable alterations to the end profiles as a consequence of the fuel swelling. Some transverse macro cracks seen in the fuel of the closure end are believed to be artifacts from sample preparation. There are a few micro cracks near the center of the fuel wall, however, that may be irradiation-induced. Although the fuel experienced a 3.6% ΔV , porosity was not optically observable in the microstructure. A replica was prepared for electron microscopy.

Metallographic study was also made on a transverse wall section from the center of an adjacent position driver element containing Fe and Al additions which had attained an exposure of 2872 MWd/T. Fuel swelling data of about 1% coincides with the expected 1% ΔV from fission product accumulation. Optical metallography has not revealed any porosity in the structure but replication and electron microscopy are being pursued.

Examination of Detached Caps from Coproduct Targets
(J. W. Goffard, Metallurgy Design Unit)

Some aluminum end caps from the inner aluminum target cans have been found detached from the aluminum can after removal of the outer Zr-2 cans of the target component. Examination of some of these caps in the Radiometallurgy facility indicated that separation from the can wall was entirely in the weld which joins the wall and the cap. The flange joint forming the closure area of the cap is deformed inwardly by the tube cutter resulting in weld joint failure. The inadvertent detachment of the aluminum caps results from the use of a tube cutter to remove the caps of the outer Zr-2 can.

N-Fuel Element Measurements

(R. K. Marshall, Metallurgy Design Unit)

During the reporting period, 72 production elements were measured.

Production data are being run using the modified NEM Program which references postirradiation data to preirradiation station locations.

Coproduct Target Support Development

(J. H. Hoage, Metallurgy Design Unit)

Results were obtained after 350 hours and 1000 hours in relaxation tests of zirconium-2% niobium W-springs. After 350 hours of testing the Zr-2% Nb spring is slightly stronger than the Zr-2 spring and after 1000 hours no appreciable difference could be detected between the springs. The test will continue to 2000 hours.

Closure Welding

(T. B. Correy, Welding and Joining Unit)

In an attempt to reduce the number of pinhole rejects in the outer welds of the driver tubes for the coproduct, a process work request was written to reverse the welding sequence by making the outer weld first and then the inner weld. The material has been processed but the data are not complete.

Improved Welding Power Supplies

(T. B. Correy, Welding and Joining Unit)

In order to improve the arc starting and crater filling ability of welding power supplies, a study has been going on jointly with GE and BNW using a 12-pulse rectifier system. A half-wave rectifier delivers two pulses of direct current in 1/60 second while a 12-pulse rectifier delivers 12 pulses in 1/60 second. The results to date indicate superior performance with positive arc starting on aluminum using helium as a shielding gas and a 2% thoriated tungsten electrode. A

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recent test produced 20 consecutive starts at five amperes with a dirty electrode. Starting with a clean electrode under the same conditions the fifth start is difficult to obtain.

A manufacturer of rectifiers was found that has indicated an interest in building a current-handling unit of this type. A specification and purchase requisition has been written and delivered to GE for purchase of an experimental unit.

Mission 5 - Other Isotopes

Code Development

(J. L. Carter, J. L. Baker, Theoretical Physics Unit)

The recent changes in BARNS-II to permit numerical integration of resonance region contributions to group cross sections are still being debugged. As a convenience to HRG users, the NUTAPE part of BARNS-II was modified to provide an alphabetic, as well as numerical, listing of the nuclides on an HRG data tape.

The programs ETOM, CRECT, EDIT, Flange-II, and Retrieval Subroutines that were received from Brookhaven National Laboratory, were compiled, absolved, and placed on tape.

The program ETOM, which processes neutron cross section data from the ENDF/B Library Tape and produces library decks for the program MUFT4 and MUFT5, was successfully executed for the isotope Li^6 .

Buckling Measurements in an NPR-Like Exponential Assembly

(G. D. Seybold, Analytical Physics Unit)

Four exponential experiments were completed in the NPR exponential assembly. One, without control rods, was analyzed. The loading consisted of a 3/1 ratio of sixty standard process tubes containing 0.947 wt% enriched metallic uranium clad with Zircaloy-2 and twenty tubes of lithium-aluminum target material. The fuel is 23.3 in. Mark-I tube-in-tube NPR geometry. Aluminum process tube coolant channels were filled with deionized water.

Lithium-aluminum target tubes consisted of sixteen target increments giving a nominal test length of 96.96 inches. Each increment was 6.060 in. x 1.430 in. diameter. Three point fitting procedures were used for like-lattice cell positions. The results are listed below along with the same experiment where the loading was identical except coolant channels were dry.

Coolant Channels	Material Buckling $B_m^2 (\text{cm}^{-2}) \times 10^{-6}$	Error on Buckling $\sigma_{B_m}^2 (\text{cm}^{-2}) \times 10^{-6}$	Relaxation Distance $B_{11} (\text{cm})$	Extrapo- lation Distance $\lambda_z (\text{cm})$	Extrapo- lation Distance $\lambda_A (\text{cm})$	Extrapo- lation Distance $\lambda_B (\text{cm})$
Dry	-272.7	± 8.1	38.01473	0.752	5.78	3.84
Wet	-268.2	± 1.9	38.28778	8.230	5.97	4.71

λ_A is perpendicular to the process tubes (9 in. cell dimension)

λ_B is parallel to the process tubes

λ_z is perpendicular to the process tubes (8 in. cell dimension)

Uncertainties on the reported bucklings are a function of the number of measurements. The three control rod experiments have yet to be analyzed. The NPR assembly is presently being changed to the same configuration except that the target material will now be bismuth instead of lithium-aluminum.

Mission 8 - Nuclear Safety

N Mark-II Fuel Gamma Heating Studies

(G. E. Hanson, Heavy Moderator Reactor Physics Unit)

Since interest was shown in continuing and refining the NPR Mark-II assembly gamma heating calculations, two new computer codes are now being utilized: ANISN and QAD-B. The use of these two codes will, hopefully, reduce the range of the value of gamma heat deposited within the fuel (51.1-67.5%) which was calculated using the ISOSHL D code.

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ANISN is a one-dimensional S_n transport code with general anisotropic scattering that permits a white boundary condition with an albedo for each energy group. It will thus allow a detailed look at one cell without having to smear the surrounding cells for an accurate description of the system. It also allows a better geometric representation of the fuel assemblies by using shell sources.

QAD-B is a point-kernel shielding program similar to ISOSHLD but allowing much more detailed geometry, including shell sources. It contains a gamma-ray cross section library. It does not allow a reflecting outer boundary but does allow dose points to be located within the various shield regions.

Some trouble has been experienced in running ANISN due to computer hardware problems. The long binary deck seems to present a problem in going cards-to-tape. Work is progressing on putting the entire program on tape. QAD-B input is also being prepared at the present time.

Fuel Temperatures Following a Loss-of-Coolant
(J. K. Anderson, Thermal Hydraulics Analysis Unit)

The detailed scope of this work was finalized.

The objective is to obtain experimental heat transfer information regarding the N-Reactor fuel temperature transient following a hypothetical loss-of-coolant accident. Preliminary analysis of fuel melting times following a primary system depressurization has shown that radiation is the predominant mechanism of heat transport over much of the temperature transient. Therefore, accuracy in specifying surface emissivity values is a necessary requirement for the prediction of melting times. Unfortunately, it is not possible to predict accurately the instantaneous emissivity of a Zr-2 surface in an oxidizing (steam) atmosphere at elevated temperatures because of the complexity of the oxide formation process. Estimates of emissivity values during a 500 to 2000°F transient indicate a possible range of values of 0.3 to 0.7. Therefore, single value estimates of emissivity could produce errors of 40 percent or greater in the rate of heat loss from

the fuel at elevated temperatures. Errors of this magnitude are unacceptable in the N-Reactor safety analysis.

In this work, information on the overall and detailed heat transfer effect between the driver element of a N-Reactor MK II fuel assembly and the graphite moderator will be obtained. The combined effects of conduction, convection, and radiation will be considered. Emphasis will be placed on obtaining the exact behavior of the materials in the reactor during a temperature transient and, therefore, experiments will be performed using only reactor-grade materials with identical surface characteristics.

In the initial phase, only the heat transfer effect between the driver fuel and the pressure tube will be studied. The test apparatus planned for this phase will be adaptable to future studies of the heat transfer effect between the process tube and the tube block and for the experimental verification of the overall temperature transient following the loss-of-coolant incident.

Hydrogen Evolution from the Metal-Water Reaction of Overheated Fuel

(D. L. Reid, Reactor Engineering Section)

Two metal-water reaction rate tests were completed using unirradiated, autoclaved, Zircaloy-2 clad inner elements of N-Reactor Mark I fuel assemblies. Testing in a steam atmosphere was under isothermal conditions at 1000 and 1100°C. The data are reported for information only since the available experimental equipment was far from desirable for the information sought. The equations on volume of H₂ per unit of time listed in the table should not be used for serious extrapolations to reactor conditions until confirmed. The reported experimental values and equations are certainly within 50% and probably within 20% of the true values. Although the constants in the equations may not be accurate, the trends in the hydrogen evolution rate are of interest since they follow to some degree that which might be predicted from the nature of the ZrO₂ produced at various temperatures and the fuel distortion that occurs with time at temperature.

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Comparison of the empirical data with those calculated earlier shows the deviations from the extrapolated Argonne data obtained with polished coupons.

Volume of H₂ from Metal-Steam Reaction

(in cc/elapsed time)

Time Min	1000°C		1100°C	
	Volume H ₂		Volume H ₂	
	Exp.	Calc.	Exp.	Calc.
1	206	940	830	1790
5	840	2100	3440	4000
10	1370	3000	5360	5660
30	3000	5150	6320	9800
60	4690	7300	6850	13900
85	7370	8670		

For the 1000°C experiment, the rate function is parabolic for the first 35 min except for the first 5 min (deviation probably due to experimental conditions) and a cubic equation fits the data from 40 to 85 min at temperature. No previous information would predict that a cubic rate law would apply although an increase in the H₂ evolution rate would be expected.

A least squares fit to the data gave the following equations:

$$1 \text{ to } 35 \text{ min } V_{(H_2STP)} = -1.5 t^2 + 135 t + 158$$

$$35 \text{ to } 85 \text{ min } V_{(H_2STP)} = 0.016 t^3 - 2.19 t^2 + 170.6 t - 1128$$

where t = time in minutes and the original Zircaloy-2 surface area was 53.7 sq. in.

For the 1100°C experiments where the oxide is more dense and adherent than at 1000°C, a plot of the data showed three parabolic rates over the 60-min test period--one for the first 10 min, one for the next 25 min, and one for the last 25 min. Since the slopes of the lines for the last two periods were not extremely different, the last 25 min period was ignored.

The second equation, then, will produce higher H_2 volumes than that shown in the table at 60 min and will give a conservative value for time beyond 60 min.

$$1 - 10 \text{ min } V_{(H_2 \text{ STP})} = -28.7 t^2 + 816 t + 71$$

$$10 - 35 \text{ min } V_{(H_2 \text{ STP})} = -0.67 t^2 + 75 t + 4674.$$

Particulate Filtration in an N-Area Soil

(B. L. Carlile, Water and Wastewater Research Unit)

Samples of a simulated ruptured N fuel element waste, before and after passing through soil columns, were prepared for examination under the electron microscope. Photo micrographs were prepared and are now being screened for significant results. Magnification is such that particles of 100 Å in diameter can be distinguished.

Initial interpretation of the micrographs indicates considerable submicron material in the influent and effluent solution. There appear to be three major types of particulates eluting from the column, (1) clay particles of less than 0.5μ in size, (2) smooth spherical particles of 0.1 to 0.3μ diameter, and (3) somewhat larger, irregular-shaped particles ranging up to 1μ in size. In addition to these, many polystyrene balls (0.08μ) that were added to the column were observed in the effluent. The smooth spherical particles appear to be ruptured fuel element debris while the irregular shaped particulates probably originate in the soil column or the river water.

As expected in a filtration mechanism, the concentration of particulates was the greatest in the initial effluent samples and decreased as additional solution passed through the column. This is the result of two different mechanisms: particulates originating in the soil would be flushed out as additional solution elutes from the column and particulates originating in the waste solution would be adsorbed on soil surfaces, decreasing the pore size in the soil and resulting in retention of smaller particles.

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testing of two high exposure N-Reactor fuel elements. Special precautions are being taken because the elements have considerably higher exposure than any previously run in the IRP facility.

Two new operators were trained in the operation of the loop and acquired operating experience during four decontamination experiments conducted in the IRP Loop. The normal loop start-up and shutdown procedures were employed during the four loop runs.

The fuel receiving basin was prepared. A recently overhauled pump was installed in the loop to minimize any pump problems during the rupture tests. Other necessary minor repairs and adjustments were made and the loop operated for a shake down run of about 50 hours at 300°C. The loop operation was satisfactory. An auxiliary decontamination facility was installed and other precautions, such as additional shielding, pictures and floor marking were made in the event of a major problem.

Oil Disposition

(J. R. Eliason, D. D. Tillson, Geochemical and Geophysical Research Unit)

The leakage to the ground of about 80,000 gallons of diesel oil at 100-N area is resulting in a study to determine means of minimizing the effect upon the Columbia River. Up to 7 wells will be drilled to determine the distribution of oil in the ground. A trench system is proposed to collect such oil as moves toward the river.

Analytical Instrumentation for N-Reactor

(G. B. Barton, Fission Product Chemistry Unit)

The recorders to be used with the Panametrics hygrometer and the MSA carbon dioxide analyzers were received. A switching box needs to be prepared, then the instrument will be ready for installation in the control room at N-Reactor.

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A few measurements were made to establish the feasibility of using salt hydrates as a source of water vapor at a fixed pressure. This would be a convenient standard for checking hygrometers. It appears feasible to use sodium hydroxide for producing a gas of dew point near -20°C (which is a convenient level for application to the reactor atmospheres). Plans were made to determine the temperature coefficient of this system ($\text{NaOH-H}_2\text{O}$).

ASSISTANCE TO ISOCHEM

Summary

Mission 1

Using IR spectra measurements, a total G value of 0.95 was measured for the production of olefins by irradiation of NPH.

Design features were worked out for a metal high-vacuum system to be installed in a glove box in 325 Building.

Preparations continued for projected studies of the high-temperature thermochemical properties of certain actinide compounds. Current efforts are directed toward the preparation of pure $ZrCl_4$ and UCl_3 .

Scouting studies were started to determine if uranium feed derived from a Zirflex decladding procedure will have any deleterious effect on solvent extraction processing. Preliminary results indicated that the performance of the columns was normal in every respect with feed containing up to 0.07M Zr and the associated fluoride (with aluminum complexant added to prevent fluoride corrosion).

Use of the gamma-counting instrument developed for detecting Am^{241} in the waste line by spectrometric analysis of samples from the W-14 Column continued. Energy peaks at 17, 30, 60 keV with several minor peaks between 100 and 130 keV were revealed. Alpha-counting and gamma-spectrometer systems were planned for other in-line installations.

Flow equipment was received and final equipment assembly is nearing completion for the mass transfer studies.

Mission 3

A precipitation technique for the separation of Am and Cm is being tested.

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Mission 5

Essentially quantitative removal of polonium from irradiated bismuth metal by fused NaOH at 450-600°C was obtained in laboratory-scale experiments. Material balances were low; reasons for polonium losses are not fully understood.

Mission 6

C-Cell in the 324 Bldg. is being readied for installation of a one-slug dissolver. Difficult working conditions in the air lock can be improved by new design.

Mission 8

A further analysis was made of the data from PCTR experiments for determining the limiting critical enrichment of uranyl nitrate, $\text{UO}_2(\text{NO}_3)_2$, solutions. The results, which depend on the chemical analyses of the uranium solutions, now confirm the previously estimated value of 2.1 wt% U^{235} . An error analysis is under way to define the experimental uncertainty which is of the order ± 0.01 .

Particles airborne from plutonium oxalate-oxide and plutonium fluoride when heated in a stream of air were found to have mass median diameters in the range of 15-50 microns when air was swept over the material and through a vertical chimney over the sources at 100 cm/sec. The release to the air stream was largely through aerodynamic pickup, rather than through temperature effects per se.

Mission 9

Measurements of equivalent weight, density, viscosity, and refractive index were made on purified D2EHPA and compared with similar measurements on as-received samples from various sources.

Work continued on various aspects of the problems arising from the formation of fission product-bearing solids in the concentrated Purex acid waste.

Laboratory and hot cell support was provided to a series of Strontium Semiworks campaigns and plant tests.

Studies of synergistic effects in the solvent extraction separation of actinides and lanthanides were continued.

An electrolytic oxidation - D2EHPA solvent extraction process is being investigated for the separation of cerium from other rare earths.

Dilute sodium carbonate solution effectively washes mono(2-ethyl-hexyl) phosphoric acid from radio-chemically degraded D2EHPA B-plant solvent. A solvent washing procedure incorporating this observation has been used in the strontium semiworks.

An empirical equilibrium distribution coefficient (K_d) equation was developed which enables strontium K_d predictions to be made in soil-solution systems containing one or all of the following cations: sodium, calcium, potassium, and magnesium.

Eight loading and elution cycles of simulated Redox alkaline supernate waste and a recent lot of AW500 showed the recent material to deteriorate as readily as previous lots. Studies with dehydrated AW500 showed that a temperature of 84°C does not significantly damage the resin.

The construction of the model tank for study of circulation patterns was completed and tests are under way to define the optimum arrangement of air-lift circulators in the new tank farm.

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Process Technology

A few combinations of N-Reactor decontamination wastes and separations processes wastes produce significant exothermic reactions during pyrolysis. These can be suppressed by dilution with separations processes wastes.

A third run investigating all the process steps (decladding, dissolution, and metathesis) and using four 3-day autoclaved NPR fuel elements was completed. The run went smoothly and all of the fuel material was dissolved with the exception of a few scraps of Zircaloy-2 cladding. The next run will use 5.5M NH_4F and 90-day autoclaved NPR fuel elements. The analytical results were received for the first of the three runs which included all of the process steps and the data are being evaluated.

The test loop pump is still running with the Purex-designed pump. The resin attrition is rather high, i.e., approximately 50 ml of fractured resin beads per hour. The elutriation procedure is being improved to enable a more precise measurement of the attrition rate. An alternative pumping arrangement will be installed and tested in an attempt to reduce the attrition rate of the resin.

Further studies were made of catalytic effects on the nitrite-nitric acid oxidation of neptunium(V).

All but three of the Np-Al target elements from N Reactor have been processed.

Simulated organic wastes containing Cs^{137} were burned in air. From 93 to 98 percent of the activity remained in the burner cup residue. The rest of the activity was recovered in condensate. Organic wastes, such as, Di2EPHA, TBP, NPH, and CCl_4 did not leach any Cs^{137} or Sr^{85} adsorbed on soil in glass columns.

Temperature and velocity measurements were made with specially designed probes in the vapor overhead line from the A Tank Farm, preparatory to performing particle sizing and loading experiments.

ASSISTANCE TO ISOCEM

Mission 1 - Basic Production Mission

Studies on Normal Paraffin Hydrocarbons

(M. L. Ravenscroft, Heavy Element Chemistry Unit)

Work on the use of IR spectra to determine the olefin content of irradiated and nonirradiated NPH was resumed. Samples of NPH were irradiated to approximately $10^8 R$ and their spectra compared with those of nonirradiated material. The data indicated the production of trans-internal and terminal olefins in roughly $0.075M$ and $0.01M$ concentrations, respectively, which correspond to a total G value of 0.95. This figure is a little lower than values reported in the literature.

Vacuum System for Actinide Element Chemistry Studies

(J. D. Christian, Heavy Element Chemistry Unit)

Design features were worked out for a metal high vacuum system (capability: 5×10^{-8} Torr or better) to be installed in a glove box in 325 Building. The pumps will be located outside the glove box and will connect through the side to a manifold inside. The manifold will include two 6-in. ports with O-rings for connection to interchangeable pieces of apparatus. Also will be included a smaller manifold for operations such as synthesis, sublimation, transferring, evacuation of cells, etc.

Vaporization Studies

(J. D. Christian, Heavy Element Chemistry Unit)

Preparations continued for projected studies of the high-temperature thermochemical properties of certain actinide element compounds. Efforts are directed toward the preparation of pure $ZrCl_4$ and UCl_3 , required for study of the equilibrium

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UCl_3 of high purity was obtained by hydrochlorination of UH_3 which was prepared from the metal. Heating in vacuum at 500°C removed volatile impurities, such as UCl_4 , and UCl_3 was separated from nonvolatile impurities by vacuum sublimation at 500° in a stream of I_2 . This formed the volatile species UCl_3I which, when condensed at 400° , dissociated to yield pure UCl_3 .

ZrCl_4 was prepared by flowing CCl_4 over reactor grade ZrO_2 (low in Hf content) at 500° . However, a colored impurity, presumably iron(III) chloride, has proved difficult to remove completely. The material was fractionally sublimed at various temperatures; it was sublimed in a hydrogen atmosphere; and it was sublimed through iron wool in attempts to reduce the iron compound to nonvolatile FeCl_2 . None of the procedures has proved satisfactory. The sample will be dissolved and treated with mandelic acid, which selectively precipitates zirconium mandelate. This has been shown to remove Ti, Al, Fe, V, Cr, Sn, Bi, Sb, Ce, Ca, Th, Cu, Cd, and Ba impurities from Zr. The procedure should then provide pure zirconium mandelate which can be subsequently fired and rechlorinated.

DPPB Extraction of Neptunium and Plutonium from Purex 1WW (W. W. Schulz, Materials and Process Chemistry Unit)

A batch extraction process employing 25% DBBP in NPH as the extractant has been proposed for recovery of neptunium and plutonium from Purex process 1WW solution. Cyclic irradiation tests to establish the useful life of the proposed 25% DBBP-NPH extractant are in progress. Conditions of such tests and properties of cyclically irradiated (54 cycles at 2.3×10^6 Rads/cycle) were reported last month. Additional tests have demonstrated that extractant irradiated to about 1.2×10^8 Rads extracts only slightly more Zr^{95} than unirradiated extractant (89 vs. 83%). Further experiments demonstrate that 50 to 95% of the extracted zirconium activity can be removed by scrubbing the organic extract with small (one-tenth or one-fifth) volumes of 0.05 to 0.25M oxalic acid. Losses of neptunium and plutonium to such aqueous scrub solutions have not been determined.

Zirflex Solvent Extraction Studies

(G. L. Richardson, Engineering Development Unit)

Scouting studies were started to determine if uranium feed derived from a Zirflex decladding procedure will have any deleterious effect on solvent extraction processing. Preliminary results indicated that the performance of the columns was normal in every respect with feed containing up to $0.07M$ Zr and the associated fluoride (with aluminum complexant added to prevent fluoride corrosion). Three 3-in. diameter pulse columns were used in the tests, representing the HA, HS, and LC columns. The nominal volume velocities, respectively, were 1200, 1000, and 1600 gal/hr-ft², sum of flows; the operating temperatures were $55 \pm 5^{\circ}C$, and the pulse amplitudes were about 1 inch. Stable operating frequencies of 110, 70, and 60 cycles/min were demonstrated in the HA, HS, and LC columns, respectively, with the organic phase continuous. The LC column dispersion performance was improved somewhat by operating with the aqueous phase continuous.

Separations - Instrumentation

(C. N. Jackson, Jr., Instrumentation Section)

Continued operational experience with the gamma counting instrument developed to detect Am^{241} in the waste line of the W-14 column indicated that for concentrations above about 2×10^{-6} g/l, the instrument will indicate concentration of Am^{241} correctly. Below 10^{-6} g/l, background radiations, which may not be constant with time, cause incorrect response. However, with the two samples tested, the sample background appeared to be nearly the same for Am^{241} concentrations of 10^{-6} and 10^{-4} g/l. Thus, the background at the installed detector in the process line may be a function of other factors. To aid in retaining analyzer calibration, especially at the low concentration levels where the 60 keV peak is not well defined, a small sample of Pu^{239} could be used as a source of 17 keV photons to provide a signal for use by the automatic gain stabilizer.

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A product sample of column W-14 was analyzed for content and prominent peaks were observed at 17, 30, and 60 keV. There were also some energies appearing in the range of 100 to 130 keV, but these were less well defined and were about two orders of magnitude below the 60 keV peak value. This was a highly active sample, thus less efficient counting geometry with greater sample-to-detector separation was necessary to achieve reasonable counting rates for the analyzer. The higher energy peaks of 100 to 130 keV are possibly from the americium.

A spectrum analysis system using either a Ge(Li) or Si(Li) high resolution detector is being developed to provide a more powerful tool for measuring these gamma spectra.

Alpha counting and gamma spectrometer systems are planned for an in-line monitoring investigation of the Pu²³⁹ concentration in the CCW line. Evaluation of both systems is temporarily delayed pending installation of specific in-line sample cells.

Electrodeless Conductivity

(O. H. Koski, Process Systems Development Unit)

Testing of the commercial ceramic-lined electrodeless conductivity system continued with particular attention given to temperature compensation as a function of concentration. Stability of the system output is within 1% per week.

The conductivity-to-pulse height system which is being developed for alternate use was improved with respect to sensitivity by redesign of the pulse height-to-DC converter. Conductivity tests in solutions ranging from tap water to concentrated acid indicate that the use of a conductive core plug or the use of large ID toroids is required to give the sensitivity required for practical use. This system appears to be applicable to solutions presenting a solution loop resistance of about 50 ohms or less. With a typical probe, conductivities down to that of tap water can be determined if the conductive plug is used.

Ferrous Ion Analysis

(O. H. Koski, Process Systems Development Unit)

Ferrous ion analysis in the extractant stream using oxidation-reduction potential (ORP) electrodes was investigated further. A change to commercial ORP electrodes resulted in a loss of sensitivity to ferric ion concentration. Tests were made utilizing two ORP electrodes with one immersed in standard 1BX and coupled to the other immersed in the test solution by a porous graphite plug. Sensitivity to make-up errors such as the lack of ferrous ion with and without sulfamic and/or nitric acid was noted. Only a slight change was noted during destruction of the ferrous and sulfamate with sodium nitrite.

Fundamental Mass Transfer Studies

(P. C. Owzarski, Chemical Engineering Analysis Unit)

The dual pumping was received and a variable speed motor was fitted. Final assembly of apparatus for continual drop formation and observation is in progress.

Mission 3 - Transplutonium

Precipitation Separation of Americium and Curium

(R. E. Lerch, Fission Product Chemistry Unit)

As a back-up to ion exchange separation of Am and Cm from each other, preparations were made to test precipitation separation methods involving oxidizing the americium and precipitating it as the pentavalent carbonate. Ozone will be investigated as oxidant. Necessary equipment, including ozonators and reaction vessels, is being fabricated for both laboratory and hot cell use.

Mission 5 - Other Isotopes

Extraction of Polonium from Molten Bismuth with Fused Sodium Hydroxide

(W. W. Schulz, Materials and Process Chemistry Unit)

Fifteen experiments to check the feasibility of the Barney process at higher and more realistic Po^{210} concentrations

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were completed. In these experiments, 2 to 8 grams of irradiated bismuth containing about 6 mCi Po^{210} per gram were contacted one-half to two hours in an argon atmosphere at 450 to 600°C with molten NaOH at bismuth-to-NaOH weight ratios of 1 to 0.25. The molten phases were contained in a boat (either graphite or 400 series stainless steel) in a silica combustion tube in a horizontal tube furnace; agitation was provided by moving the furnace assembly through a two-inch stroke in a horizontal plane at about 47 cycles/minute.

Polonium was readily removed from the molten bismuth metal in all cases; in some experiments the bismuth retained less than one percent of its original Po^{210} content. The ease and degree of removal of Po^{210} increased with increased temperature and contact time and with decreased thickness of the molten bismuth phase (lower weight of bismuth) indicating, perhaps, a volatilization mechanism. In two favorable cases essentially all the Po^{210} removed from the bismuth phase reported to the NaOH phase. Generally, however, 10 to 20% of the Po^{210} removed from the bismuth was not found in the NaOH phase. It is presently believed that such losses occurred because of the particular experimental setup used and represent polonium deposited on or in boats and on the silica tube. A new, closed stainless steel boat was being fabricated for studies aimed at improving material balances.

Mission 6 - Enriched Fuel Processing

C-Cell Modification

(V. L. Hammond, Process Systems Development Unit)

C-Cell in 324 Bldg. is being readied for installation of a one-slug dissolver, plus experimental apparatus for characterizing solidified radioactive wastes. The design of the transfer mechanism between C-Cell and the air lock was reviewed and comments are being incorporated into the final design. Due to the difficult working conditions in the air lock (three suits of SWP clothing and fresh air masks are required) the new design has provision to reduce the amount of installation and in-place fabrication work required. The strength of C-cell door hinges was evaluated to determine if a shelf could be placed on the door

to transport 2000 pounds between the air lock and C-Cell. Strength calculations show that the hinge will not be overstressed and the hinge deflection will be less than 0.001 in. where the hinge is attached to the door.

Mission 8 - Nuclear Safety

Minimum Critical U^{235} Enrichment for Uranium Nitrate Solutions (S. R. Bierman, G. M. Hess, Critical Mass Physics Section)

Analysis of the data from the PCTR experiments for determining the limiting critical enrichment of slightly enriched uranyl nitrate solutions continued. The results are contingent upon the chemical analysis of the uranium solutions used which were of two different enrichments, 2.14 wt% and 2.26 wt% U^{235} . The highest value of k_{∞} is obtained at a hydrogen-to-total uranium atom ratio of about 8 with the 2.14 wt% solution and a H/U ratio of about 9.7 for the 2.26 wt% solution. Based on the relative reactivity of these H/U ratios and enrichments the limiting critical enrichment for homogeneous hydrogen-moderated mixtures of uranyl nitrate, $UO_2(NO_3)_2$, is 2.1 wt% U^{235} . At this enrichment, the highest value obtainable for the reproduction factor k_{∞} , irrespective of moderation, is unity. The value now computed by means of the GAMTEC II code is 2.09 wt% which agrees well with experiment. An error analysis is being made of the measurement.

Atmospheric Dispersion of Radioactive Particles

(J. Mishima, Particulate and Gaseous Waste Research Unit)

Study continued on the evolution of airborne particles from overheated plutonium compounds in an airstream. Particles from plutonium oxalate-oxide mixture and plutonium fluoride at ambient temperature, 700°C and 1000°C, were evaluated and particles within the visual range of the optical microscope used were measured. The mass median diameter of fluoride particles airborne at 700°C was about 48 μ and the particles airborne from oxide-oxalate at 1000°C, about 18 μ . The particles are quite large and doubtless represent sizes present in the parent material, rather than reflecting a temperature-activated breaking apart of the material.

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The fraction of the heated compound made airborne per minute in a vertical flow of 100 cm/sec was measured and compared with the fractional release under ambient temperatures, but the same flow. In most cases, the heating enhanced the release rates; however, for plutonium fluoride at 1000°C, the release rate was lower than for ambient temperature, presumably due to some caking or fusing. Release rates as high as 0.5% per hour were reached.

Mission 9 - Waste Management

Salt Waste Processing, Properties of Di-(2-ethyl hexyl) Phosphoric Acid

(J. A. Partridge, Heavy Element Chemistry Unit)

A sample of di-(2-ethyl hexyl) phosphoric acid (D2EHPA) was purified (Cu(II) salt procedure), washed with distilled water, dried in a Buchi Rotavapor at 15 mm pressure and 50-55°C, and used for measurement of a number of physical and chemical properties.

An equivalent weight determination was made by titrating the purified D2EHPA in an ethanol-water solvent with NaOH solution using a Metrohm Potentiograph E336 automatic titrator. The pH curve showed a sharp strong-acid type end-point; three titrations gave an experimental equivalent weight of 322.7 ± 0.5 (theor. 322.4). Only one end point was observed with the purified D2EHPA in contrast to the starting material which gave two weak-acid-type end points due presumably to the presence of some mono-(2-ethyl hexyl) phosphoric acid (M2EHPA).

The density, viscosity and refractive index of the purified D2EHPA were determined and are listed below. The density and viscosity were also determined for D2EHPA saturated with water. A significant increase in the viscosity was observed upon saturation with water as is seen from the values in the table.

	Density (g/ml)		Index of Refraction 25°C	Viscosity (Centipoises) 20°C
	20°C	25°C		
Purified D2EHPA	0.9727	0.9692	1.4418	39.8 ± 0.1
Purified D2EHPA saturated with H ₂ O	0.9739	0.9715		59.2 ± 0.1
Purified D2EHPA by Peppard	0.973		1.4469	42.2

The viscosity determinations were made at 20°C with a modified Ostwald viscometer calibrated with two glycerine-water solutions.

The molar refraction, $(R) = \{(n^2-1)/(n^2+2)\} \cdot (\text{mol. wt.}/\rho)$, of D2EHPA as determined from the experimental data is 87.99. For comparison, a value of 87.989 for the molar refraction was calculated from the atomic refractions.

Some physical measurements (summarized below) were also made on the plant D2EHPA, as well as on D2EHPA obtained commercially. Measurements were made on the as received materials and on samples which had been in the Rotavapor at 15 mm pressure and 50°C for 2-3 hours to remove any dissolved water. The D2EHPA from the above sources had an odor, and it is possible that this treatment in the Rotavapor may have partially removed some volatile organic impurities in addition to water that may have been present. Thus, the viscosity differences between the as received and the dried products may be due to removal of some of the organic impurity rather than to removal of water.

The values for the percentage composition were calculated from NaOH titration data. It was assumed that the only acids present in the samples were D2EHPA and M2EHPA.

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HDEHP	Density 20°C	Index of Refraction 25°C	Viscosity (Centipoises) 20°C	Wt. % D2EHPA	Wt. % M2EHPA	Wt. % Non-Acid
Victor Chem, As Received	0.9740	1.4419	49.7 ± 0.1			
Victor Chem, Dried	0.9740	1.4419	47.5 ± 0.1	94.5	2.5	3.0
Eastman, As Received	0.9747	1.4417	47.4 ± 0.1			
Eastman, Dried		1.4419	46.4 ± 0.1	96.4	2.1	1.4
Plant, As Received	0.9761	1.4425	51.3 ± 0.1			
Plant, Dried		1.4426	47.9 ± 0.1	94.7	1.4	3.9

Purex Solids

(F. P. Roberts, Fission Product Chemistry Unit)

Work continued on various aspects and perturbations of the problems arising from the formation of fission product-bearing solids in the concentrated Purex acid waste. Previous work demonstrated an efficient metathesis and leaching process for recovering the Ce and Sr which precipitate on the sulfate solids (which are formed when the dilute Purex acid waste is concentrated for transfer to B-Plant). It would be more desirable to prevent this precipitation in the first place. The effect of adding excess iron (to complex the sulfate) was investigated in the laboratory and demonstrated in the hot cell. Over 90% of the cerium was retained in solution when the iron/sulfate ratio exceeded unity whereas 70% precipitated when iron was not added. Strontium analyses are not yet available. Although this solution to the problem appears to be technically feasible, addition of gross quantities of iron has obvious drawbacks.

A hot-cell experiment was described last month in which an attempt was made to drive the Ce and Sr quantitatively to the solids--by evaporation to a volume of only 18 gal/ton and to an acidity of only 0.37M H⁺. The analytical data show that the effort was only partially successful; 93% of the Ce precipitated but only 85% of the Sr. Waste Management goals require removal of about 95% of each. Increasing the sulfate may help and will be tried.

In a third series of experiments, exploring further the implications of the metathesis-leach process, fate of the leachate on recombination with the waste at B-Plant was determined. Reprecipitation occurred. However, it was found possible to prevent this reprecipitation by prior addition of complexant. D2EHPA extraction behavior of the fission products under B-Plant processing conditions was also studied. With addition of complexant prior to reconstitution of the waste, cerium extraction and material balance were excellent. Strontium, on the other hand, did not extract ($E_a^0 = 0.01$ to 0.03). The reason for this behavior is unknown and will require further study.

Semiworks Assistance

(L. A. Bray, Fission Product Chemistry Unit)

Close laboratory and hot cell support was provided to a series of Strontium Semiworks campaigns and plant tests. Included were hot cell experiments to typify the Am-Cm concentrate (from the Redox Shippingport campaign), which is awaiting processing at Semiworks, and precipitation problems with several cerium feeds (which are being processed for 324 Building WSEP use). The Am-Cm concentrate was found to contain approximately 30 vol% solids--apparently similar to the zirconium-bearing interface "crud" which complicated the Redox recovery program. A sample of the solids was leached to determine their americium and curium content.

Semiworks cerium processing was plagued with rare earth precipitation problems, caused by organic residues in the feed. Extraction conditions were defined which permitted processing one batch of this material; discard of another batch was strongly recommended.

Promethium - Cerium - Americium Separation

(L. A. Bray, Fission Product Chemistry Unit)

Work continued on the solvent extraction separation of actinides and lanthanides in support of Semiworks processing of the Am-Cm concentrate. The laboratory experiments indicated that a critical "synergistic ratio" of the two or more complexants

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used must be maintained for good separation. Within the lanthanide series, single-stage separation factors as high as 18 were obtained for the Ce(III)-Pm(III) separation. The possibility of separating Pm from Nd and Sm with this system is also being explored. If this can be achieved, the cost of promethium purification could be substantially reduced.

Cerium-Rare Earth Separation - Electrolytic Oxidation Process (R. E. Lerch, Fission Product Chemistry)

Electrolytic oxidation, followed by D2EHPA solvent extraction, is being investigated as an alternate process for the separation of cerium from the other rare earths. (Ozone oxidation was investigated earlier and abandoned because of slow kinetics; silver-catalyzed persulfate oxidation is currently used but is expensive; the promising synergistic solvent extraction process is reported on above.) Equipment was assembled and preliminary experiments performed.

B-Plant Solvent Quality (W. W. Schulz, Materials and Process Chemistry Unit)

Results reported earlier have shown that the strontium extraction capacity of a 0.3M D2EHPA-0.2M TBP-NPH extractant falls by a factor of 1.5 to 2.0 upon irradiation to about 10^8 Rad. This decrease in extraction capacity occurs even though the D2EHPA concentration decreases only slightly; furthermore, the strontium extraction capacity of an irradiated extractant cannot be restored fully simply by addition of virgin D2EHPA. Mono (2-ethylhexyl) phosphoric acid (M2EHPA) is one of the primary radiolysis products of D2EHPA. It is presently believed that D2EHPA and M2EHPA in irradiated extractants form association compounds which do not extract strontium as well as, or in the same manner as, D2EHPA itself.

Experiments completed this month and last demonstrated that the strontium extraction capacity of irradiated extractants can be restored by washing either with ethylene glycol or with dilute sodium carbonate solutions. In both cases the strontium extraction capacity of the washed irradiated extractant corresponds

reasonably well to that expected for D2EHPA concentration and also responds in the manner expected when virgin D2EHPA is added to alter the D2EHPA concentration.

Electrotitrations show that both ethylene glycol and Na_2CO_3 washes remove M2EHPA very effectively from irradiated solvents. For economic reasons, however, Na_2CO_3 washes are recommended for use in B-plant operations. In this connection what appears to be a satisfactory wash sequence for removing inert and fission product contaminants as well as M2EHPA and for adjusting the extractant sodium-to-hydrogen ratio was devised and tested with SSW solvent. Subsequently this wash treatment, which involves successive washes with NaOH-sodium citrate, Na_2CO_3 , and HNO_3 solutions, was used in the SSW to clean up the current solvent inventory.

Radiation Effects on pH Sensing

(O. H. Koski, Process Systems Development Unit)

The irradiation of an operating pH flow cell system continued throughout the month. The experiment is being carried out in the 3730 Bldg. Co^{60} pool facility at a radiation intensity of about 6×10^5 R/hr. The experiment was interrupted several times during the month due to irradiation failures of the signal cables, plus failures of the flow cell sealing system with subsequent flooding of the containment vessel. Consequently, the irradiation received during the month was limited to about 2.4×10^8 R, bringing the total irradiation dose received by the pH sensing electrodes to about 3.8×10^8 R. While the pH electrodes are beginning to show outward evidence of irradiation damage, they are still functioning normally. A prototype pH flow cell is currently being built for an in-plant demonstration.

Soil Chemistry

(B. F. Hajek, Water and Wastewater Research Unit)

Studies on the development of criteria for improved ground disposal of 300 Area laboratory wastes continued. Laboratory experiments designed to evaluate the use of theoretical and empirical equations for strontium K_d predictions were conducted.

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The data showed that a theoretical ion exchange model did not adequately predict strontium K_d 's in multi-ionic systems. Solution of a regression equation developed from statistical analysis of the data resulted in adequate K_d predictions within narrow concentration limits of the competing cations: Na, K, Ca, and Mg.

The following equation from which strontium K_d 's can be calculated for a waste solution, similar in ionic composition to tap water, was developed:

$$K_d = \frac{1 \times 10^3}{22.2 + 0.217 \text{ ppm K} + 0.0116 \text{ ppm Na} + 6.4 \text{ ppm Ca} + 0.57 \text{ ppm Mg}}$$

The equation proved satisfactory for competing ion concentrations between 0 and 500 ppm Na and up to 100 ppm of the other cations.

Alkaline Supernatant

(B. W. Mercer, Water and Wastewater Research Unit)

Eight load and elute cycles were completed with simulated Redox alkaline supernatant waste and a recent lot of AW500 (Lot No. 565001). According to the vendor this lot of AW500 had an improved binder which was believed to be more resistant to caustic attack. Laboratory results showed deterioration of this lot of AW500 after 7 cycles which is no improvement over previous lots.

Wet attrition tests were performed with dehydrated AW500 to determine the effect of water temperature on fines generation. A small increase from 6 to 7% fines was evident by increasing the temperature of the water from 24 to 84°C. This increase does not indicate significant damage to the AW500 by hydration in water at elevated temperatures.

Heat Transfer Associated with Underground Waste Storage Tanks

(G. Jansen, W. E. Willingham, Chemical Engineering Analysis Unit)

Construction of an in-tank circulation test stand was completed. The model consists of a 10 ft diameter tank with 6

independent circulators. Calibration of orifices on the tank exit lines and rotameters on the tank inlet lines was completed. Flow and measurement capabilities range from very low flows to 150 gpm. The flow to and from each circulator can be controlled independently, and although the flow is motivated by a pump, air can be introduced into each circulator to simulate an air lift. In-tank flow measuring devices to be used include thermistors and dye tracers. Initial experiments will include variation of length-to-diameter ratios in the circulation cell around one circulator and investigation of interference patterns between circulators. The initial work is directed toward early recommendation for design of the new Isochem AY tank farm.

Safety Considerations of Fission Product Storage on Zeolites
(W. E. Willingham, Chemical Engineering Analysis Unit)

The equipment for measuring flame velocities in zeolite beds was moved from 321A to the 324A Bldg. and testing will continue shortly.

A further effort will be made to stop the interaction between the two time interval counters. If this new approach does not work, then the runs will be made using the counters separately.

Process Technology

N-Reactor Decontamination Waste Studies
(J. F. Phillips, Materials and Process Chemistry Unit)

N-Reactor decontamination wastes are scheduled to be sent to B-plant facilities for concentration and disposal. These waste solutions, singly or in combination with separations processes wastes, will be concentrated in a steam-heated evaporator. They will then be sent to underground waste storage tanks for subsequent further concentration to solids by immersion heaters. A survey was made to determine if this sequence of treatments presents any hazards from the standpoint of highly exothermic reactions since some of the N-Reactor wastes contain strong oxidants and some contain organic materials. Portions

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of the proposed decontamination wastes were combined with appropriate simulated separations processes wastes and evaporated to dryness. Differential thermal analyses were made on the solids over a temperature range up to 500°C. Combinations tested involved seven proposed N-Reactor decontamination solutions with Purex stored waste supernate, high-aluminum in-tank solidification waste and low-aluminum in-tank solidification waste. Significant exothermic reactions were noted in a few cases, particularly with N-Reactor wastes alone (not combined with separations processes wastes). Dilution of the N-Reactor wastes with separations processes wastes suppressed the exothermic reactions.

Zirflex Dissolution Studies

(W. J. Van Slyke, Engineering Development Unit)

A third run investigating all the process steps (decladding, dissolution, and metathesis) and using four 3-day autoclaved NPR fuel elements has been completed. The run went smoothly and all of the fuel material was dissolved with the exception of a few scraps of Zircaloy-2 cladding. The next run will use 5.5M NH_4F and 90-day autoclaved NPR fuel elements; a delay of several days has been necessary to enable the replacement of a failed pH cell in the condensate return line. The analytical results have been received for the first of the three runs which included all of the process steps and the data are being evaluated.

Purex Ion Exchange Resin Pump Studies

(W. J. Van Slyke, Engineering Development Unit)

The test loop pump is still running with the Purex-designed pump. The resin attrition is rather high, i.e., approximately 50 ml of fractured resin beads per hour. The elutriation procedure is being improved to enable a more precise measurement of the attrition rate. An alternative pumping arrangement will be installed and tested in an attempt to reduce the attrition rate of the resin.

Oxidation of Neptunium(V)

(J. L. Swanson, Chemical Separations Unit)

Investigation of the oxidation of neptunium(V) in solutions prepared from the aci- form of 1-nitropropane continued. It was found that the rate of oxidation in 3.1M HNO₃ solutions prepared by adding HNO₃ to basic solutions containing 1-nitropropane and sodium nitrite varies with the age of the solution eventually reaching the point where no effect of the nitro compound is obtained. Substantial rate improvement is still obtained after 20 hours, but best results are achieved with shorter aging times.

Np-Pu²³⁸ Processing

(T. S. Soine, Chemical Separations Unit)

All but three of the Np-Al target elements from N-Reactor have been processed. The last three elements have been dissolved, the solution being held for solvent extraction studies in support of Isochem's R&D program. These studies will begin as soon as equipment can be installed in C-Cell. A more detailed report on material balances and recoveries for the Np-Pu²³⁸ processing will be prepared as soon as all the data are available.

Organic Waste Oxidation

(K. C. Knoll, Water and Wastewater Research Unit)

The fate of radionuclides when a hexone waste containing them is burned was investigated. A two-foot long glass pipe was used as a chimney with the fuel burned in a stainless steel cup at the bottom. A suction pump at the end of the train pulled air and combustion products through an asbestos filter, a condenser and a trap, and then through a final trap. The burning cup and chimney were washed out with 8N HNO₃ as were the condenser and trap. Aliquots of all washings and scrubber solutions and the entire filter were measured for gamma activity. Two solutions were burned, one hexone alone and the other a hexone-TBP-NPH mixture. Both were spiked with Cs¹³⁷.

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Sample	Hexone		Hexone-TBP-NPH	
	Total c/m	% of Recovered Activity	Total c/m	% of Recovered Activity
Burner Cup	15,272	98	16,740	93
Chimney	200	1.3	Bkg	
Condenser and Trap	Bkg		980	5.5
Filter	140	0.9	380	2.1
HNO ₃ Scrubber	Bkg		Bkg	
H ₂ O Scrubber	Bkg		Bkg	
c/m in Burned Soln.	15,380		20,076	
% Activity Recovered	101		90	

As can be seen, most of the activity remains in the burner vessel, the rest appears as a condensate.

Organic Waste Disposal

(K. C. Knoll, Water and Wastewater Research Unit)

The effect of organic solutions on radionuclides present on soil has been studied for three of the possible organic wastes from the 234-5 Plant. One hundred fifty column volumes of a mixture of 0.4M Di2EHPA-0.2M TBP in NPH was put through a soil column and showed no uptake of Cs¹³⁷. A TBP-CCl₄ (20-80%) mixture has had 50 column volumes through with no uptake of Cs¹³⁷ and at 150 column volumes none of Sr⁸⁵. As reported last month DBBP-CCl₄ (30-70%) also extracted no Cs¹³⁷ or Sr⁸⁵ from loaded soil.

The determination of the permeability of a soil for the nominal aqueous waste was completed. The final result was lower than anticipated from earlier observations. At the end of 16 days it showed a permeability of 9.2×10^{-7} cm³/cm²/sec at hydraulic gradient of 100%.

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Characterization of Gaseous Effluents

(A. K. Postma, L. C. Schwendiman, Particulate and Gaseous Waste Research Section)

A study is under way to determine the nature and amount of aerosol transported in the 24 in. vapor vent line from the A Tank Farm (Purex). Progress included fabrication and installation of the access port and valve through which temperatures and flow rates will be measured and the radioactive aerosol sampled. Gas temperatures were measured over a period of several days. This temperature measurement was required to permit isothermal withdrawal of particulate samples, thus avoiding condensation of the near-saturated gas in the delivery lines and on the collector. A velocity-sensing pitot tube was installed and initial measurements were completed. A particle sampling probe, a fibrous filter analyzer, and an adapter for a cascade impactor were fabricated for use in characterizing the airborne constituents in this stream.

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TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Summary

Environmental Evaluations

The presence of significant concentrations of Sb^{122} in river water, if confirmed in Tri-City drinking water, could result in increases in estimated GI tract doses for local residents. Increased radioiodine releases from either Battelle-Northwest or Isochem facilities resulted in a slight increase in the I^{131} concentration in milk from local farms.

A study of oil seeping from beneath 100-N to the river is directed toward estimates of the rate of seepage.

A pilot study of environmental air quality with regard to stable pollutants was initiated.

Dosimetry Studies

Studies continued on the performance and possible uses of thermoluminescent and glass materials in dosimetry work. Development work on a portable neutron survey instrument continued as did development of equipment methods and processes in support of biological and internal dosimetry programs.

Instrumentation Services

Minor time-delay circuitry changes improved the operational performance of the laundry monitoring system and preparation of the operation and maintenance manual moved forward.

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TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Environmental Evaluation

(J. F. Honstead, Environmental Studies Section)

The U. S. Testing Laboratory has reported the presence of Sb^{122} in river water samples, in at least one sample at a concentration about equal to As^{76} . This would indicate a significant change since 1964 when the abundance of Sb^{122} was last measured. If this nuclide is also present in drinking water in concentrations comparable to that of As^{76} , increases in estimated doses to the GI tract for Tri-City residents are indicated; however, the dose will still remain well below appropriate limits.

Processing of fuel elements with short cooling periods again resulted in releases of radioiodines from Radiometallurgy and Radiochemistry Buildings in the 300 Area. A total release for the month of about 0.5 curie of I^{131} was measured with charcoal filters, with a maximum weekly release of 160 millicuries from the 325 Radiochemistry Building. From previous studies, the equivalent measurements with caustic scrubbers, upon which current release standards were set, would have been about half those measured with the charcoal.

No traces of these releases were found in environmental samples, with the possible exception of a slight increase to a maximum of 7 pCi/l I^{131} in milk from farms north of Pasco. However, an increase in radioiodine release from Isochem facilities during the same period could also have accounted for the observed milk concentration. The programs responsible for these releases are continuing programs; continued releases at current rates would be expected to result in significant increases in estimates of thyroid doses.

Oil Contamination in Columbia River at 100-N

(J. F. Honstead, Environmental Studies Section)

Efforts are under way to determine the amount of diesel fuel entering the Columbia River at 100-N along a 100-yard front, as the result of a line leak in the fuel oil storage

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area. It is estimated that approximately 80,000 gallons of oil were released to the ground during a 14-month period before the leak was detected and repaired.

Air Quality Study

(J. F. Honstead, Environmental Studies Section)

A study of air quality in the Hanford environs, near selected operational sites, with reference to nonradioactive pollutants, was initiated using special continuous analyzers designed for this purpose. The specific measurements being performed at these sites include the determination of concentrations of sulfur dioxide, nitric oxide, nitrogen dioxide, total aldehydes and hydrogen sulfide. During a five-day period in the 300 Area, oxides of nitrogen were detected on three days and a trace quantity of sulfur dioxide was detected on another day. The measurements currently under way are regarded as a pilot study to help design an environmental monitoring program.

Dosimetry Studies

(C. E. Newton, Jr., Biophysical Sciences Section)

Portable Neutron Survey Instrument

Development of a portable neutron survey instrument using a tissue equivalent proportional counter continued with fabrication of a sealed unit. This sealed unit provides for a permanent gas filling and also shields the outer surface of the counter which is operated at a high negative potential. Experiments to determine effects of the plastic on the gas are continuing using a special copper counter and an alpha radiation source. These experiments are being done to measure counter resolution for various angles of entry of the radiation so that optimum counting geometry can be determined as a function of electrode shape.

Recent theoretical treatment of proton energy loss relationships has indicated that proper operation of the tissue-equivalent proportional counter can be assured through the

observation of the energy loss distribution of high-energy protons. We are presently attempting to arrange a high-energy proton irradiation of our proportional counter at the 184 in. cyclotron of the Lawrence Radiation Laboratory. With the proton data acquired during this run, we can assure that the counters will respond properly when utilized as a mixed neutron-gamma field instrument.

Thermoluminescent Dosimetry

The thermoluminescent dosimetry program continued with an experiment to improve the response of a two-unit TLD-disc system to uranium beta. Sets of discs were exposed to uranium beta radiation behind 0.0035 in. and 0.032 in. of aluminum. Exposures were made from 25 m Rads to 2 Rads. Exposures were also made to radium γ radiation using the same filters. The results, incomplete at this time, will be reported next month. A set of exposures to radium γ using the TLD discs behind 0.010 in. and 0.040 in. of Lexan plastic showed essentially no difference in reading in the range from 25 mR to 2 R. Thus, this difference in thicknesses of Lexan made no difference in the TLD reading from penetrating radiation.

A series of experiments designed to determine the response of the TLD system after prolonged exposure to temperature extremes was started. Sets of TLD discs were exposed to 1 R radium γ and then divided into three groups: One group is kept at room temperature, the second group is being held at -44°C , and the third group is being held at $+150^{\circ}\text{F}$. The first results show essentially no change in sensitivity after one day at -44°C ; no change is expected at low temperature. After one day at $+170^{\circ}\text{F}$, the reading on the discs was 20% low. The experiment is scheduled to continue for a period of two weeks.

Internal Dosimetry

Theoretical excretion of plutonium in urine based on the new ICRP lung model was developed for inhalations of plutonium oxide and plutonium nitrate of various particle size distributions. A study in dogs of the metabolism and excretion of

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plutonium resulting from inhalation of plutonium fluoride dust and as modified by DTPA treatment for enhancement of excretion was established in the Biology Department.

Radio-luminescent Glass Study

The program of cross-checking the river monitoring pencil dosimeter using glass block and thermoluminescent dosimeters was extended for an indefinite period. The results of the second monthly dosimeter test indicated that the glass and thermoluminescent dosimeter responses were essentially the same for the period measured. The pencil data appeared scattered with some of the results being higher and some lower than the glass block and thermoluminescent dosimeters.

Personnel Dosimetry

(H. V. Larson, Personnel Dosimetry Section)

There were 573 bioassay samples submitted for plutonium analysis during the month, including 23 from new employees, who had either previously worked with plutonium or who were assigned to work with plutonium in their assignment at Hanford. There were no new plutonium deposition cases confirmed from evaluation of bioassay data. Also, 165 samples were submitted for uranium analysis, 230 for tritium analysis, 28 for strontium analysis, 121 for promethium analysis and 76 for americium analysis.

Special occupational exposure summaries were prepared in response to requests from new employers of 11 former Hanford employees, under provisions of the AEC regulations 10 CFR 20 and AEC Manual Chapter 0502.

Special external exposure evaluations were performed for 9 Hanford contractor employees; however, no exposure over permissible limits was revealed.

Records processing of 2888 employees' beta-gamma film badge dosimeter results, 244 neutron results and 269 finger ring results were conducted. None of the results exceeded limits established in the AEC Manual Chapter 0502.

Stationary and mobile whole body counter use factors for the month were 45 and 68 percent, respectively. Twenty incident cases were counted including twelve for possible deposition of plutonium or americium. Evaluation of the results of these examinations indicates internal deposition of gamma-emitting radionuclides in excess of 1% of the applicable permissible limits in 5 cases. All five cases involved americium-241 with deposition ranging from 2 to 14% of the maximum permissible lung burdens.

Potassium-40 measurements were made on sixty-three Richland high school students who were examined in the whole body counter. These data were used in a thesis study of stress versus obesity being conducted by a Battelle employee on educational leave of absence.

Tours of the stationary whole body counter were conducted for seven groups totaling 152 persons including symposium attendees, Prosser and Granger High School students, Oregon State University and Oregon Technical Institute students and professors, and an adult nuclear information class from the University of Washington.

Examination of the pulse height spectrum from the Maushart lung counter indicates that there is a problem with counter response as a function of source location. Other experimenters also have found double peaks when exposed to mono-energetic sources indicating two separate, distinct counter pulse-height distributions associated with each X- or gamma-ray energy. This "Compton-like" effect could give rise to interference in the plutonium counting channels from non-plutonium radioisotopes in the body.

Alpha energy analysis and plutonium particle size distribution measurements were made on nasal smears taken in connection with three unusual incident exposures in the Isochem Plutonium Purification and Fabrication Facility. The three nasal smears showed particle size activity median diameters of 4.4, 5, and 10 microns, respectively. The $\text{Pu}^{239}/\text{Am}^{241}$ ratio was 13.5 and 13.9 for the two nasal samples

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which contained sufficient plutonium activity for accurate measurement. This information was useful in evaluating the deposition of plutonium in the lung of the employees involved.

Aquatic Biology Services
(H. A. Kornberg, Biology Department)

Surveys of nesting Canadian geese and gulls were completed. About 600 goslings were produced by the Hanford goose population this season, or about two-thirds normal production. This decrease was due to heavy coyote predation on Locke Island, where all nesting attempts were defeated. This island usually yields about 100 nests, of which 75 are successful and produce 300-350 goslings. Clutch size increased over last year (6.2 eggs per nest compared to 5.8 last year) and was significantly greater than the 5.6 average for 14 years of record.

The gull colonies at Ringold and Coyote Rapids contain about 4,000 nests this year, or about 8% fewer than last year. Average clutch sizes were 2.6 at Coyote Rapids and 2.8 at Ringold, which is the same relationship of clutch size to location as noted before. Mapping of the colonies was begun by using telescopes in the 30-foot towers erected prior to the nesting season. Several banded birds were observed by telescope. This technique substantially increases our band returns and provides the opportunity for double recovery of banded birds.

From just-completed studies with fish previously immunized to C. columnaris, it is clear that fish exhibit the classic response to a so-called booster immunization. That is, in response to a booster shot, they exhibit a very rapid rise and somewhat higher titer than is developed by previously non-immunized fish.

Preliminary studies were initiated to determine whether river fishes residing in warm water areas (70 to 80°F) of the Columbia River within the Hanford Reservation may be serving as carriers of the columnaris disease during the

winter months. River temperatures during this period were 47 to 52°F. Agglutinating antibody production and presence of the pathogen were used as the criteria of infection. Average titer for 40 fish from the 100-D hot springs area was 1 to 104 as compared to average titers of 1 to 106 for 35 fish taken from the river proper. No columnaris organisms were isolated from any of the fish during the 30-day test period. These results suggest that animals residing in warm water areas of the project are not being exposed to the pathogen during the cold water months and that they are probably not carriers of the disease.

Some of the experimental lots of young chinook salmon reared under different temperature regimes for mortality and growth studies have been terminated. The results are not complete, but growth and mortality data suggest that temperature increments greater than 5°F above the base river temperature may result in significant detrimental effect for fall run chinooks. For example, three lots reared 3°F above the river temperature experienced a total mortality of about 12% compared with about 25% mortality for lots reared 5°F above the base river temperatures.

Instrumentation Services

Laundry Monitoring System

(E. M. Sheen, Instrumentation Section)

With the completion of minor modifications which improved the temperature stability of the time-delay circuitry, the experimental automatic laundry monitor continued to function successfully. Machine down-time during the repairs was eliminated by modifying one channel at a time as small single-width garments were being surveyed.

Completion of the first draft line drawings and preparation of tracings are facilitating preparation of the operating manual. The document will be comprehensive enough to allow the supporting personnel to properly maintain and operate the system.

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Completion of the first draft line drawings and preparation of tracings are facilitating preparation of the operating manual. The document will be comprehensive enough to allow the supporting personnel to properly maintain and operate the system.

Meteorological Services

(E. H. Phinney and D. E. Jenne, Atmospheric Sciences Section)

Meteorological services, viz., weather forecasts and observations and climatological services were provided to plant operations and management on a routine basis.

MAY 1967
Forecasting

<u>Type</u>	<u>No. Made</u>	<u>% Reliability</u>
Production	93	83.6
General	62	86.0
Special	124	95.2

Number of calls processed by Code-A-Phone: 5,956

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