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AEC
RESEARCH
and
DEVELOPMENT
REPORT

PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
FOR AUGUST 1966

DIVISION
OF
PRODUCTION AND HANFORD PLANT ASSISTANCE PROGRAMS

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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
AUGUST 1966

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of

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September, 1966

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PACIFIC NORTHWEST LABORATORY
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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

Adsorption tests with carbon at the Richland Water Plant made during reactor shutdown showed that nitrate ion is removed in significant quantities and that Mn^{54} and Zn^{65} are physically attached to the filter medium.

Development of the digital simulation model for heat flux in the river downstream from the Hanford Project continued. All input data are being converted to EDP format and improved data bases developed.

An experiment designed to provide calibrations of the whole body counter for Zn^{65} in school children failed to provide the necessary data because of very low uptake of the nuclide from the oysters used as a source.

The estimated catch of fish from the Columbia River from the Tri-City area for the period February 7 to April 15 exceeds 10,000. Most of the fish are spiny-rayed species and nearly half of the fishermen were students.

ADVANCED CONCEPTS

The economics chain of computer codes was used to obtain isotope production data for the PWR and HWOCR reactor types.

U²³⁵ ANALYZER

Solid state circuitry development progressed on the portable gamma spectrometer being developed for use by the AEC Division of International Affairs in measuring the U^{235} content of unirradiated fuel elements and for general portable gamma spectrometry.

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DIRECT AEC SPONSORED PROGRAMS

COLUMBIA RIVER STUDIES

Effect of Reactor Effluent on the Quality of Columbia River Water
(Environmental Studies Section)

Continued progress was made on the test for effectiveness of activated carbon for the removal of selected dissolved inorganic ions and total organic material at the Richland Water Plant. Water Plant flow distribution tests were completed and a trial background run was made on August 23 to determine the effectiveness of the plan and to obtain radionuclide distribution on the filter bed during the shutdown period of the reactors. The primary radionuclides measured were Mn^{54} and Zn^{65} which appeared to persist in significant quantities after five weeks of no release. Additional tests are planned for September 1 and September 8 at which time various carbon feeds will be used to determine any related effects. Significant quantities of nitrate ion appear to be removed from the raw water influent.

An investigation was conducted in Ice Harbor reservoir to determine the validity of the routine temperature data reported by the Fish and Wildlife Service. These data form an important part of the information used in a mathematical model that simulates the flux of heat in the McNary reservoir. It was determined that the generator cooling temperatures reported can be readily corrected to report actual stream temperature, but that improved temperature detectors should be installed.

Preparations were made to begin monthly reporting of river temperatures on an unclassified basis. In addition, all data in the file are being prepared for electronic data processing in order to permit greater flexibility in use.

EXPOSURE MECHANISMS

(Environmental Studies Section)

An experiment was begun last month to provide a Zn^{65} calibration function for the shadow-shield whole body counter. The function is to relate the instrument calibration to the height and weight of the individual counted. Such functions are particularly important for application to children's whole body counting data. The experiment involved six individuals of different sizes. They were given Zn^{65} by providing them with several meals of oysters from the Pacific Coast. Each individual ingested 45 to 55 nCi Zn^{65} with the oysters and the amount absorbed was

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determined by excreta analyses. The experiment did not fulfill expectations because the subjects absorbed an average of only 3.4% of the Zn ingested. In an earlier study involving a single individual, 14% of the Zn ingested with oysters was absorbed; and in the case of Columbia River fish it is about 35%.

Data collected for us by the State Game Department indicate that about 4000 persons fished the Columbia River in the area immediately below the project between February 7 and April 15. The estimate was calculated from the results of a statistical sampling program. About 85% of the fishermen were Tri-Cities residents and 57% of them were contacted on weekends rather than weekdays. The largest occupational category found was students, who were estimated to represent 44% of the fishermen. The data indicate that about 10,000 fish were caught during the period, of which 60% were spiny ray varieties and 20% were catfish.

Behavior of an Idealized Stream (Mathematical Analysis Section)

The first attempt at developing a computerized Monte Carlo type model for the lateral dispersion of a pollutant which has been injected into an idealized stream was successfully completed. The results suggest that extensive modifications will be necessary to account adequately for velocity variations. Analytical work seems to predict that both vertical and horizontal velocity profiles should be on a logarithmic scale.

U235 ANALYZER (Instrumentation Section)

Solid state circuitry development progressed on the portable gamma spectrometer being developed for use by the AEC Division of International Affairs in measuring the U²³⁵ content of unirradiated fuel elements and for general portable gamma spectrometry. A light weight, constant current battery charger, which uses no relays and operates without adjustment from a 100 to 230 Vac, 50 to 60 Hz power source, was developed for use with the instrument. Developed low voltage regulators operate from the two 12V, 2A-hr, nickel-cadmium batteries to provide ± 8 Vdc, regulated to within $\pm 0.002\%$ for the working voltage variation of the batteries.

Automatic gain stabilizer improvement was achieved by addition of a circuit developed to indicate loss of gain control by the stabilizer, where the loss of control is shown by a flashing red lamp operated by a dual trip circuit that tests an analog signal from the stabilizer for operation within linear limits. Mechanical design for the portable probe and stand was started and, in addition, engineering tracings were completed

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for the pulse amplifier, gain stabilizer, counting rate circuits, scaler-timer, high voltage supply, battery charger low voltage regulators, and the front panel with controls.

ADVANCED CONCEPTS

(Advanced Concepts and Analysis Section)

EPIMETHEUS

The economics chain of computer codes was used to obtain isotope production data for the PWR and HWO CR reactor types. Operating conditions included 0% and 100% recycle of plutonium alone, U^{236} alone, and plutonium and U^{236} together. The data obtained are to be input data for the EPIMETHEUS subroutine of VESTA. The EPIMETHEUS output--available concentrations, as functions of time, of 10 by-product isotopes--will be part of a report documenting the EPIMETHEUS subroutines.

Curium Model

Cost calculating routines are now being added to the burnup code (CUPID) and the separations code. Calculations as to the need for segregating plutonium and americium discharge batches are being made.

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ASSISTANCE TO DOUGLAS UNITED NUCLEAR

SUMMARY

MISSION 1

Preliminary planning was performed for a combined analytical and experimental investigation of the consequences of a loss-of-coolant accident to a single reactor process channel. The basic equations describing the coolant behavior in the process channel were formulated, and the development of a solution for the equations with appropriate boundary conditions was started.

MISSION 10

Experimental supercell measurements were completed in the PCTR using three 1.25% enriched uranium drivers of a reduced outer diameter to each thorium target in a 7.5 in. graphite lattice. Preliminary values of the mass of copper to poison the test supercell to k_{∞} of unity have been obtained.

An experimental study was completed of the neutron distributions in the thermal column of the PCTR. The results will improve the understanding of the activation of normalizing foils used in the PCTR- k_{∞} measurement.

Studies on the diffusion of electroplated nickel into 8001 aluminum alloy cladding were continued. Several pre and post plating treatments did not alter the diffusion characteristics.

MISSION 13

Analytical results were completed for the release of fission products from full-size, aluminum-jacketed irradiated uranium fuel elements which were thermally stressed to failure. Low noble gas and iodine fractional releases were observed.

MISSION 14

In preliminary computations involving data from the recent As⁷⁵ addition in KE reactor, it has been learned that only two active species can be detected in the effluent As⁷⁶ build-up curve. So far, there is no explanation as to why only two species are detected, whereas in the

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decay curve study by Silker, 1960, four species were found.

The concentrations of parent isotopes in film taken from an exposed fuel column were generally lower than those observed on process tube film. A longer average retention period of the parent isotopes in the fuel element films resulted in significantly higher radioisotope concentrations in the fuel element film than in the process tube film.

BEN-FRANKLIN DAM

Temperature data for the reach of the Columbia River being modeled for the Ben-Franklin Dam studies were compiled in document form.

A two-dimensional study of the effects of the proposed dam upon the 100 Areas ground water system was completed for 385 and 400-foot reservoir levels.

PROCESS TECHNOLOGY

A review of the data collected monitoring DUN process equipment corrosion over the period 6-63 to 6-66 was completed. A portion of the data is included here.

Samples from process tube 3966 KW, taken from locations immediately adjacent to those where coupons were obtained for additional reactor exposure, were examined for hydrogen. Maximum hydrogen concentrations, before removing the hydride case, 8 and 59 in. from the downstream end of the tube, were 1700 and 325 ppm, respectively. Average concentrations, after removing the case and at the same locations, were 75 and 45 ppm.

Studies on electrolytic hydriding of Zircaloy-2 in synthetic 100 Area process water have shown hydrogen pickup levels similar to those in experiments with actual process water.

The effect of temperature on the CO₂-graphite reaction was measured for several grades of nuclear graphite. In the temperature range 700 to 965°C the ratio of oxidation rates for several grades of graphite were: TSX = 1.0, EGCR = 37, CS = 38, KC = 39, AGOT-LS = 64, GLC = 68, and KS = 141.

Strength measurements were completed on samples oxidized by H₂O vapor in the large furnace. The strength was reduced by one-half after 10-15% oxidation and to one-fourth after 30-40% oxidation.

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ASSISTANCE TO DOUGLAS UNITED NUCLEAR

MISSION 1 - BASIC PRODUCTION MISSION

Reactor Boilout Studies
(Reactor Engineering Section)

The purpose of this study is to investigate analytically and experimentally the consequences of a loss of coolant flow to a single reactor process channel. In the event of a loss-of-coolant accident (by blockage or rupture), the following sequence of events may be postulated to occur in a process channel:

1. Coolant flow rates will be immediately reduced.
2. Local boiling will be initiated at some point between the center and the downstream end of the fuel element.
3. The pressure rise due to void formation will expel liquid from one or both ends of the process channel, depending on the accident and the reactor geometry.
4. Void formation will progress rapidly through the reactor channel resulting in complete steam blanketing of the fuel element.
5. Upon complete coolant expulsion, process channel pressures will decrease and coolant, if available, may again enter the reactor channel causing possible unstable flow conditions.

A complete evaluation of the boilout process must include the determination of four items at each interval of time: (1) Fuel surface to coolant heat transfer coefficients. These will rapidly change from the high values associated with local boiling to very low values associated with film boiling or mist cooling. (2) Coolant heat sink capacity. (3) Pressures throughout the system. The boilout process may create local pressures sufficiently high to damage the process tube. (4) Void distribution throughout the fueled section of the process channel.

Preliminary plans for the current fiscal year call for the following action: (1) Analytic models describing coolant behavior during the boilout process will be developed. These models will be used to determine the rate of void propagation, coolant expulsion rates, pressures,

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and fluid enthalpies during boilout. (2) Supplementary small scale experiments will be performed to test the hydraulic characteristics of process channel and fittings. (3) The information from the coolant models will be used to determine surface heat transfer coefficients for a transient conduction analysis of the fuel element. This phase of the study will provide temperature profiles in the fuel element following the loss of coolant. (4) Experimental boilout tests will be performed on a full scale mock-up of a K-Reactor process channel. These tests will verify the analytic models and will provide valuable experimental data on loss-of-coolant accidents.

These tests will be performed at initial power levels ranging from 100 to 200% of the present tube power levels existent in the K Reactor.

Work was started on the formulation of an analytic model describing coolant behavior. The equations of momentum, energy, and mass were expressed in Eulerian reference system. In their present formulation, the equations do not have closed form solutions and, therefore, various schemes of numerical solutions are presently under consideration. The most promising appears to be a method outlined by Richtmyer. It is difficult to fit boundary conditions into the Eulerian formulation of the equations, and this will comprise the next phase of the analytic study.

Neutron Physics*
(Neutron Physics Section)

Work continued of the analysis and interpretation of previously obtained scattering law results for H_2O (20 and 95 °C), D_2O , and ice.

Most of the effort on combining individual measurement runs to final cross-section values was completed for previously obtained MeV total cross section data.

Attention is called to a revised value of $\alpha(2200 \text{ m/s}) = 0.356 \pm 0.017$ for Pu^{241} from reactor irradiation measurements at Harwell which is significantly lower than that recommended from most evaluations.

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* Partially supported by General Electric N-Reactor Department.

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MISSION 10 - HIGH POWER DENSITY FUEL

Supercell Experiments in the PCTR (Reactor Lattice Physics Section)

Experimental supercell measurements were completed in the PCTR using three 1.25% enriched drivers of a reduced outer diameter to each thoria target in a 7.5 in. graphite lattice. All radial uranium buffers and all thoria targets contained the standard sized elements. Reactivity measurements in the perturbed lattices were made in the poisoned test core to determine the correct mass of copper to poison the test supercell to a k_{∞} of unity. The preliminary results of these measurements with various coolant configurations are tabulated as follows:

Poisoned Lattice: All Radial Buffers Wet:

<u>CENTER CELL</u>		<u>END BUFFERS</u>		<u>Mass of Copper</u> <u>M₀^{cu} (grams)</u>
<u>3 U tubes</u>	<u>ThO₂ tube</u>	<u>3 U tubes</u>	<u>ThO₂ tube</u>	
dry	wet	dry	wet	950.83
dry	wet	wet	wet	1008.49
wet	dry	wet	wet	533.44
wet	dry	wet	dry	536.77
wet	wet	wet	wet	475.89

Poisoned Lattice: All Radial Buffers Dry:

<u>CENTER CELL</u>		<u>END BUFFERS</u>		<u>Mass of Copper</u> <u>M₀^{cu} (grams)</u>
<u>3 U tubes</u>	<u>ThO₂ tube</u>	<u>3 U tubes</u>	<u>ThO₂ tubes</u>	
dry	dry	dry	dry	933.70

The 5 mil gold cadmium ratio measurements were made in the completely wet case. Two major irradiations were made in the completely wet case, to measure flux traverses in the graphite, fuel, target, and poison, by bare and cadmium covered copper foils. A bare copper major irradiation was made in the completely dry case. Results from the copper foil activation data are presently being processed by computer code APDAC and analysis is in progress.

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PCTR Thermal Column Study
(Reactor Lattice Physics Section)

The investigation of the PCTR thermal column was completed. Horizontal and vertical flux traverses and cadmium ratios using 5 mil thick, one-half inch diameter foil foils were obtained for three different cases: the column with no changes made; the column wrapped with polyethylene and Lucite reflectors; and the column with reflectors and a 3.5 x 3.5 x 8 in. void in its center.

Since the thermal column serves as a normalization facility for the PCTR, it was desired to determine the cadmium ratio as a function of position in the column, with and without reflector, and also to determine the sensitivity of foil activation to position, both with and without a void.

Some of the more important results are given here. The measurements on the unchanged column show the expected exponential and cosine curves for vertical and horizontal traverses, respectively. The cadmium ratio varies vertically from about 30 at the normal rotator position, $4\frac{1}{2}$ in. from the bottom of the column, to a maximum of about 280 at a distance of 12 in. from the top. These ratios were measured along the north side of the central block in the thermal column, making the traverse $1\frac{3}{4}$ in. north of the vertical center line of the column. Horizontal cadmium ratios were taken along the graphite plugs which are the horizontal center line of the column. These ratios ranged from a maximum of 200 at the center of the column to values near zero at both east and west faces.

The addition of the reflector served to increase and flatten out the total flux, varying from a 10% increase $4\frac{1}{2}$ in. from the bottom of the column to a 250% increase 2 in. from the top. The cadmium ratio showed a corresponding increase, and reached a peak of 960 at 8 in. from the top. The horizontal values lie on a fairly straight line at about 300 all the way across the column. The addition of the void in the center of the column tended to flatten the flux even more across its vertical dimension. There was a 24% decrease in the flux variation over the void between the voided and unvoided cases. A further decrease in flux variation could be obtained with a void wider than 3.5 x 3.5 in.

Fuel Plating
(Water Reactor Corrosion and Chemistry)

Studies were continued on the diffusion of electrodeposited nickel into the substrate 8001 aluminum alloy. No differences in diffusion characteristics were observed between samples pretreated by zincating or

etching. Autoclaving the plated aluminum 170°C for 20 hr in water did not change the subsequent diffusion characteristics. Tests of plate porosity showed pores occasionally developed at points of surface roughness such as scratches.

MISSION 13 - NUCLEAR SAFETY

Fission Product Release from Overheated Fuel (Particulate and Gaseous Waste Research Unit)

Additional analytical data were completed for the fractional release of noble gases, I₂, and Cs from the two K-Reactor fuels thermally stressed to failure. The heating cycle raised the temperature of the two fuel pieces to 1010°C. The first fuel element was heated to temperature in 20 min and held at temperature for 10 min. The second fuel element was carried to 1010°C in 11 min and held at temperature for 20 min. Sampling of the released material during three periods in the test showed that the major fraction of the total release occurred during the interval the fuel was held at 1010°C and then cooled. The release of Xe^{131m} during the total cycle averaged 3.8% of the calculated fuel inventory and both pieces released about the same percent. Krypton-85 was released to the extent of 0.8%. The second fuel released somewhat less than the first and the results are being reevaluated.

About 0.02% of the Cs¹³⁷ and 5% of the I¹³¹ were released in the first test. Results are not available for the second fuel. About 94% of the iodine released was elemental I₂; about 6% was associated with particles. A very small fraction (about 0.1%) was indicated to be organic compounds; however, the fraction is so small that it is possible that this may also be elemental I₂ which by-passed the Ag screens in the May-pack sampler used.

Results are in general agreement with N-fuel tests which showed low noble gas and I₂ fractional release.

MISSION 14 - COLUMBIA RIVER STUDIES

Effluent Control Program (Advanced Systems Corrosion)

As part of the continuing program to study the kinetics of river effluent activation, As⁷⁵ was added to the single pass test facility in KE reactor during June, 1966. During this addition period, samples were taken to determine the amounts of As⁷⁵ and As⁷⁶ in the effluent. To date, only data on the As⁷⁶ are available.

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BNW computer program LEARN is being used to fit the theoretical equation to the data by the use of unknown parameters. The preliminary results of these computations have been compared to similar earlier work.

In the model used to describe the system, it has been assumed that the As^{75} attaches to the wall of the channel with some probability p and detaches with a frequency B . In the present study, in which only the activity build-up has been examined so far, two species have been detected. They both have the same detachment frequency, $1.9 \times 10^{-2} + 1 \times 10^{-3} \text{ hr}^{-1}$, but one has an attachment probability of 4.5×10^{-2} and the other 1.8×10^{-2} . In comparison, earlier work showed four species, during the decay period after As^{75} addition was halted, with detachment frequencies of 2.1 , 1×10^{-1} , 1.8×10^{-2} and $2.3 \times 10^{-3} \text{ hr}^{-1}$. The attachment probability for the previously announced $1.8 \times 10^{-2} \text{ hr}^{-1}$ species was 4.5×10^{-2} , which is in good agreement with one of the values found in the present study.

Computation is being continued to determine whether there are other species present in the build-up period and what species are present in the decay period.

Film Studies

(Water Reactor Corrosion and Chemistry)

Measurements of the inventory of several radioisotopes and their precursors in film taken from selected fuel elements and downstream dummies were completed. The concentrations of parent isotopes were generally lower than those observed on film taken from a process tube; however, a longer average retention period resulted in significantly higher radioisotope concentrations in the fuel element films. The peak radioisotope concentrations in the fuel element films were five to six feet downstream from the maximum neutron flux. The concentration of parent isotopes was, in most cases, higher at downstream sampling locations. In these instances, the parent isotopes exhibited an exponential deposition pattern of the form $[C] = Ae^{-XD}$, where D is the distance upstream from the reactor rear face; A and X are constants depending on the element. The major exceptions from this trend were the inert rare earth elements, uranium, zirconium, and nickel, which evidenced maximum concentrations five to six feet downstream from the maximum neutron flux.

BEN-FRANKLIN DAM

River Data

(Environmental Studies Section)

Accumulated river temperature data, including special measurements requested by Douglas United Nuclear, from the reach of the river being modeled for the Ben-Franklin Dam studies were compiled in document form for submission to Douglas United Nuclear.

Ground Water Level Studies

(Geochemical and Geophysical Research Unit)

A two-dimensional study of the effects of the proposed Ben Franklin Dam upon the 100 Areas ground water system was completed. River flow rates of 60,000 cfs, 300,000 cfs and 725,000 cfs were considered for both the 385 and 400 ft reservoir levels. Expected increases in the altitude of 100 Area ground water mounds (at present disposal rates) for the 385 ft pool will be about 3 ft at 100-D and 4 ft at 100-K Areas during typical winter low flow conditions. At higher river stage the range will be between 4 and 13 ft for those two areas, respectively. Altitude changes will be about twice these values for the 400 ft pool elevation. The higher changes at 100-K in contrast to those at 100-D Area are attributable to the larger extent of the ground water mound at 100-K.

ADVANCED PLANNING

(Advanced Concepts and Analysis Section)

Several additional polynomial regressions were run during the month. This work completes this series of runs on the study of the effect of several reactor response variables on production costs.

PROCESS TECHNOLOGY

Dimensional Stability of Fuel Elements

(Statistics Section)

An analysis of variance was completed on X-ray diffraction data for uranium cores and contour curves drawn up showing the effect of different ingot composition and different heat treatments, quench media, and core types during fabrication upon dimensional stability index (G3 index) uranium cores.

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A calibration test was designed for Production Fuels personnel for relating 105-C Basin fuel element measurements to measurements taken at 300 Area station.

Treatment of Temperature Data (Mathematical Analysis Section)

Further progress was made on the development of an accept or reject decision method for strap-on-type resistance temperature detectors. The fitting technique used for temperature data has been generalized to include the ability to include complex exponentials, i.e., to allow for the presence of harmonics in the temperature data. This computer program has been revised and satisfactorily debugged. All difficulties which were encountered in reading the 7090 binary data tape on the 1107 using NIBAN have been overcome. The data reading operation can now be performed in an efficient manner. There remains the need for an executive program for manipulating and processing the input data prior to employing the fitting program and the response evaluation program.

Radiation Detection and Measurement Instrumentation (Instrumentation Section)

Pending receipt of comments regarding possible desired changes, development was discontinued temporarily on the experimental, pocket-size, meter-indicating, gamma dose rate meter, which provides audible signalling and has been fabricated in breadboard form using six encapsulated circuit modules.

Continued successful field test results achieved with the BNW-modified, pocket-size, gamma detection instrument, which provides a readily apparent audible signal when the preset gamma level is exceeded, served to establish the desirability for similar conversion of the other units.

Transducer Development (NDT Methods Development)

Development efforts were continued in order to provide 30 - 50 MHz ultrasonic transducers with improved resolution and sensitivity for various fuels testing applications. Lithium sulfate crystal blanks were procured and are being prepared using five newly developed techniques for producing high frequency transducers.

An improved spatial and angular manipulation system for use in general transducer evaluation work was designed and partially acquired.

An experiment was conducted to monitor the resonant frequency of a lithium sulfate disc while the surfaces were being dissolved away. These frequencies were easily monitored up to 35 MHz; however, the signals faded out above 40 MHz where the crystal thickness was still in excess of two mils. This fadeout was attributed to two causes: the slow rise time of the transmitter pulse (i.e., 25 nanoseconds where the optimum pulse rise time is $1/4$ wavelength of the crystal frequency--5 nanoseconds would be optimum for 50 MHz), and the relatively low frequency response of the scope and amplifier (i.e., 25 MHz). A scope and amplifier system with 85 MHz response is now available, and a transmitter with a 2.5 nanosecond rise time has been fabricated to facilitate the development of 100 MHz transducers.

Low Temperature Zircaloy Hydriding
(Corrosion and Coolant Chemistry Section)

Coupons, machined from process tube 3966 KW (installed November 1963 and removed December 1965), are being exposed in KW reactor in aluminum and stainless steel holders. Samples from this tube, taken from locations immediately adjacent to those where the coupons were obtained, were examined for hydrogen to obtain data which can be compared with those from the coupons after they are removed from the reactor.

Maximum hydrogen concentrations, before removing the hydride case, 8, 34, and 59 in. from the downstream end of the tube were 1700, 460, and 325 ppm, respectively. Average concentrations, after removing the case and at the same locations, were 75, 50, and 45 ppm. Data scatter associated with the analyses obtained after removing the case was too great to define concentration gradients through the wall of the tube (at the three locations along the length of the tube). In general, hydrogen concentration before removing the case (C_1) could be related to the average concentration after removing the case (C_2) by the following:

$$C_2 = C_1 / (0.011 C_1 + 3.7)$$

The coupons for in-reactor exposure were machined from a strip (about 1 in. wide) from the bottom of the tube; the above analyses were obtained from material on both sides of this strip. Hydrogen concentrations (before removing the hydride case) of samples from one side were higher than those from the other side; the values differed by a factor of almost two at the point 8 in. from the downstream end.

The electrolytic hydriding of Zircaloy-2 by galvanic contact with corroding aluminum or by application of a 3 volt source of emf to an inert anode was studied as a function of water chemistry. Zircaloy-2

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cathodes exposed 10 days to a slow flow of deionized water with added salts at 90°C were analyzed for hydrogen. The salts are added to the water to examine the effect of the various impurities present in reactor process water on the hydriding process. The pH and conductivities of all the solutions tested were adjusted with sodium bicarbonate and sulfuric acid to closely match the pH and conductivity of reactor water.

Typical hydrogen analyses of coupons thus exposed to reactor process water are from 70 to 150 ppm in the 3 volt system and 10 to 20 ppm when galvanically coupled to aluminum. Additions of CaCO_3 , SiO_2 , $\text{Al}_2(\text{SO}_4)_3$, MgSO_4 , NaCl , FeSO_4 in amounts typical of the reactor process water yielded scattered but low hydrogen (10 to 25 ppm) pickup in the 3 volt system but results typical of process water (10 to 20 ppm) in the galvanic system. Addition of sodium dichromate to the synthetic process water caused the Zircaloy to pick up hydrogen at roughly the same rate as in process water in the 3 volt system and galvanic system.

Studies on the effects of dichromate, silica and hydrogen peroxide on the hydriding process continued.

Corrosion Probes (Corrosion Engineering Unit)

Corrosion probes have been placed in selected locations throughout the Douglas United Nuclear complex to monitor the corrosion of process equipment. A total of 27 probes are installed or scheduled to be installed. Typical installations are: 105 KE and KW foundation cooling systems; 183 KE and KW raw and semi-treated water; steam lines in 100 B and D; and reactor effluent lines.

A review of the corrosion probe results from 6-63 to 6-66 has been completed and a detailed report will be issued in the near future. A portion of the data to be included in the report for the period 6-63 to 6-66 is summarized below.

1. The maximum penetrations observed in the 105 KE and KW foundation cooling systems are 0.084 and 0.075 mil, respectively.
2. Penetrations observed in the 105 D near and far inlet temperature boxes (representative of inlet header temperature) are 1.5 mils and 2.8 mils, respectively (for the period 10-63 to 6-66).
3. Penetrations observed in the 190-C water storage tanks range from 0.7 to 2.3 mils.

4. Penetrations observed in the 190-C high lift pumps (higher velocity than #3) range from 1.6 to 3.6 mils.

This comprehensive program will continue and the data will be reported at regular intervals.

Multisample Experiment

(Materials Compatibility Research Unit)

The effect of temperature on the CO₂-graphite reaction for 10 grades of nuclear graphite is being determined. The graphite types are TSGBF, CSGBF, CSF, TSX, CS, EGCR, KC, AGOT-LS, GLC, and KS. The rates of the first four graphites are complicated by changes in rate with accumulated weight loss. Therefore, relative rate data for these samples are not yet determined. The latter six graphites oxidized in 100% CO₂ in the temperature range 700 to 965°C gave the same activation energy as previously determined for TSX graphite. It is thus possible to use the following rate ratios relative to TSX: TSX = 1.0, EGCR = 37, CS = 38, KC = 39, AGOT-LS = 64, GLC = 68, and KS = 141.

Large Furnace Experiment

(Materials Compatibility Research Unit)

A fifth and final test was completed in a series of large-furnace experiments. In this series TSGBF graphite blocks were arranged to simulate the gas-flow pattern in K Reactor. Two bars, each with dimensions 4 x 4 x 44 in., were placed in the furnace and heated to a maximum temperature of 950°C. Nitrogen containing 25 to 30 mm of water vapor was allowed to flow around the graphite bars. These bars contained about 100 surface samples of TSGBF graphite and another 100 samples were located at various depths inside the graphite bar.

The primary purpose of this test was to obtain compressive strength measurements on samples with a relatively large amount of oxidation. The maximum weight loss found on an interior sample was 2.4%. Most of the interior samples were oxidized to less than 0.5%. However, the surface samples were severely oxidized, sustaining weight losses up to 44%. At the downstream end of the bar the surface samples were oxidized to only about 0.5% as a consequence of reduced oxidant concentration and inhibition by hydrogen and carbon monoxide.

The wide range of burnoffs on the surface samples made them most suitable for the strength study. The original compressive strength varied from 7000 to 8000 psi. After 10 to 15% burnoff, this strength was reduced by one-half. After 30 to 40% burnoff the compressive strength was about 1/4 of the unoxidized value.

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ASSISTANCE TO GENERAL ELECTRIC N-REACTOR DEPARTMENT

SUMMARY

MISSION 1

The material buckling of the dry lattice with standard Mark I fuel was measured. This completes a special study on water flooding effects in the exponential assembly.

Three failures, #9, 14, and 18, have shown defects in the eutectic braze closures which may have led to the observed end-associated failures.

Tests on a pressure tube discharged from N-Reactor indicate that the maximum irradiation dose received to date by the tubes has had a negligible effect on the fracture characteristics of the material.

MISSION 2

The gas released from high and low density irradiated LiAlO_2 target elements was analyzed. Gas composition was not markedly influenced by the factor of three difference in the quantity released in these elements.

Development work continued on an alternate support system for coproduct elements. Fatigue testing of projection-welded end spiders shows good performance of the basic support and weld attachments. Work is in progress to develop welding techniques adaptable to the co-product target element.

A special flowmeter was designed, assembled and delivered for the NPR-PCE Loop to allow the rapid determination of the flow splits between the inner and outer flow annuli in a column of N-Reactor coproduct elements.

MISSIONS 1, 2, 4, 6, 10

Examination of irradiated uranium alloy specimens continued to show good swelling resistance by a high-iron (1053 ppm) high-aluminum (895 ppm) additive fuel.

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MISSION 16 - OTHER ISOTOPES

BNW Master Library*

The cross sections in the BNW Master Library are being updated. Work the past month has progressed on the following isotopes: U^{236} , Pu^{242} , N^{14} , Cl^{32} , and Pu^{240} .

PROCESS TECHNOLOGY

Metallographic and visual examinations of Inconel tubing removed from steam generator 4A did not reveal any accelerated corrosion attack.

An ASTM B-147 Alloy 8B (manganese bronze) sample was found to be cracked after being wetted by a stream of water from a leaking valve in the effluent riser pit.

Preliminary oxide thickness measurements on a removed Zircaloy process tube have been made. Oxide thickness and hydrogen levels were roughly as expected for its service history based on out-of-reactor experience.

The first interim report on NPR graphite irradiations, BNWL-CC-768, was issued. The report covers the discharge and measurement of the first four boats of samples.

Graphite oxidation monitors were removed from N Reactor. The maximum oxidation observed was 0.21% per thousand operating days.

Assistance continued in the areas of preparation of $LiAlO_2$ pellets and the drawing of Zircaloy-2 tubing to final dimensions for use as co-product cladding material.

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* Partially supported by Douglas United Nuclear Incorporated and partially funded by the 04 Program.

ASSISTANCE TO GENERAL ELECTRIC N-REACTOR DEPARTMENT

MISSION 1 - BASIC PRODUCTION MISSION

Buckling Measurements in an NPR-Like Exponential Assembly
(Reactor Lattice Physics Section)

Exponential determinations of material buckling and relaxation length were completed in the N-Reactor exponential assembly. The loading consisted of standard Mark I 0.947 wt% enriched metallic uranium clad with Zr-2, a 23.2 in. tube-in-tube NPR geometry fuel. Aluminum process tubes were used and coolant channels were water or air-filled. Similar measurements were made during 1962 using an Al clad fuel with slightly different dimensions. The present measurements using the new fuel are believed to yield more accurate results plus additional measurements of flooding cases which were never analyzed clearly before.

The sixth loading was the standard dry case (air coolant, no flooding). Extrapolation lengths were found to be $\lambda_A = 5.27$ cm perpendicular to the process tubes (9 in. cell dimension), $\lambda_B = 3.72$ cm parallel, and $\lambda_Z = 3.84$ cm perpendicular. The measured relaxation distance was $B_{11} = 49.93$ cm. These results give a material buckling of $B_m^2 = 19 \pm 5 \mu B$.

FUEL AND TARGET DEVELOPMENT

N-Reactor Fuel Failure Analyses
(Metallurgy Design Unit)

The end cap from N-failure #18 (inner component) was examined metallographically and it has been concluded that the failure most probably occurred by coolant leakage through a defect in the Be-Zr eutectic braze-weld end closure. Several hot-tears or shrinkage cracks were found in the outside braze at the base of the cap adjacent to the fuel. One of these defects showed continuity from the cap base to the zone of the fusion weld. A large number of similar defects were observed in both the OD and ID braze material underlying the fusion zone of the weld. Three failures, #9, 14, and 18, showed defects in the eutectic braze closures which may have led to the observed end-associated failures.

No progress has been made in the investigation of failure #19, which appears nearly identical to #18 including the absence of the end cap, since the missing end cap has not been found.

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Failure #8, 20, and 21 were received in Radiometallurgy and will be scheduled for examination during September.

N Fuel Element Measurements
(Metallurgy Design Unit)

Measurement data from preirradiated fuel elements have been taken in the past with pneumatically operated instrumentation. Electronic instrumentation is being installed to replace the pneumatic system. It is expected that the measuring machine will be out of service for about one week.

Calibration data from the Whizz fuel element measuring machine at N Area indicated that the machine is operating satisfactorily but the computer program which converts the raw data to meaningful results is not satisfactory. Either the part of the program which processes Whizz data is in error or it is not adequate to do the job required. This phase of the program is being investigated to either correct existing errors or modify the program so that satisfactory results can be obtained.

Pressure Tube Evaluation
(Engineering Materials and Mechanics Section)

Tests continued on the pressure tube recently discharged from N-Reactor. Three slot-type crack propagation tests were conducted during the past month. A section from the downstream portion of the tube was tested with a 0.81 in. slot and failed at 73,800 psi hoop stress. This is virtually identical with the behavior of a similar tube in the as-installed condition. The adjacent specimen from the discharged tube was tested with a 3 in. slot. It failed at 29,700 psi as compared with 33,000 psi for an as-installed specimen. These two specimens, by virtue of their position near the end of the tube, had received very little irradiation. Their performance in the crack propagation test was not significantly different from that of as-installed material.

A section from the center of the discharged tube, representing an area of maximum irradiation, was also tested with a 3 in. slot. It failed at 28,600 psi hoop stress. The maximum irradiation dose received by N-Reactor tubes to date thus appears to have had negligible effect on their fracture characteristics.

Pre-Load Enrichment Tester
(NDT-Systems Engineering Unit)

Fabrication of the 100-N fuel enrichment tester which measures gamma emission from U^{235} in the fuel core, has been completed. The tester has been assembled and tested satisfactorily in the laboratory but lack of craft personnel has prevented installation at the reactor site.

Cap-to-Clad Bonding Tester
(NDT-Systems Engineering Unit)

The ultrasonic cap-to-clad bond tester (UT-9), which is used on a production basis to insure the integrity of the end closure braze metal, was overly sensitive as applied to the coproduct driver fuel element. The reason for the increased sensitivity was the difference between the nominal attenuation of the calibration standard and the nominal attenuation of the product. The cause of the attenuation difference has not been clearly defined.

A new standard was fabricated and a complete nondestructive-destructive correlation study was performed to certify the standard. The tester is now operating normally. Further work will be performed to determine the cause of attenuation differences between the old and new standard.

MISSION 2 - COPRODUCT MISSION*

Coproduct Target Development
(Fuel Materials Development Unit)

Coproduct targets of lithium aluminate ($LiAlO_2$) ceramic cores double-canned in aluminum and Zr-2 were irradiated to characterize their irradiation behavior and to provide irradiated core materials for product extraction tests at Savannah River Laboratory. The free gas volume of two irradiated aluminum canned targets, DE-036 and SV-247, was measured and samples collected for mass spectrometer analysis. The volumes and compositions of the gases were:

	Vol., cm ³						
	STP	T ₂	HT	He ³	He	H ₂	N ₂
DE-036	344	7.81	9.2	1.4	78.2	3.4	< 0.1
SV-247	980	9.22	4.27	1.33	81.7	0.7	0.9

* See also page 28 for additional reporting on this Mission.

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Target DE-036 had been fabricated using high energy impaction to yield a compact with approximately 88% of theoretical density (ρ theoretical = 2.62 g/cm³). Target SV-247 was fabricated by vibrational compaction of LiAlO₂ which had previously been bulk sintered to 82.8% of theoretical density, crushed and screened. Overall density of the vibrationally compacted target was 68.7%.

Coproduct Target Support Development (Metallurgy Design Unit)

Development work continued on an alternate support system for coproduct fuel with the principal effort being directed toward determining the fatigue strength and welding procedures for the sheet metal end spider concept.

Four arm spider supports were hand fabricated from 0.050 in. thick Zr-2 sheet. These were successfully projection-welded with four projections on outer and inner Zr-2 spacer pieces to produce fatigue specimens simulating the coproducer fuel geometry. Longitudinal fatigue tests on one projection-welded specimen with 50 lb preload and 6 lb oscillating load were terminated after 10.3×10^6 cycles without failure. Transverse fatigue tests on a second projection-welded specimen were conducted with a 20 lb oscillating load for 3.5×10^6 cycles and 10×10^6 cycles with a 40 lb oscillating load without failure. The load will be increased until failure occurs.

Brass tooling to permit electric discharge machining of spiders for use in a flow test is being fabricated by an offsite vendor. Development efforts to establish a means of attachment for the end spider are described in the section "Coproduct Fuel Support Welding."

A detailed study is being conducted of the deformation characteristics of buggy-springs in the hard-sized and interference fit configurations. A photographic record of geometry change is being obtained along with load deflection information. This information is being sought in conjunction with the overall study of the fretting corrosion process associated with buggy springs.

Coproduct Fuel Support Welding (Welding and Joining Unit)

The end support program is directed toward developing a dependable spacing device and without the buggy springs currently used. The spacing device is a relatively thin section when compared to the ends of the driver tube and target rod. A concept of fastening this support to the

to the ends of the driver tube and coproduct rod is being developed. The fact that the end closure of the driver is brazed limits the maximum temperature rise it may have while attaching the spacer. It was decided therefore to use projection welding for attaching the end supports to the driver tube and target rod. End supports of wire, wire spokes between sheet metal rings, and supports punched from sheetmetal were evaluated. The supports punched from sheet were far superior from the welding standpoint. From four to twelve projections were studied for attaching the outer ring; fatigue tests so far indicate that four are adequate.

The first attempt to attach the inner ring or hub to the target rod was by welding both ends simultaneously, the same current passing through the tube and welding both ends. As the Al tubes are not in complete intimate contact with the Zircaloy tube, the welding current passed into the Al at the points of contact and out at the other end of the tube. Heat discolored spots and holes blown in the Zircaloy-2 cladding were obtained. An attempt was therefore made to weld the attachments to the end closures of the full length Zircaloy-2 tubes that did not contain the Al tubes; none of the available resistance welders had sufficient voltage. A method is under development using concentric electrodes for welding the end support to the target rod. The end support will have a hole in the hub which allows the center spring loaded and insulated electrode to contact the end of the target rod. The outer electrode will contact the projection circle of the hub. No welds of this type have been made. Fabrication of the tooling to perform the tests is approximately 75% complete.

Welding tests on the driver tube indicated that it is possible to weld the supports on both ends simultaneously without raising the temperature of the end closure more than a few degrees Fahrenheit. The only heat observed is at the projections. The tooling is virtually the same as for the target rods but requires changing the electrical contacts. It is planned to weld the support first to the target rod and then to the driver tube by either forming the end support so that it will not touch the driver while the end support is welded to the target or by inserting a piece of sheet insulation between the driver and end support. It may be possible to alter the tooling so that these precautions are not necessary.

In case it is necessary to make a rapid change to the end support, preliminary tests indicated that it is possible to projection-weld a boss that is fully welded onto the end of the target rod. It is planned to then TIG weld the hub of the spider to the projection.

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All of this work anticipates that the driver tube and target rod either have not been autoclaved or that the autoclave film will be removed in the areas required for electrical contact and welding.

During the welding of the fatigue specimens, which were solid Zircaloy-2 with cross sections equivalent to the driver and target, it was possible to produce excellent welds in both the inner hub and the outer ring. This indicates that it should be possible to weld this type of end support to the present tube and tube assembly without difficulty.

Vibration of Coproduct Fuels and Fuel Spacers (Nondestructive Testing Section)

Vibrational resonant frequency studies continued on the fuel assemblies. Tests were made in conjunction with support spring constant evaluation tests. On both the hard-sized mockup and the actual driver-target fuel assembly, on which resonance characteristics data were taken, it was observed that as the buggy-spring supports were loosened by loading to plastic deformation, the sharp resonant frequency peak which occurred at around 250 Hz shifted downward approximately 50 Hz. This is a significant shift and should prove useful in detecting support loosening.

The main study is now being conducted in conjunction with the flow loop tests at 189-D. The resonance characteristics of each driver-target fuel assembly are recorded after initial assembly and then again after each flow test. These are then compared. Inspection after disassembly showed that, where significant wear damage to the fuel assembly has occurred, it was accompanied by a change in vibrational resonance characteristics.

Single axis (longitudinal) vibration measurements of a coproduct fuel element target under simulated service conditions were made using accelerometer equipment to provide preliminary data for use in evaluating the target support problem. More thorough data analysis with a wave analyzer supported the preliminary interpretation that the longitudinal vibration was concentrated in a narrow frequency band centering on 240 Hz with a peak to peak amplitude of 0.5 to 0.75 mil. More extensive vibration measurements on these elements will be made by Computer and Controls Section to improve data analysis with the analog computer.

Flow Distribution Measurements for N-Reactor Coproduct Elements (Reactor Engineering Section)

A special annular venturi flowmeter was designed, assembled and

turned over to GE Thermal Hydraulics for the rapid determination of flow splits between the inner and outer flow annuli in a column of N-Reactor coproduct fuel elements. Reports indicate that performance of the flow-meter was very satisfactory; measured flows appear to be in agreement with calculated values.

N PROCESS TUBE SCRATCHING STUDIES - MISSIONS 1 AND 2
(Metallurgy Design Unit)

The mechanism and factors that permit the generation of Zr-2 chips or curls during charge-discharge operations of N-Reactor process tubes was studied. Loading of the iron-shod support with an additional 100 lb force has not caused the production of Zr-2 curls by the sliding action of the iron-shod support over the Zr-2 tube surface. A sand particle lodged in the leading edge of the sliding iron shoe did, however, promote the production of a Zr-2 curl from the tube by the sliding motion of the support over the tube surface. Additional tests will be made to determine the roles of magnetite on the iron shoes, loose magnetite and loose ZrO_2 on the sliding surfaces, and surface roughness on the interaction between the sliding support and the Zr tube.

ALTERNATE URANIUM COMPOSITION - MISSIONS 1, 2, 4, 6, 10
(Fuel Materials Development)

An irradiation test was conducted to evaluate the irradiation performance of seventeen uranium alloys containing small additions of Zr, Mo, Nb, Al, Si, Fe, P, and C, in terms of composition, heat treatment, fabrication history and corrosion behavior.

Burnup determination by cesium analysis was completed on 14 irradiated specimens. These results are in reasonable agreement with recently corrected burnups calculated from the reactor operating conditions. The data showed that the actual burnups are 25 to 50% higher than originally quoted, with maximum burnups as high as 0.55 at. %.

Density measurements were performed on de-clad wafer fuel samples from two of the 0.6 in. diameter by 3 in. long irradiation specimens. These were specimens of the alloy which showed the smallest irradiation volume increases of all the alloys tested. The composition of the alloy is U + 1053 ppm Fe + 895 ppm Al + 150 ppm Si + 470 ppm C. In these two specimens the alloy had been heat treated prior to irradiation by β quenching, then α annealing in vacuum at 600°C for 100 hours. Calculated volume increases for these specimens are as follows:

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Irradiation Specimen	<u>A</u>	<u>B</u>
Volume Average Irradiation		
Temperature, °C	580	460
Burnup, at. %	0.52	0.26
Measured Fuel Volume Increase, %	4.0	1.1
"R", $\frac{\% \Delta V}{\text{at. \% Burnup}}$	7.6	4.1

The confirmation of the low swelling rate in this alloy makes this alloy attractive for further irradiation studies.

MISSION 16 - OTHER ISOTOPES

BNW Master Library*

Updating of the BNW Master Library continued with the following isotopes receiving extensive modifications:

1. U^{236} was updated by using the resonance parameters contained in BNL-325, Sup. 2, Vol. 3. The cross section data above 1 keV were obtained from the ENDF/A library with the source listed as AWRE O 30/64. The infinite dilution resonance integral for this version was 310 barns with 0.45 eV cutoff. The recommended value in BNL-325 is 400 barns but this value could not be obtained with the recommended resonance parameters.
2. Pu^{242} was also updated but the 2200 m/sec capture cross section was assumed to be 30.0 barns following the measurements at Livermore, Harwell, and Phillips. The resonance parameters were obtained from the values published in AERE - PR/NP 9 and Physical Review Vol. 146, number 3. The cross sections above 1 keV were assumed to be identical to those of Pu^{240} which is the nearest even Pu isotope. The resonance integral obtained from the record was 1175 barns for a 0.45 eV cutoff. The measured integrals average 1280 barns with an error of 60 barns. Several changes will still be made to this isotope from the available fission cross section data. This change should not affect any calculations because of its similarity to Pu^{240} .
3. N^{14} was updated using UNC-5002 as the source. Previous versions of N also used the same reference but had been updated under the limitation of only 50 points representing differential elastic scattering data. The current record has 69 such points with improved structure in the resonance region.

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* Partially supported by Douglas United Nuclear and partially funded by the Q Program.

4. C^{12} was updated with more differential scattering data. The source of cross sections was KFK-120. Differential scattering coefficients were obtained from UNC-5002. Accuracy for the total cross section of C in the resonance region is significantly improved.
5. In the updated version of Pu^{240} the resonance region has several new parameters for fission widths and the new record will reflect these changes. The new data were obtained from LA-DC-9623. The cross sections above 1 keV were obtained from AWRE 91/64. The fission cross sections in this energy region need to be re-updated to reflect the PETREL data which are much higher than the Aldermaston record in the energy region of 1 keV to 100 keV. The Aldermaston record drops to 0.001 barns fission where the Los Alamos data remain approximately 0.1 barn.

The valley at 10 eV has an experimental measurement for sigma total of approximately 32 barns and the single level parameters have always estimated a value of approximately 10 barns. To account for the vast difference, a very large potential scattering term was assumed.

PROCESS TECHNOLOGY

CORROSION AND WATER QUALITY STUDIES

Inspection of Inconel Tubes

(Water Reactor Corrosion and Chemistry Unit)

Two Inconel tubes were removed from N-Reactor Steam Generator ⁴A to determine their condition after one year of operation. Visual examination and film thickness results have been reported. Metallography at selected areas indicated minor, infrequent intergranular and transgranular surface attack to 0.4 mil deep. This attack is very similar to that found on the as-received material used in the cell 6 steam generators; therefore, it is concluded that no localized corrosive attack has occurred during the one year of operation. Samples of the tubing were submitted for radioisotopic analyses.

Inspection of Process Tube Nozzles

(Water Reactor Corrosion and Chemistry Unit)

The front and rear forged steel nozzles removed from an N-Reactor process tube after 1 1/2 yr of operation are being examined. The results of the visual examination have been reported. Photography of the nozzles and pits was completed. Samples of typical pits were cut from the nozzles and were submitted for metallographic examination.

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Oxides present in the bottoms of other pits were removed and submitted for X-ray diffraction and fluorescent analyses. Other pits are being measured for depth using X-ray and ultrasonic techniques and some will then be exposed to filtered water, others to primary system water, to determine if the pits will be reactivated.

Monitoring of Copper-base Alloys (Water Reactor Corrosion and Chemistry Unit)

Samples of several copper-base alloys are installed at various locations in N-Reactor cells, pipe galleries, etc. to determine if stress-corrosion cracking will occur due to traces of ammonia in the atmosphere from leaking equipment. When the samples were first installed, approximately two years ago, several ASTM B-147 alloy 8B (manganese bronze) samples cracked due to leaking ammoniated primary system vapors. No additional samples have cracked until this month when one cracked B-147 sample was found in the effluent riser pit. A small stream of water from a leaking valve had been falling directly on the samples.

Zircaloy Tube Evaluation (Water Reactor Corrosion and Chemistry Unit)

Tube 1756 removed from N-Reactor is being examined to determine corrosion and hydriding resulting from use. Oxide thickness measurement by metallography at the midpoint and near the outlet end were 3.1 and 4.0 microns, respectively, on the inside surface. The external oxide at these same positions was 2.6 and 3.0 microns, respectively. Hydrogen measurements at these two positions ranged from 16 to 22 ppm. Necessary corrections to these measurements to assess corrosion and hydriding during use are being developed.

GRAPHITE STUDIES

NPR Irradiations (Ceramics and Graphite Research Section)

The first interim report on NPR graphite irradiations, BNWL-CC-768, has been issued. This report covers the discharge and measurement of the first four boats of samples. All NPR samples in rod channel 74 and ball channel 80 are scheduled for discharge and reinsertion in FY-1967.

Burnout Monitoring
(Ceramics and Graphite Research Section)

Channel 1648-N in N Reactor, charged in October 1965, was discharged in May 1966. A maximum burnout rate of 0.21% per thousand operating days was measured. A burnout profile of the reactor shows considerable scatter of the data with no locations having unusually high rates.

Target Element Cladding Development
(Metallurgy Department)

Some change has been made in the tube resizing procedure in order to improve ovality and bowing of drawn tubes.

After the drawing operation the 20 ft. tubes are crooked and in some cases out of round (ovality) due to irregularities in the initial wall thickness. These tubes are stretch straightened, at 300-400°C, just enough to allow them to pass through the roll straightener. This operation greatly improves the ovality and straightens the tube but leaves it in a highly stressed condition and slightly larger in diameter than required.

The next step, stretching and annealing at 610°C, reduces the diameter to 1.337-9 in. and removes the residual stresses. A 20 to 25% improvement in yield was made by this change in procedure. Ten thousand feet of tubing has been drawn, straightened and annealed to date.

Target Element Development
(Fabrication Metallurgy Section)

Target element fabrication assistance continued for the Coproduct Demonstration Loading. Technical assistance was provided in the areas of pelletizing, powder preparation and straightening of warped Zircaloy-2 tubing. A portion of the green pellet production of GE-HAPD continues to be sintered in the Battelle facilities.

A study characterizing the current powder inventory was completed.

Vibration Studies
(Systems Analysis and Simulation Unit)

A request was received from N-Reactor personnel to provide service in tape recording coproduct target vibration signals. These signals were the outputs of accelerometers mounted in the target element. Data analysis service requested was to double integrate these acceleration signals to obtain displacements.

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Three separate tests were set up by N-Reactor personnel and tape recordings of the vibration data were taken. These data were converted to displacement on the analog computer and reproduced on the eight-channel pen recorders. The displacement data are to be used by reactor personnel to determine what design changes may be required to prevent mechanical failure in spring clips used to maintain position of the target elements.

Functional specifications to be used for bids on a tracking filter and sweep oscillator system were completed. This equipment will provide capability in vibration studies.

ASSISTANCE TO ISOCHEM

SUMMARY

MISSION 1

A trial lot of commercially produced HAPO-20 pipe received by Isochem shows some tendency to heat sensitization; this can be eliminated by an anneal at 1150°C.

A study was started to evaluate magnetic flowmeters for use with Purex flow control systems.

MISSION 5

A study to define the behavior of transplutonium elements in D2EHPA and DBBP solvent extraction systems is continuing.

MISSION 7

Further data for nuclear safety guidance were obtained on the criticality of arrays of bottles containing aqueous solutions of U²³³. Up to 18 bottles (~17 Kg U) were used in the interaction experiments. The effect of internal moderation on interaction was studied, and the effect of geometry on criticality in changing from a planar array to a cubic array.

MISSION 9

The precipitate formed with bismuth in dibutyl carbitol solutions containing polonium is believed to be bismuth oxalate on the basis of experiments done in the cobalt-60 irradiator.

Concentrations of impurities in bismuth giving rise to 100 mR/hr surface dose rates were calculated. Cobalt, tantalum, antimony and silver were the elements affecting the dose rate most at lowest concentrations.

Capacity for extraction of polonium by chemically degraded dibutylcarbitol can be almost completely restored by an acidic permanganate wash.

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MISSION 15

A new and effective complexant for use in the B-Plant D2EHPA solvent extraction system was identified. This material, nitrilotriacetic acid (NTA), is considerably cheaper than hydroxyethylenetriacetic acid (HEDTA), previously found to be the most effective complexant.

In studies to define the important factors in solids formation in Purex waste concentration, it was learned that the iron-to-sulfate ratio may be a critical variable. If this is borne out in future studies, there may be added incentive to eliminate (or decrease) sulfate in the Purex process.

Preliminary experiments have not established plant ash as a source of emulsification in 236-Z solvent extraction systems.

The presence of 2-ethylhexanol in D2EHPA reduces strontium, Ce(III) and Ce(IV) extraction under B-Plant flowsheet conditions.

Residues from some combinations of waste streams which will be evaporated in the B-Plant evaporator showed exotherms when examined by differential thermal analysis techniques.

Ultrasonic and incremental conductivity probes were modified for use with the new waste management vessel connectors.

Studies are being made of parameters relating to potential hydrogen-oxygen explosions during fission product storage in containers loaded with molecular sieves.

The applicability of burning contaminated organic solvents in the spray calciner is being investigated.

Laboratory experiments confirmed that upflow elution can be used, without excessive AW-500 zeolite bed expansion, to permit greater elution efficiency in alkaline waste treatment.

PROCESS TECHNOLOGY

The catalysis of oxidation of neptunium(V) when carbon is present in dissolving uranium was also observed when uranium carbide was dissolved. Efforts continue to establish the identity of the catalytic agent.

Although hydrazine reduces neptunyl(VI) much more rapidly to neptunium(IV) than it does plutonium(IV) to plutonium(III), so far the difference in kinetics has not been sufficient to result in co-existence of the tetravalent states of both elements.

Compton build-up factor data have been provided for revision of ISOSHLD; six absorbing materials, five energies, and seven shield thicknesses were considered.

325A Building C-Cell equipment for processing the irradiated Np from the N-Reactor demonstration has been delayed by the strike but will be ready for operation September 26.

The checkout, final modifications, and calibration of the equipment required to conduct a series of Zirflex process studies in the 324 Building were completed.

An exploratory well drilled adjacent to the Redox process condensate (S-9) crib encountered a "perched waste" zone at a depth of about 133 feet.

Initial performance of the in-tank solidification mist eliminator (employing polypropylene fibers) on the off-gas stream from the condenser showed the unit efficiency expected.

Calculations of structural temperatures in waste storage tanks are being made to allow stress calculations aimed at determining maximum safe levels of filling.

Bremsstrahlung photon spectra may now be calculated by the revised ISOSHLD Program. Source attenuation and field point dose rate logic has been coded and is being debugged. A sample problem has been prepared for use with the successfully compiled program.

The instrument for the continuous monitoring of Am^{241} concentration in a waste line at the Reclamation Facility was made ready for installation.

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ASSISTANCE TO ISOHEM

MISSION 1 - BASIC PRODUCTION MISSION

Commercial HAPO-20 Alloy
(Materials and Process Chemistry Unit)

A trial lot of HAPO-20 alloy in the form of 2 in. pipe was received by Isochem. Evaluation of this material to date indicates good corrosion resistance to nitric acid, sulfuric acid, neutral fluoride and acidic fluoride solutions when tested in the as-received condition. The material is subject to sensitization, especially when evaluated in acid fluoride solutions. A solution anneal (one hour at 1150°C) prior to sensitizing heat treatment appeared to reduce the sensitization effect to an insignificant level.

Corrosion of 304-L by Modified Citraflex Solutions
(Materials and Process Chemistry Unit)

Scouting was started on the possibilities of modifying the Citraflex process ($\text{NH}_4\text{F}-\text{NH}_4\text{NO}_3$ - citric acid) for dissolving Zircaloy cladding to make it applicable to Purex plant equipment. Zirflex dissolvent containing 0.1, 0.25, 0.50 and 1.0 M citric acid corroded 304-L stainless steel at 66, 83, 122 and 210 mils/mo, respectively. Zirflex dissolvent containing 1.0 M citric acid into which Zircaloy-2 had been dissolved to 0.67M zirconium corroded 304-L at a rate of 3.3 mils/mo. These rates are markedly higher than normally encountered in the Zirflex process.

Magnetic Flowmeter Evaluation
(Process Systems Development Unit)

Magnetic flowmeters are being evaluated for use in the solvent flow control systems in the Purex Plant. The conductivity of a series of typical Purex organic fluids was measured to determine the operating range required for the flowmeters. These tests showed that the conductivities ranged from 1.7 to 0.04 micro mhos-cm for 30% TBP after three equilibrations with equal volumes of from 2M HNO_3 to distilled water, respectively. Commercial flowmeters will operate when the conductivity of the fluid is as low as 0.1 micro mho-cm. These flowmeters will be evaluated to determine their radiation stability and compatibility with the Purex Plant flow control system.

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MISSION 5 - TRANSPLUTONIUM PROGRAM

Solvent Extraction of Pu, Am, and Cm (Fission Product Chemistry Unit)

A comprehensive laboratory study is in progress to define the behavior of selected transplutonium elements in both the D2EHPA and the DBBP solvent extraction systems. The work is in support of possible large-scale processing of some or all of these elements in 234-5 Building facilities and is an extension of earlier work on the development of the D2EHPA americium recovery and purification flowsheet (which was adopted for plant installation). Both systems appear extremely promising.

Dose Rates and Shielding Requirements for Transuranic Isotopes (Fission Product Chemistry Unit)

Last month results were reported of calculations for the Plutonium Chemistry Laboratory (Isochem) on the dose rates and shielding requirements for alpha emitters, particularly some transuranic elements. These calculations were based on spherical sources of metals. Since the dose rates are principally due to very low energy radiations (usually X-rays of 0.13 to 0.17 MeV), the dose rates are quite sensitive to the surface area of the sample. A 1 g Am source spread out in a 2 in. diam. circle would yield a gamma dose rate at one foot almost 100 times greater than the dose rate from the same amount of Am as a metal sample. Revised calculations for a few geometries are being calculated.

MISSION 7 - URANIUM-233 PROGRAM

Criticality of Interacting Arrays of U²³³ Solution Containers (Critical Mass Physics Section)

Further data were obtained on the criticality of interacting arrays of aqueous U²³³ solutions. Subcritical neutron multiplication measurements were performed with planar arrays of 9 and 16 bottles and with a double tier array containing 18 bottles. The arrays were assembled on the Remote Split-Table Machine with the critical spacing being determined for a fixed number of bottles. The effects of internal moderation brought about by positioner Lucite sheet in various thicknesses in 9 and 16 bottle arrays were studied. Data were obtained on the effect of geometry change on criticality from experiments on a double tier array comprised of 18 bottles - 9 in each layer.

The polyethylene bottles used in the experiments were 18 in. tall. The O.D. of the bottles was 4.74 in., and the wall thickness was 0.10 in.

The bottles contained uranyl nitrate solutions (acid molarity ~ 0.5) at a concentration of ~ 330 g U^{235}/l , but since the average depth of solution was approximately 12 in., each bottle contained about 960 g U^{235} .

The results of the experiments are in the following table. In the case of the bare array of 9 bottles, the maximum effect of internal moderation is obtained for a Lucite thickness of about 2 in. between bottles. In changing from a single tier of 9 bottles to a double tier of 18, the critical spacing (surface-to-surface) changed from 0.60 in. to 0.75 in. The vertical separation between tiers was 6 in.

INTERACTION DATA ON CRITICALITY OF BOTTLES OF U^{235} SOLUTION IN ARRAYS

<u>Description</u>	<u>Config- uration</u>	<u>Critical No. Bottles</u>	<u>Surface-to- Surface Spac- ing (inches)</u>
Bare array; internally moderated with $1/2$ in. Lucite between bottles	3 x 3	9	1.17
Bare array; internally moderated with $1\frac{1}{2}$ in. Lucite between bottles	3 x 3	9	1.78
Bare array; internally moderated with $1\frac{3}{4}$ in. Lucite between bottles	3 x 3	9	1.87
Bare array; internally moderated with 2 in. Lucite between bottles	3 x 3	9	1.90
Bare array; internally moderated with $1\frac{1}{2}$ Lucite between bottles	4 x 4	16	2.42
Bare array; internally moderated with 2 in. Lucite between bottles	4 x 4	16	2.50
Bare array; internally moderated with $2\frac{1}{2}$ in. Lucite between bottles	4 x 4	16	2.47
Bare array; double tier of 9 bottles in each layer	3 x 3 (two high)	18	0.75

Solvent Extraction Dedaughterizing of U^{235} Product
(Materials and Process Chemistry Unit)

A previous report cited difficulty in obtaining adequate stripping of uranium from di-sec-butyl phenyl phosphonate- CCl_4 solvent if the solvent had not been washed before use and that adequate stripping was achieved

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if the solvent was washed with sodium carbonate and nitric acid prior to use. Studies this month involved miniature mixer settler runs simulating extraction, scrub and strip operations using washed solvent. These may be summarized as follows. Adequate extraction of uranium from 3.0M HNO_3 solution was not obtained (1AX uranium losses about 0.4%). Addition of 0.1M $\text{Al}(\text{NO}_3)_3$ to the feed reduced extraction losses to about 0.025%; addition of 0.25M $\text{Al}(\text{NO}_3)_3$ reduced uranium losses to about 0.001%. Stripping of uranium was satisfactory (<0.1% loss to 1CW). Extraction and stripping was satisfactory with second pass solvent which was washed between passes.

MISSION 9 - POLONIUM

Polonium Chemistry

(Heavy Element Chemistry Unit)

It has been reported that a dibutylcarbitol-nitric acid-bismuth nitrate solution, containing Po^{210} forms a precipitate upon standing for a week or so; analyses indicated that the unknown compound was largely bismuth.

Experiments were started to identify this compound. Dibutyl carbitol (DBC) was pre-equilibrated twice with 1M Bi(III) - 3M HNO_3 solution. The separated organic phase was then irradiated in the Cc-60 irradiator for 89 hr at a dose rate of 5.7×10^5 R/hr. A fine white precipitate, which first became noticeable at about 24 hr, was filtered off, washed with methanol and dried. Comparison of X-ray diffraction patterns of this material with patterns of known Bi compounds confirmed that the precipitate was bismuth oxalate. Presumably the slightly soluble $\text{Bi}_2(\text{C}_2\text{O}_4)_3$ had been formed by reaction with radiolytically generated oxalate whose source must be the DBC.

It is interesting to note that the 89 hr cobalt-60 exposure corresponds to the radiation dose achieved in a 10^{-5} M solution of Po^{210} after 19 days. A DBC solution, saturated with HNO_3 and Bi(III), did not generate a precipitate when allowed to stand for six days.

Of concern to chemical processing is the question of whether Po^{210} is co-precipitated with the bismuth oxalate. This has not been determined. If it should be the case then limitations would need to be put on the storage of solutions containing Po, Bi and DBC.

Activation of Impurities in Bismuth (Fission Product Chemistry Unit)

Calculations were made of the dose rates in recycled Bi elements due

to neutron activation of impurities in the elements. An allowable dose rate of 100 mR/hr at the surface of the element was assumed. For the geometry of the target element (assumed to be 2.5 in. dia. x 8 in. long) this corresponds to about 3.6 mR/hr at one foot. The results are expressed as the amount of impurity which yields a dose rate of 100 mR/hr at surface at the specified time after reactor discharge. (Dose rates from the bismuth isotopes were not considered in this study.)

ACTIVATION OF IMPURITIES IN BISMUTH TARGET ELEMENTS

<u>Days After Reactor Discharge:</u>	<u>Concentration for 100 mR/hr Surface Dose Rate (ppm)</u>				
	<u>5</u>	<u>10</u>	<u>30</u>	<u>60</u>	<u>90</u>
<u>Element</u>					
Na	0.36	92	*	*	*
Mg	*	*	*	*	*
Al	*	*	*	*	*
Ca	*	*	*	*	*
Ti	*	*	*	*	*
Cr	2.1	2.4	4.0	8.5	18
Mn	*	*	*	*	*
Fe	1.6	1.7	2.0	2.5	3.1
Co	0.0012	0.0012	0.0012	0.0013	0.0013
Ni	*	*	*	*	*
Cu	20	120**	130**	140**	150**
Zn	0.26	0.26	0.28	0.30	0.33

<u>Days After Reactor Discharge:</u>	<u>Concentration for 100 mR/hr Surface Dose Rate (ppm)</u>				
	<u>5</u>	<u>10</u>	<u>30</u>	<u>60</u>	<u>90</u>
<u>Element</u>					
As	0.17	3.9	*	*	*
Mo	9.8	35	*	*	*
Ag	0.021	0.021	0.022	0.024	0.026
Cd	0.97	3.5	18	29	47
Sb	0.0081	0.0097	0.013	0.018	0.025
Ta	0.0074	0.0085	0.011	0.013	0.016
W	0.29	9.3	*	*	*

* More than 1000 ppm required for 100 mR/hr surface dose rate.

** Limit is due to Zn⁶⁵ radiation. Otherwise, limit is over 1000 ppm.

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ENCLOSURE

Solvent Extraction Recovery of Polonium (Materials and Process Chemistry Unit)

Poor scrub column performance (excessive back extraction of Po) observed in early mixer-settler tests of Po recovery flowsheets has prompted continued studies of the chemical and radiation degradation of dibutylcarbitol (DBC). Po extraction capacity of DBC decreases rapidly when (1) DBC containing extracted nitric acid stands at 50°C, (2) the DBC phase is in contact at 50°C with a 2.6M HNO₃-0.025 to 0.1M NaNO₂ solution and (3) the DBC phase is in contact at 50°C with an aqueous phase of LAFS composition (2.6M HNO₃-1.2M Bi[NO₃]₃-polonium).

The identity of the degradation reaction or species responsible for reduced Po extraction capacity has not been determined. Spectral studies on HNO₃-NaNO₂ degraded DBC indicate that organic nitrites are present with Po extraction. It has been shown that addition of a nitrite suppressor (sulfamic acid, hydrazine) to the LAFS solution prevents loss of the capacity of DBC to extract Po. Also, the extraction capacity of DBC degraded by reaction with HNO₃ and/or nitrite can be restored almost completely by washing the DBC once with an equal volume of 2M HNO₃-0.1KMnO₄ solution at 25°C. Washing degraded DBC with NaOH or Na₂CO₃ solutions did not restore Po extraction capacity.

MISSION 15 - WASTE MANAGEMENT

Selection of Complexing Agents for Purex Acid Waste Processing (Fission Product Chemistry Unit)

Prior to B-Plant processing of Purex acid waste by D2EHPA solvent extraction, a complexing agent must be added to retain certain ions (notably Fe⁺⁺⁺, Pb⁺⁺ and Al⁺⁺⁺) in solution and to inhibit their extraction. Results of an evaluation of candidate complexants was reported earlier. An additional complexant was now tested. This is NTA (nitrilotriacetic acid, N(CH₂COOH)₃). NTA was found to be very suitable for the proposed plant use. Its price is only 17¢ per gram mole, compared to 36¢ for HEDTA, the most effective reagent previously tested.

Purex Solids Problem (Fission Product Chemistry Unit)

The behavior of Sr, R.E., and other fission products during concentration of Purex Acid Waste is of concern for several reasons:
1) if more than a small fraction of the long-lived heat-generating isotopes become irreversibly associated with the Purex solids, the basis of

the Hanford Waste Management program is jeopardized and 2) some of the isotopes have substantial value and large losses cannot be tolerated for this reason. Plant tests and hot-cell experiments (reported earlier) have given inconclusive and contradictory results. Laboratory work with synthetic solutions was accordingly undertaken in an attempt to show the physical-chemical phenomena occurring in this very complicated system.

The ratio of sulfate to iron (plus aluminum) in the unconcentrated waste is a critical variable. When the $\text{SO}_4/(\text{Fe} + \text{Al})$ ratio exceeds a value of about 2, Sr and rare earths tend to precipitate. Other experiments showed that Mo, Zr, Ce, and Ru have little effect on solubility. Ba has a strong effect on Sr solubility but very little on Ce, unless present at four times the concentration currently present. Effect of Pb was similar to that of Ba.

Phosphate had little effect at any concentration. Results on the effect of Si were not conclusive, and additional work is being done. Hot-cell experiments will be performed as soon as a meaningful feed solution can be obtained from the plant. (The 40-gallon sample of PAW obtained is almost devoid of cerium and rare earths.) If the preliminary results on the importance of the sulfate ratio are borne out, it may become necessary either to add iron to the Purex waste or (probably better) to eliminate sulfate from the Purex flowsheet.

Emulsification Problems in 236-Z Solvent Extraction Systems
(Chemical Separations Unit)

A study of the factors which contribute to emulsification (and hence to flooding) in Pu and fission product recovery solvent extraction systems was started. The first endeavor was to learn something about the effect of process solids and additives on the behavior of the CCl_4 -TBP-aqueous system used in the Pu recovery facility. Experimentally, the problem to date has been approached by the measurement of disengaging times using the perforated plate plunger device. The results of the work to date are summarized as follows:

- 1) The data do not support the supposition that an emulsifying agent, extracted from plant ash and/or leach aqueous feed solutions, builds up in the organic to a critical concentration which causes the CA column to flood. Excess mistron, however, does appear to have this type of effect.
- 2) Thus far, the solids in the plant ash and leach solutions have not been observed to contribute to emulsification. Under certain undefined conditions, these solids actually cause a reduction in disengaging

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times. It must be remembered, however, that there is a great variability in the composition of incinerated materials, and therefore in the resulting ash and leach solutions. Since only a few plant systems have been tested, we cannot yet rule out solids or leach solutions as the source of emulsions.

B-Plant Solvent Studies

(Materials and Process Chemistry Unit)

Commercially available D2EHPA, the extractant used in the Strontium Semiworks and to be used in the B-Plant, is known to contain 2-ethylhexanol as an impurity. A shipment of D2EHPA recently received contained 1.3 vol% 2-ethylhexanol. Batch contact experiments were made to determine the effects of this impurity on Sr, Ce(III) and Ce(IV) extraction under proposed B-Plant flowsheet conditions. Solvents of the composition 0.3M D2EHPA-0.2M TBP-NPH - 0 to 10 vol% 2-ethylhexanol, prepared from specially purified D2EHPA, vacuum distilled TBP, as-received NPH and Eastman practical grade 2-ethylhexanol were used. Distribution ratios for Sr, Ce(III) and Ce(IV) decreased as the concentration of 2-ethylhexanol in the solvent increased. At one vol% 2-ethylhexanol, Sr, Ce(III) and Ce(IV) E_a^0 values were 3.80, 30.0 and 140, respectively; corresponding E_a^0 values in the absence of 2-ethylhexanol were 4.80, 410 and 2100. This effect of 2-ethylhexanol was observed previously for Sr and Ca at ORNL and attributed to the formation of an association complex between D2EHPA and the alcohol.

Technetium Ion Exchange Hazards Study

(Engineering Development Unit)

The heats of reaction for Permutit-SK and IRA-401 resins (following equilibration with 7M HNO_3 and a drying of the resins) have been determined in the Parr bomb calorimeter. The values for the self charring reaction in a dry nitrogen atmosphere are 470.0 ± 18.1 g-cal/g for Permutit-SK and 374.1 ± 4.7 g-cal/g for IRA-401.

Evaporation of B-Plant Wastes

(Materials and Process Chemistry Unit)

Laboratory studies are in progress to determine if there is any potential for exothermic reactions during evaporation of B-Plant waste streams.

Solutions simulating appropriate wastes were prepared and, individually or in combinations, were evaporated to near dryness. The residues were then examined, by differential thermal analysis, for temperature surges as they were heated from 30 to 500°C. No temperature

excursion was found for the residue from any single waste stream. Combinations which gave significant exotherms within the temperature range studied were LOW-Pm contactor (375°C), Cs IX-Fe Al Separation (320°C), LOW-CsIX (330°C), and CsIX-Pm contactor with excess HNO₃ (75°C).

Resin Level Instrumentation
(Process Systems Development Unit)

The ultrasonic and incremental conductivity probes were modified to operate with the new waste management resin vessel connectors.

Safety Problems in Fission Product Storage
(Engineering Analysis Unit)

A new series of tests was planned to establish more firmly the parameters relating to potential hydrogen-oxygen explosions during fission product storage in containers loaded with molecular sieves. Tests will be basically similar to previous ones with additional instrumentation to follow the deterioration through the system. The parameter of surface-to-volume ratio in the container will be studied particularly.

Disposal of Contaminated Organic Wastes
(Engineering Analysis Unit)

The applicability of disposing of contaminated organic wastes by burning in a spray calcination device is being evaluated from the standpoint of safety and economics. The small laboratory calciner, developed in connection with the waste solidification program, is being installed in the engineering development laboratory to carry on this investigation.

Errors in Waste Tank Inventory
(Statistics Section)

Consultation was continued with respect to a formula for assessing the negative error in the continuing waste tank inventory of radioactive isotopes. No further work of a substantial nature will be done till the program has been shaken down and final form of the computation determined.

Alkaline Waste Treatment
(Water and Wastewater Research Unit)

Expansion of a bed of AW-500 zeolite as a function of flow rate (upflow) was determined for both water and ammonium carbonate-ammonium

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hydroxide eluant. A bed of 20x50 mesh AW-500, 5.08 cm in diameter and 61 cm in height, was made in a glass column 199 cm high. Water and 3M $(\text{NH}_4)_2\text{CO}_3 + 2\text{M NH}_4\text{OH}$ were pumped upflow at various flow rates and the bed expansion was determined. The bed expansion was less than 1% at the design flow rate of 1 gal/min/ft² for the B-plant Cs recovery column with both water and eluant. The data confirm that upflow elution can be used which will permit greater elution efficiency.

MISSION 16 - OTHER ISOTOPES

Isotopic Separation

(Fission Product Chemistry Unit)

Work was started to evaluate the resin membrane electromigration method for the separation of isotopes. The migration cell was constructed and related equipment assembled. Experimental work will begin as soon as the membranes are received.

PROCESS TECHNOLOGY

Neptunium Chemistry

(Chemical Separations Unit)

It was reported earlier that the oxidation of neptunyl(V) in nitric-nitrous acid solutions is accelerated by an agent formed or released during the dissolution of U metal in HNO_3 . In seeking the cause of this effect, kinetic experiments have now been run with solutions containing spikes of a solution prepared by dissolving UC in HNO_3 . The resulting data indicate that the rate of oxidation of neptunyl(V) is increased by a 0.01 M uranium carbide solution to the same extent as by an 0.8 M UO_2^{++} solution from dissolving U metal containing about 600 ppm C (which gives the equivalent of a 0.01 M carbon solution if all the C is converted to aqueous-soluble compounds). The oxidation reaction rate was found to vary with the UC concentration.

Studies were also made of the relative rates of reduction of neptunyl(VI) and plutonium(IV) by hydrazine. Using a solution initially containing 0.0037 M neptunyl(VI), 0.16M hydrazine, and 8 M HNO_3 , the disappearance of neptunyl(VI) at 48°C followed first-order kinetics with a half-time of 23 minutes. After the neptunyl(VI) was all gone, the neptunyl(V) disappeared via first-order kinetics with a half-time of 35 minutes. The reduction of plutonium(IV) by 0.15 M hydrazine in 8 M HNO_3 at 48°C was also found to follow first-order kinetics with a half-time of about 4.7 hours. Thus, while hydrazine does reduce plutonium(IV) much more slowly than it reduces neptunyl(V-VI), the

difference in rates is not large enough that a solution containing essentially all of the Np and Pu in the (IV) state can be prepared by a simple hydrazine treatment of a solution containing predominantly plutonium(IV) and neptunyl(V-VI) species.

Revisions to Program ISOSHL
(Fission Product Chemistry Unit)

Bremsstrahlung calculations for inclusion in this program were completed and given to those actually writing the program changes.

Compton buildup factors were calculated for six materials, five energies, and seven shield thicknesses to extend the buildup factor information available from other sources. The results are believed to be accurate within 20% for buildup factors below 10 and 30% for higher buildup factors. These buildup factors do not include the effect of X-ray buildup, which is quite important near the K edge, especially when the attenuation coefficient for the K X ray is less than the attenuation coefficient for the primary gamma photon. This X-ray buildup is being treated separately.

C-Cell Equipment Installation
(Chemical Separations Unit)

The equipment installation in C-Cell for reprocessing irradiated Np has continued to be delayed because of the strike. It now appears that the cell should be ready for operation by September 26. Since discharge of the Np targets from N-Reactor has also been delayed, irradiated feed for C-Cell is not expected to be available until mid-October. Hence, C-Cell will be ready for this job when needed. The September 26 readiness date is probably not early enough, however, to permit the use of C-Cell for recovery of the scrap generated during fabrication of the Np target elements, since the AEC has asked that this material be returned to production channels early in September.

Zirflex Process Studies
(Engineering Development Unit)

The checkout and calibration of the equipment required to conduct a series of Zirflex process studies in the 324 Building were completed. It was found necessary to make some equipment modifications to allow the dissolver test section to reach boilup rates duplicating the plant dissolvers; these modifications have been made.

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Soil Chemistry (Water and Wastewater Research Unit)

Characterization of soil-sorbed Pu beneath the 216-Z-9 crib continued. An experiment was made to determine the amount of exchangeable Pu on a soil sample. About 4.5% can be removed with 1N NaNO_3 . This is in contrast to 0.2% which can be leached by ground water. Total removal of Pu from soil without complete soil destruction has been a problem. A reduction (Na dithionite), chelation (citrate) method was investigated and found to remove most of the Pu. Two extractions by this method at pH 7 and 70°C for 5 min each removed 90% of the Pu obtained by soil digestion ($\text{HNO}_3 + \text{HF}$) or fusion (Na_2CO_3). The dithionite-citrate method was used for iron oxide removal from soil material without detectable soil degradation.

Waste Disposal to Ground (Geochemical and Geophysical Research Unit)

An exploratory well drilled adjacent to the Redox process condensate (S-9) crib encountered a "perched waste" zone at a depth of about 133 ft. Samples of the waste contained 36,000 pCi $\text{Ru}^{106}/\text{cc}$ and 0.7 pCi Sr^{90}/cc . The higher than expected Sr^{90} concentration at this depth may be an indication that the waste is channeling downward along the outside of the well casing to become "perched" at the clay and caliche layer. There is no evidence of channeling below the low permeability layer, however, as samples of ground water from the well showed appreciably lower gross beta activity and no detectable Sr^{90} . This phenomenon is being further investigated.

Entrainment Separator Study (Particulate and Gaseous Waste Research Unit)

The previously laboratory-tested mist eliminator employing a packed bed of polypropylene fibers was installed at the in-tank solidification facility according to recommendations given to Isochem. Initial performance on the off-gas stream from the condenser showed that the unit was achieving about the same particle removal efficiency as had been determined in the laboratory, about 98%, using a uranine aerosol of 0.3 μ diameter particles. The long term pressure drop buildup will be an important index to successful operation.

Heat Transfer in Underground Waste Storage Tanks (Engineering Analysis Unit)

Calculations of the structural temperatures in the Isochem waste storage tank farm are being made from experimental data and from mathematical

models. The temperatures will be used by the Illinois Institute of Technology Research Institute in stress calculations for FEO-Isochem to determine maximum safe filling levels for the storage tanks. Approximately 400 temperatures for each of 20 tanks are to be supplied by Battelle-Northwest. A more flexible computer program for calculating steady-state tank and soil temperatures in variable geometry tank farms neared completion. A trip to IITRI in Chicago succeeded in decreasing the number of steady-state temperatures required for stress calculations.

Field data on transient wall temperatures during start-up of tanks 103AX and 104AX are being analyzed to determine the soil thermal conductivity and the instant of maximum temperature gradients in the concrete.

Analyses of the radiation probe and thermocouple readings are being made for the inside of the 103AX tank to determine the slurry, sludge, and temperature distributions within tanks containing gas lift recirculators. Several models for local temperature rises in the sludge are being considered. In the first model to be evaluated, a uniformly heat-generating sphere growing by crystallization at a constant rate in a constant temperature environment suddenly begins to redissolve at a constant rate because of addition of water to the system. The temperature in this idealized model will then be compared with experimental temperatures. No analytical solution has been found to date for this local crystallization model of in-tank hot spots.

In a visit to Idaho Nuclear Corporation at Idaho Falls, a transient temperature computer program was found that is applicable to single region buried heat sources such as fuel elements, waste containers, and buried transformers. Unfortunately, it cannot easily be used for underground waste storage tanks with vapor spaces.

Modification of ISOSHL D to Incorporate Bremsstrahlung Calculations (Engineering Analysis Section)

Coding of the revised program was essentially completed and final debugging is underway. Bremsstrahlung photon spectra may now be calculated with the code for problems having the same material capacity allowed formerly in ISOSHL D. The program has been changed to allow computation of Bremsstrahlung radiations in all twenty-five energy groups, including the nine lowest ones, which were added below 0.1 MeV. Final work has concerned adding attenuation coefficients and buildup factors for this added group structure. A sample problem containing promethium isotopes has been prepared and the gamma dose rate has been calculated for several shield thicknesses. This problem, for which there is a known experimental result, will be used to test the added Bremsstrahlung capacity of ISOSHL D.

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Separations Plant - Instrumentation (Process Instrumentation Unit)

Technical reviews provided evaluation of the multichannel analyzer data obtained, over a period of time, for process solutions from the 880 Facility. These measurements are directed toward establishing the technical bases for development of an in-line, continuous gamma measurement of the plutonium concentrations at the facility. A successful technique would offer significant savings and improved control in comparison to the present method, which involves laborious wet chemical analysis. Gamma spectral measurements of samples from the Column Concentrated Waste (CCW) unit resolved the 384 keV gamma peak, which was counted easily with the test instrumentation to provide a plutonium concentration measurement. Similar gamma spectral data obtained on Column Aqueous Waste (ACW) samples were not adequate, and additional samples will be analyzed using a different detector assembly.

To determine when the concentration of Am^{241} in a waste line at the Reclamation Facility becomes excessive, thus indicating a need for process change, the 60 keV gammas from Am^{241} are detected by a thin NaI(Tl) crystal placed to view a sample chamber in the waste line. The thin crystal detector along with energy discrimination circuits enhances the signal to noise ratio for the developmental system, where the desired detection limits are 1×10^{-3} mg/liter to 1×10^{-1} mg/liter. The detection head and the assembled instrument are ready for installation at the facility.

Fabrication was delayed temporarily on the compact, solid state, gamma spectrometer instrument, developed to measure plutonium concentrations in hoods and glove boxes at the 234-5 Building. The spectrometer design incorporates advanced solid state electronic circuitry to assure high reliability and maximum versatility.

MAINTENANCE STUDIES (Statistics Section)

Estimates were made on a rush basis of the number of welds that had to be examined on a bubble cap tower to determine 1) the integrity of the welding on bubble caps, 2) the integrity of the welds between pipes and the floor of the shelf. The estimates were to meet certain specified confidence levels with respect to the number of good welds.

ASSISTANCE TO HANFORD OCCUPATIONAL HEALTH FOUNDATION

STATISTICS SERVICES
(Statistics Section)

A rough draft report was written on the computer program designed to score and interpret batteries of personnel tests. A request has been made for further information on clinical profiles and test/re-test data.

Further analyses were carried out in connection with the reliability research program on safety.

Several requests for correlations were completed on reactor operator certification data.

Work was started on a study of all births at Kadlec Hospital for the years 1944 to 1956. The purpose of this study is to determine the possible effect of irradiation received by the parents on a number of variables, such as size and weight of the baby, gestation period, and multiple births.

Data are being accumulated for a correlation study on exempt male employees between age and education on the scores on a battery of tests. Simple correlation analyses were run on part of the data.

The available data on obesity for females were re-analyzed with a sample size increased from 47 to 61. It is felt, however, that this sample size is still too small to give reliable conclusions as to relationships present.

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TECHNICAL ASSISTANCE TO THE HANFORD PLANT

SUMMARY

ENVIRONMENTAL EVALUATIONS

Special radiation surveys along the river prior to reactor startup showed little difference from pre-shutdown levels along the shoreline within the McNary pool and downstream. The expected increase in river water activity was observed following reactor startup.

DOSIMETRY STUDIES

Work continued on the lung model and its application to the routine practice of Pu evaluation. Graphs were drawn of the expected excretion rate from one nanocurie of Pu available to the blood in two chemical forms and in three different particle size distributions. The lung model was also used in calculations relating the burden and the urine excretion rate.

PERSONNEL DOSIMETRY

No results were obtained exceeding established limits.

TECHNICAL ASSISTANCE TO THE HANFORD PLANT

METEOROLOGICAL SERVICES
(Atmospheric Sciences Section)

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

AUGUST 1966
Forecasting

<u>Type</u>	<u>No. Made</u>	<u>% Reliability</u>
Production	93	87.1
General	62	88.5
Special	99	90.9

Number of calls processed by Code-A-Phone: 4,461

ENVIRONMENTAL EVALUATIONS
(Environmental Studies Section)

Special surveys were performed at the end of the strike-caused reactor shutdown to determine radioactive contamination levels along the river. A special survey from 100-K to Richland on August 22 showed a considerable reduction in shoreline contamination and in dose rates at the routine check points. Comparison with survey data obtained in late March showed a typical reduction from 160 to 20 μ r/hr and 10,000 to 150 c/m. Readings at the 300 Area and Richland were about 25 μ r/hr and essentially unchanged over previous routine measurements at these areas. An aerial survey of the river from Priest Rapids to the ocean on August 17 showed little change from a survey made on June 24, particularly over islands downriver from Richland. Although a valid comparison is difficult because of varying river flow and the extent of exposed shoreline, the results confirm the predominance of the longer-lived isotopes, especially Zn^{65} , as the source of radiation from the shoreline and islands below Ringold.

Startup of the reactors caused the expected increases in continuous radiation measurements on river and sanitary water at the 300 Area and Richland, normal levels being reached by the morning of August 27.

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DOSIMETRY STUDIES

(Dosimetry Technology Section)

Study continued on the lung model presented by the Task Group on Lung Dynamics for Committee II of the International Commission on Radiological Protection (ICRP) and on its application to routine practice of Pu evaluation. Graphs of the expected excretion rate from one nanocurie of Pu available to blood were prepared for plutonium nitrate and plutonium oxide for particle size distributions of 0.01, 1.0 and 10 μ activity median aerodynamic diameter (AMAD). The inhaled quantities for these depositions were calculated to be 6, 11, and 9 nCi for Pu nitrate and 16, 50, and 67 nCi for Pu oxide for the above aerosol sizes, respectively. The initial lung burdens corresponding to these inhalants (after rapid clearance) were calculated as 2.7, 1.7, and 0.5 nCi for nitrate and 9, 7.5, and 3.7 nCi for oxide for the above aerosol sizes, respectively. These calculations do not include material transferred to blood from the GI tract. The Task Group on Lung Dynamics of the ICRP concluded that this amount was highly significant but offered no published experimental support.

Further calculations using the ICRP lung model predict that a urine result of 0.05 d/m found one year after an inhalation of AMAD 0.01 μ Pu oxide would imply an initial lung burden of about 7 nCi and a burden at one year of 4 nCi. The presently accepted permissible lung burden is 16 nCi. A urine result less than 0.05 d/m is not regarded as a significant deposition by current Hanford procedures. Also of interest is that for a given amount of Pu available to blood, the ICRP model predicts that the excretion rate at one year after intake is essentially the same regardless of route of entry or chemical form (citrate, oxide, nitrate) or particle size distribution.

Comparison studies of the Teflon-LiF discs with film finger ring dosimeters and other monitoring instruments were completed. Response to Pm^{147} bremsstrahlung was generally lower than with the other monitoring instruments but appears to be more consistent than film from the film finger ring dosimeter.

PERSONNEL DOSIMETRY

(Personnel Dosimetry Section)

There were 224 bioassay samples submitted for Pu analysis during the month, including 27 from new employees who had either previously worked with plutonium or who have been assigned to work with it at Hanford. No new deposition cases were confirmed by evaluation of bioassay data. Also, 171 samples were submitted for U analysis, 64 for T analysis and 35 for Sr analysis.

There were 131 examinations conducted using the whole body and/or wound counters, including six examinations requested as a result of radiation occurrences, 39 examinations of new hires and 54 terminees. Based on evaluations of the results of these examinations, three employees received trace depositions of Zn^{65} and one employee received a deposition of 0.02 μCi Pu in a wound. The wound was excised and the evaluation of internal deposition will be based on bioassay results. Evaluation of the results of the rest of the examinations does not indicate any internal deposition in excess of 1 percent of the applicable permissible limits.

Special external exposure evaluations were performed for 26 Hanford contractor employees; however, no exposures over permissible limits were revealed.

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

Records processing of 2,338 employee beta-gamma film badge dosimeter results, 216 neutron results, 251 finger ring results, and 367 visitor film dosimeter results was conducted. None of the results exceed limits established in AEC Manual Chapter 0502.

AQUATIC BIOLOGY STUDIES
(Biology Department)

Average columnaris agglutinating titers of 1:1000 were developed in experimental yearling rainbow trout using heat-killed columnaris plus adjuvant. Individual high titers up to 1:2560 were found. A virulent C. columnaris strain, isolated from the McNary ladder, capable of producing 90% mortality of young trout within 48 hours, will be used for various immunization studies with the experimental trout. Further evidence of columnaris exposure and infection in fish ladders was obtained by comparison of ladder fish and fish gill-netted several miles downstream from Bonneville Dam. Average titers of Bonneville ladder suckers were 1:500 as compared to 1:250 for the gill-netted suckers. Three of 10 ladder suckers were infected whereas only 1 of 15 gill-netted fish was infected.

Aquatic ecosystems are being established in troughs at KE preparatory to testing the biological availability of radionuclides in reactor effluent treated with ferric salts. Over-growth of algae necessitated the shading of the troughs. Initial stocking was made with young carp and periphyton-bearing rocks.

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An excellent photographic mosaic of the Columbia River from Priest Rapids Dam to Richland was made from negatives borrowed from the U. S. Corps of Engineers. This aerial photograph will serve many useful purposes. For example, enlargements of the island photographs will permit vegetation mapping needed for the proposed study of Canada geese. About \$2,000 in photographic services was saved through this cooperative effort.

INSTRUMENTATION SERVICES

Laundry Monitoring System (Process Instrumentation Unit)

Fabrication and testing continued with a significant advance achieved through completion of the lower detector probe assembly and electronics, and through mechanical fabrication and assembly of the upper conveyor frame, shafts, bearings, and support rollers.

Tests conducted with the lower detector probe indicated excellent alpha to beta-gamma discrimination, with reliable detection and trip indication for 500 dis/min of Pu^{239} and 3.0×10^4 dis/min of RaDEF . These sources were easily detected when moved over the probe with a conveyor belt speed of 80 in. per min. The alpha channel count rate calibration is approximately 50 counts/min full scale with a beta-gamma full scale calibration of 5×10^3 counts/min; the time constants are seven seconds and five seconds, respectively.

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