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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
FOR AUGUST 1967
DIVISION OF PRODUCTION
AND
HANFORD PLANT ASSISTANCE
PROGRAMS
SEPTEMBER 1967

AEC RESEARCH &
DEVELOPMENT REPORT

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C-65, Plutonium Production
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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
AUGUST 1967

Division of Production
and
Hanford Plant Assistance
Programs

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

September 1967

PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON

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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

Effect of Nuclear Effluent on the Quality of Columbia River Water

Data collected in support of this program have had additional use in development of the technical facts regarding proposed water temperature standards. A report summarizing the mathematical modeling of the Hanford plumes was issued during the month.

Mechanisms of Environmental Exposure

Modification of the mobile whole-body counter to permit utilization of punch-paper tape for diet data and personnel information was suspended in August pending receipt of necessary parts. Calibrations of the whole-body counter detector for Na^{24} , K^{40} , Zn^{65} and Cs^{137} began at month's end and should be completed in early September. Pasco school officials have approved the schedule which calls for utilization of the mobile whole-body counter for measuring radioactivity in children at the six Pasco elementary schools during the coming school year.

U^{235} Analyzer

Completion of minor modifications to improve noise interference operation permitted return of the U^{235} analyzer and spectrometer to the AEC-Division of International Affairs, thereby effecting termination of the project except for the final report.

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DIRECT AEC SPONSORED PROGRAMS

Columbia River Studies

Effect of Nuclear Effluent on the Quality of the Columbia River Water

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

Extensive use of the data stored as a result of analysis programs supported by the study has been made in regard to the proposed water temperature standards.

The 7090 version of the concentration model for use by Environmental Health Department was delivered. Discussion relative to tests and utilization in the emergency planning activities of that Department are under way.

Further development of the use of the LEARN computer routine for the least squares analysis of water quality data was completed during the month. A successful model of the form:

$$Q \text{ (flow in cfs)} = A + B e^{-\left(\frac{d-C}{D}\right)^2}$$

A = a constant related to minimum flow,

B = a constant related to maximum flow,

d = day of the year,

C = day of year where peak flow occurs,

D = 2σ for fitted normal curve,

was used to generate a series of functions for the U. S. Geological Survey water years 1937 through the present. The successful application of this model in conjunction with the sinusoidal functions previously developed for temperature analysis allows direct relationship of the plant heat output with the regional energy output for extended time periods.

A published analysis summarizes a portion of the Hanford plume data collected during calendar years 1964 and 1965 and suggests a series of mathematical models by which the size and temperature distribution of the Hanford discharge plumes can be

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predicted. A striking relationship to the general rules governing all dispersion phenomena is noted.

Mechanisms of Environmental Exposure

(J. F. Honstead, Environmental Studies Section)

Modification of the mobile whole-body counter, to incorporate a Serial Number Device which would permit use of punch-paper tape output for personnel and diet data, was delayed pending arrival of two stepping switches. Other modifications completed to date included shock mounting of the electronic instruments, further improvement of the height gauge to permit rapid return of the whole-body counter bed, and complete checking and repairing of numerous minor faults in the 1024-channel analyzer. Calibration of the detector for Na^{24} , K^{40} , Zn^{65} and Cs^{137} began at month's end and will be completed in early September.

The mobile whole-body counter will be used throughout the coming school year to measure radioactivity in children at the six Pasco elementary schools. The detailed schedule to be followed in Pasco was presented to the six elementary school principals involved, at their staff meeting on August 22, 1967. The school officials indicated that the schedule was acceptable and reaffirmed their willingness to cooperate completely in the project.

Work on the final report of the statistical survey of fishermen utilizing the Columbia River in the vicinity of the Tri-Cities continued. Essentially all of the graphs and tables necessary were completed. The computer file containing the survey data was utilized to perform additional calculations, including a comparison of the total number of fishing trips made in a year calculated in two ways. As expected, the number of fishing trips based on fishermen's estimates was significantly higher than the number of fishing trips calculated from numbers of fishermen observed during the survey.

U²³⁵ Analyzer

(W. G. Spear, Instrumentation Section)

Return of the instrument to the USAEC Division of International Affairs will permit its routine use in measuring the U²³⁵ content of unirradiated fuels and in general purpose gamma spectrometry. This compact, portable gamma spectrometer was developed for use in safeguarding inventories of U²³⁵ contained in nuclear reactor unirradiated fuel elements or in various other sources.

Main chassis solid state circuitry, excluding the scintillation probe and preamplifier, comprises a wide-range high voltage supply, double delay line pulse amplifier, split-window gain stabilizer, single channel pulse height analyzer, six decade scaler, automatic timer, count rate meter, low voltage regulators, and a battery charger. Instrument power is supplied by a 3.5 lb, 24 V, 2 A-hr NiCd rechargeable battery, also located in the 17 x 13 x 3 inch main chassis, providing a maximum of 20 hours continuous operation. All electronic circuits, except the battery charger are fabricated in plug-in modules. The main chassis mounts in a glass fiber transit case, measuring 14 x 19 x 6 inches, which provides approximately one inch of form padding to the chassis and components. Opening the hinged transit case lid exposes the main panel and module controls for convenient use by the operator. An identical transit case with shaped padding is provided for the probe with preamplifier, collimators, and probe stand.

Submission of the formal technical report essentially completes the project with only minor tracing preparation and modification remaining.

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ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Summary

Mission 1

Simulated rupture testing of a defected, high exposure, Mark I inner component in the IRP facility was completed. A surprisingly long incubation period, low fuel corrosion, and slight rupture damage was experienced. Improved corrosion resistance of the high burnup fuel appears to be the explanation for the behavior.

Forty-eight inner and outer components (Mark IV) were pre-irradiation weighed.

Room temperature measurements during a 400°C N-Reactor pressure tube stress rupture test indicated 3.93% maximum hoop strain after 7100 hours at 25,000 psi hoop stress.

The vibration tester system for N-Fuel assemblies was delivered to the production area and is available for on-line use.

A wall thickness measurement test was evaluated on zirconium billets at various stages of extrusion. Measurement accuracies of ± 0.005 in. were obtained with this pulse echo method when the billet impurities were evenly distributed.

Development was initiated on an ultrasonic test to distinguish between the alpha and beta phases in N-Fuels after heat treating. Initial attempts were unsuccessful because the needed transducers were not available; however, test efforts will be resumed upon receipt of the proper transducers.

Development of an ultrasonic fuel core crack detection system was completed and the test is ready for production line use. This system was designed to detect cracks with 50 mil radial and 1/2 in. longitudinal minimum dimensions. It is sensitive to cracks at all points on the core radius.

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Design of the new tester chassis for the ultrasonic system in 313 Building was about half completed.

An analysis was completed evaluating several sets of hypothetical percentages to illustrate the partial separation that could be expected in the classification of four U alloys according to observed performance in the incidence of core cracking in the upcoming production test A083.

The computer program prepared for calculating the volume change in U cores during irradiation was applied to the data in PT-A040; agreement with water displacement results on eight test pieces was only fair.

Mission 2

A weld failure of the outer Zr-2 can closure of a ceramic core target component was verified and was concluded to have been caused by internal end loading from thermal expansion of the inner aluminum-ceramic composite.

Raidometallurgy study of high power density fuel elements containing uranium of several compositions and heat treatments has continued. Density measurements obtained on declad uranium sections from each of five different fuel elements showed no difference in swelling behavior between elements of the nominal "adjusted" uranium and elements of "adjusted with silicon added," both having 2% ΔV at 2400 MWD/T exposure. At the same exposures the "high silicon" alloy of U + 150 ppm Fe + 350 ppm Si + 40 ppm Al swelled slightly more, 2.3% ΔV , and the standard alloy U + 150 ppm Fe + 110 ppm Si + 50 Al swelled 3.2%. Metallography revealed extensive porosity in the control zones of the fuel of the four tubular elements examined. Electron microscopy completed on one element indicated that the porosity is characteristic of the grain boundary tearing type but that there is also some evidence of the "aligned" type porosity.

Mission 4

A process was developed for the fabrication of graphite-neptunium oxide pellets.

Preparation of the eight neptunium oxide-graphite target elements was completed.

Mission 5

ETOM, the code for processing ENDF/B data into multi-group format as required by MUFT and HAMMER, has been successfully converted to the UNIVAC 1108.

EDIT, the processing code for editing the ENDF/B library tape, has been modified to permit editing of secondary energy distributions.

A short code has been written to compare HRG data tapes and to assist in the detection and elimination of errors. Improved processing methods for generating HRG data tapes from the BNW Master Library are being incorporated into the processing code, BARNS II.

An 1108 version of the computer code THESEUS modified to calculate collision probabilities and THERMOS type T matrixes for cylindrical lattices with arbitrary radial leakage is now available. An estimated savings in THERMOS running time of over 50% may well be possible using THESEUS as a subroutine.

Mission 7

X-ray diffraction results for four intermetallic compounds that had been prepared indicated that NiU_6 , Ni_2Al_3 and NiAl_3 were essentially single phase. The patterns obtained for Ni_5U were inconclusive and are being rerun.

Operational difficulties have prevented forming long aluminum alloy ingots in the direct chill casting apparatus. Sections of short ingots show metallographically that the fine second phase structure can be obtained where there is sufficient chill to quench the structure.

Out-of-reactor tests of nickel plated fuels with and without defects in the plate did not show any localized corrosion.

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Analysis was completed of several perturbation measurements made in the supercell 2.1% enriched uranium 2.93 wt% lithium in aluminum target, in a 3-1 ratio, to determine the reactivity changes due to limited water loss in the target and fuel. The reactivity decrease due to loss of coolant water in the target cell alone is estimated to be 12 ± 3 mk.

The infinite medium multiplication constant, k_{∞} , for the 2.1 wt% enriched uranium lithium-in-aluminum target wet supercell in a 3-1 ratio has been calculated ($k_{\infty} = 0.9975$) and compares reasonably well with experiment ($k_{\infty} = 1.0035$).

The PCTR experimental measurements of the bismuth overbore and KVE supercell lattice (3-2 fuel-to-target ratio) were completed.

Mission 8

An increase in the tritium concentration in waste discharged to the 1301-N crib in late July is being followed through a program of sampling wells and springs to obtain more information on the flow system at this site.

Mission 10

The effectiveness of activated silica as a coagulant aid was evaluated under three different conditions of water treatment. Use of these waters as reactor coolant in no instance provided significantly beneficial effects in the concentrations of effluent radioisotopes. It is felt that further experimentation with the use of activated silica for this purpose is unwarranted.

Steady state concentrations of various effluent radioisotopes have been established since conversion of the deionized water transport system to stainless steel. The study of reconstitution of process water will be started in the near future.

Process Technology

An arc melting and casting process for producing high purity Be-Zr braze alloy components is being studied. Nine primary melt ingots have been melted and are being assembled for second melt electrodes. These will be assembled by threading due to circumferential weld bead cracking in electron beam weld joints.

Pitting up to 2 mils deep occurred on carbon steel crevice samples and carbon steel to stainless steel weld samples during laboratory and reactor phosphoric acid decontamination tests.

Corrosion samples removed from the half-plant dichromate test of B-Reactor did not show any ledge or groove corrosion. Data from electrically measured corrosion probes indicated that the 1/2 ppm dichromate is slightly more corrosive than the 1 ppm dichromate.

Tests of aluminum samples in high flow process water with applied potentials displayed some anode pitting.

Two Zircaloy process tubes from K-Reactor containing special varieties of anodized spacers were examined for hydrogen content. Hydrogen levels were low.

Twenty polyelectrolytes are being evaluated as filter bed conditioners. The performance of each is evaluated relative to Magnifloc 990 for headloss, effluent turbidity, bed penetration, and run length.

Progress was made on the procurement of analytical instrumentation for monitoring the N-Reactor cover gas.

Further studies of required performance characteristics of personally carried dose rate and dose monitoring devices were started to aid in satisfying such needs for the operating reactor locations.

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ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Mission 1 - Basic Production Mission

Simulated Rupture Testing of High Exposure Mark I Fuels
(J. W. Goffard, Metallurgy Design Unit)

High exposure Mark I inner and outer components from near the center (Position #9) of high exposure tube 2855 (~5300 MWd/T tube average) are being rupture tested in the Irradiated Rupture Prototype (IRP) facility. The 6500 MWd/T exposure inner component, which contains Fe-Si additive fuel, was defected near its center with a 0.025 in. diameter hole through the clad to the fuel. The component was exposed for about three hours to 300°C, 1650 psi, flowing water in the IRP facility without incident. Since the defected component did not rupture it was returned to Radiometallurgy for redrilling. Visual examination of the component showed it to be unaltered but probing of the drilled hole indicated that the fuel was adequately defected and should have failed. The element was redrilled at a site several inches from the initial defect and returned for retesting in the IRP facility. After testing for about seven additional hours the component ruptured as indicated by the increasing activity in the IRP loop filter. The ruptured element was returned to Radiometallurgy for study. Visual examination revealed that the clad had blistered and split through the initial defect. The second defect showed some superficial staining but no deformation of the clad. Metallographic examination through the sites of both defects is under way. There is some loss by fuel corrosion at the site of the rupture but there has been no severe fuel cracking or fuel fragmentation to accelerate corrosion attack and rupture damage. The mode of failure and damage is circumferential cracking and corrosion of the fuel at or adjacent to the bond with the development of massive hydride on the inside clad surface. The manner of fuel corrosion in the radial direction appears to be uniform and at a lower rate than for unirradiated fuel. Study at the site of the unruptured second defect shows a uniformly shaped pocket of corrosion products with evidence of what is thought to be uranium hydride in the adjacent fuel. Study is continuing and IRP testing of the outer component is planned for next month.

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N-Fuel Measurements

(R. K. Marshall, Metallurgy Design Unit)

During the reporting period 48 inner and 48 outer Mark IV fuel components were weighed. Because of time limitations the components were not measured.

N-Reactor Component Evaluation; Pressure Tube Evaluation

(R. J. Evans, Materials Engineering Function)

A study is being conducted to determine the effects of hydrogen absorption, irradiation, aging and other in-service conditions on the properties of N-Reactor pressure tubes. The work includes investigations of creep-tensile properties, brittle fracture and fatigue.

Room temperature circumferential measurements of an N-Reactor pressure tube specimen during a 400°C stress-rupture test indicated 3.93% maximum hoop strain after 7100 hours at temperature. This tube, at 25,000 psi hoop stress, has now been at temperature over 2800 hours longer than an earlier tube which was pressurized at a hoop stress of only 20,000 psi. Although the lower stress test failed sooner than had been expected, the current test has given more confidence to our prediction of long term stress-rupture properties of Zircaloy-2 tubing. The long term predictions are based on the curve of hoop stress versus log failure time made from a number of earlier, higher stress tests.

Initial success was achieved in adapting a computer program obtained from offsite sources to run in machines onsite. This program calculates and then plots the stress intensity at the tip of a growing crack in a large flat plate caused by point loads at various distances from the crack tip. Work is now continuing to make this program more closely fit analytical models of "damaged" N-Reactor pressure tubes.

Vibration Test

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

The vibration tester for N-Fuel assemblies was delivered to

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the 333 Building and is available for production line use. Analysis of support structure characteristics in tester-rejected fuel assemblies is expected to begin shortly.

Billet Wall Thickness Test

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

A wall thickness measurement test was established for use on zirconium billets throughout various stages of extrusion. An accuracy of ± 5 mils was obtained by pulse echo in pure billets of approximately five inches initial wall thickness. Several billets were found to contain unevenly distributed impurities, thereby voiding the test results because of the varying ultrasonic velocity.

Alpha-Beta Phase Transformation Measurement

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

An ultrasonic test was started to distinguish between the alpha and beta phases in N-Fuel pieces after heat treating. The phase transformation should be discernible by a grain size difference in the uranium. Initial attempts were unsuccessful due to inappropriate transducers. More desirable search units were secured and test efforts will be resumed.

Fuel Core Crack Test

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

Work on a bare core crack tester was completed and the device is ready for production line use. The test is primarily designed to detect cracks of 50 mil radial and 1/2 in. longitudinal minimum dimensions. It is sensitive to cracks at all points on the core radius.

Ultrasonic Tester Redesign

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

Development of a universal ultrasonic tester for needed replacement of old-style tester chassis is well under way. Design criteria for most of the circuitry have been established.

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Literature and vendor searches are currently in progress for final selection of components.

The new model will fully utilize state-of-the-art design techniques, and will employ integrated circuits whenever possible. IC video amplifiers will provide wide-band gain (to 50 MHz) and timing will be accomplished with high level integrated logic. Several types of pulser PC boards will be supplied, one of which will provide independently variable pulse width, and rise and fall times.

Statistics Services

(H. D. Huber, Statistics Section)

Design of new tester chassis for the ultrasonic system (UEI) in the 313 Building production lines is 50% complete. The new design utilizes state-of-the-art design techniques and employs integrated, solid state circuitry whenever possible. The objective of this work is to provide a design for suitable replacement equipment for the 10-year-old UEI systems.

An analysis was completed evaluating several sets of hypothetical percentages to illustrate the partial separation that could be expected in the classification of four uranium alloys according to observed performance in the incidence of core cracking during irradiation in the upcoming production test A083. The charging pattern tentatively allows for the irradiation of forty tubes of each alloy, charged in separate tubes, with eight to ten tubes of each alloy to be discharged and tested for core cracking during irradiation at each of four or five successive levels of tube exposure. Standard procedures for nonorthogonal multiple comparisons were used in analyzing the hypothetical data, after use of an averaged angular transformation proposed by Freeman and Tukey for stabilization of variance. Care was taken to point out several alternatives for defining the population of test cores in the charging design so as to reasonably satisfy the assumption of a binomial population. After receiving the data, the dimensional stability of the alloys will also be evaluated at the successive levels of tube exposure to supplement the information obtained in PT-A040 on the dimensional stability of the alloys.

Application of the computer program, previously prepared for calculating the volume change in uranium cores during irradiation, was completed to the data in PT-A040. Agreement with results from water displacement tests conducted on eight test pieces in PT-A040 can at best be described as fair. However, this was not adjudged to provide a sound test of the premises involved in developing the computer program, since personnel in Fuels Engineering did not have much confidence in the water displacement results. Consequently, the program results were deemed worthwhile analyzing and assistance was provided in evaluating the effect of the test treatments in PT-A040 upon the computed volume changes. The analysis was performed using the series of Latin squares occupying the central positions in the charging pattern, where the environment for dimensional distortion was not severe.

Analysis of the remeasured data in PT-A004 was resumed to evaluate the effect upon dimensional stability of the HDS core during irradiation of two heat processing treatments and the concentrations of three uranium core additives investigated by means of a cuboctahedron design replicated for each of the two heat processing treatments. The data are being analyzed by combining the results from Youden square and triangular partially balanced incomplete block charging patterns.

Mission 2 - Coproduct Mission

Examination of Cracked Closure of Ceramic Target (J. W. Goffard, Metallurgy Design Unit)

A 1.3 in. diameter coproduct ceramic core target component has been under examination in the Radiometallurgy facility to verify the existence of a suspected weld closure crack and to determine its cause. Early study of the target failed to reveal a cracked closure even though the component was visually examined, dye penetrant tested, and inspected for the presence of gas in the double can interface. No cracks were observed and no gas pressure was detected in the interface region. Subsequent leaking of a white material from the inspection hole drilled into the interface hinted that the outer closure must have indeed leaked. The end closures were cut from the target and the outer can was slit open. The presence of white aluminum corrosion product on the inside of the outer Zr-2 can indicated that water had leaked

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into the double can interface region. The end closures from the outer Zr-2 can were carefully reexamined and a cracked weld was found. There was nothing superficially evident such as Zr-2 corrosion or mechanical damage associated with the cracked weld. Metallographic study of the cracked closure did not reveal any manufacturing deficiencies in the materials or the weld, but did show that the weld bead had failed by cracking, presumably by a shearing force. Since no mechanical damage could be found on the exterior of the element, preirradiation handling and discharge forces are not strongly suspect as a cause. It appears more probable that expansion of the inner aluminum can and ceramic core led to the closure failure.

High Power Density Alloy Fuel Evaluation
(J. W. Weber, Fuel Materials Development Unit)

Examination of fuel elements containing alloys being evaluated for high power density applications has continued in Radiometallurgy. Ten elements with a variety of compositions and heat treatments have been selected for detailed postirradiation examination with optical metallography, replications and electron microscopy, density, and hardness. Five of these elements operated at or near a maximum uranium temperature of 500°C. This would place them in the temperature range where swelling by grain boundary tearing can occur. The element identification, nominal compositions, and heat treatments are as follows:

<u>Element</u>	<u>Alloy Type</u>	<u>Composition</u>	<u>Heat Treatment</u>
51P21	Standard	U + 150 ppm Fe + 110 Si + 50 Al	Induction heated - oil quench
51P20	Adjusted w/Si added	U + 350 ppm Fe + 350 Si + 800 Al	Induction heated - oil quench
51P19	High Sili- con	U + 150 ppm Fe + 350 Si + 40 Al	Salt heated - oil quench
51P22	"Adjusted" (British)	U + 350 ppm Fe + 40 Si + 800 Al	Salt heated - oil quench
51P18	"Adjusted"	U + 350 ppm Fe + 40 Si + 800 Al	Induction heated - oil quench

Density measurements, obtained on de-clad pie sections of wafers cut from each of the above elements, were used to calculate postirradiation volume changes and R* values as follows:

<u>Element</u>	<u>% ΔV</u>	<u>Exposure MwD/T (At.% BU)</u>	<u>R*</u>
51P21	3.16	2407 (0.30)	10.5
51P20	2.0	2425 (0.30)	6.7
51P19	2.3	2443 (0.30)	7.7
51P22	1.95	2362 (0.29)	6.7
51P18	2.0	2434 (0.30)	6.7

$$R^* = \frac{\rho_{\text{initial}} - \rho_{\text{final}}}{\rho_{\text{final}}} \times 100 \times \frac{1}{\text{At.\% BU}} = \% \Delta V / \text{At.\% BU}$$

Under the irradiation conditions of these elements there appears to be no difference in swelling behavior of the "adjusted" uranium in both heat treated conditions and the "adjusted" with Si added. The high silicon uranium exhibited a slightly higher swelling rate and the standard uranium alloy had the highest swelling rate.

Metallography was completed through photographing in the as-polished conditions on all elements except 51P22. Examination of the four elements on the metallograph reveals extensive porosity extending in a central band of more than 50% of the thickness of the tubular fuel cross sections. The band extends from within 0.07 in. of the inner bore uranium surface to within 0.13 in. of the outer uranium surface. Electron replicas obtained on sample 51P19 show porosity ranging from several microns in size down to less than 0.1 micron. There is grain boundary tearing evident in this sample of the high silicon alloy although the tears are not as large as are frequently observed with this type of porosity. There is also evidence of the aligned type of porosity generally observed at irradiation temperatures higher than those calculated for these elements. Further metallographic examination through replication and electron microscopy is planned for these five elements.

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Mission 4 - Plutonium-238

Preparation of Neptunium Target Elements

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

A graphite-NpO₂ dispersion target element was developed in support of a DUN program to produce Pu²³⁸ with very low Pu²³⁶ contamination. Graphite does not give rise to high energy gamma emission which, through a γ -n reaction, is one of the sources of Pu²³⁶ contamination in an Al-Np alloy target. The target element, K Reactor type, will contain 40 g Np and will be aluminum clad, 9 inches long and 1½ inches diameter. The target element will contain pellets fabricated to size by cold pressing the blended graphite and NpO₂ powders. The pellets will be vacuum outgassed and inserted into an aluminum can. After closure welding, the can will be isostatically pressed to eliminate diametral assembly gaps.

During the short term irradiation, the target elements will operate in the temperature range of 300 to 500°C. Under these conditions, internal pressures may result from internal gas pressure, thermal expansion of the pellets, and radiation damage to the graphite. To accommodate these expansions, six percent void space will be built into the target elements by dishing the ends of the pellets. In addition, the pellets themselves will contain between ten and fifteen percent internal void volume.

In prior work, oxide-graphite pellets were fabricated to the exact size required with up to twice (15%) the oxide content required, and the high initial gas content of the graphite was reduced to 0.02 cc/g through vacuum outgassing at 700°C.

Neptunium Oxide - Graphite Target Elements

(H. T. Fullam and C. J. Mitchell, Fission Product Chemistry)

Preparation of the eight target elements by the impregnation process was completed.

Mission 5 - Other Isotopes

Code Development*

(C. W. Lindenmeier, Theoretical Physics Unit)

BARNS-II - J. L. Carter

BARNS-II has recently been revised to permit numerical integration of group cross sections in energy ranges which use resonance parameter tables. Some calculations using this option have converged slowly in the integration over the wings of a resonance when a broad group is used. Several methods for improving this convergence are being examined in an effort to speed the calculation.

Background calculations done by BARNS-II for some nuclides for the HRG data tapes have been higher than expected. A probability of reducing this background by including parameters for lower energy resonances which have been omitted from the resonance parameter tables for these nuclides is being tested. A special operating procedure for BARNS-II has been developed for these tests.

HRG Data Tapes - J. L. Carter

Some users of HRG have reported details of errors in data for some isotopes; these errors have apparently been introduced during several special-purpose data manipulations during the past few months. While known errors can readily be corrected, their unexpected presence raises the serious question that other errors have been introduced that are not yet detected. To check this possibility, a short code has been written to compare data on HRG library tapes in some detail and provide information on points of disagreement. The code is being debugged.

ETOM - K. B. Stewart

Recent modifications received from WAPD have been incorporated

* Partially supported by the 04 Program.

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into the computer program ETOM, which calculates multi-group cross sections for MUFT and HAMMER from the ENDF/B library. Previously, ETOM generated incorrect output when the input data contained negative or thermal resonances. The calculations now appear to be correct.

EDIT - K. B. Stewart

The computer program EDIT, which is used to print, punch, or copy an ENDF/B neutron cross section library tape, was modified to allow the manipulation of the secondary energy distributions contained in File 5 on the library. The necessary programming to handle File 5 was not included in EDIT as it was originally distributed. Several successful debug runs have been made to test the modifications.

Code Modifications

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

A modified version of the computer code THESEUS was debugged and is now available for use on the UNIVAC 1108. THESEUS calculates monoenergetic first flight collision probabilities for an up to 60 annular region infinite length cylindrical lattice. The collision probabilities in the original version were calculated for both the isolated lattice and for the lattice surrounded by an infinite array of like lattices. The original version was modified to allow the calculation of first flight collision probabilities for systems with arbitrary radial leakage (either in-leakage or out-leakage) using an inputted outer boundary albedo supplied by the user. At present the modified version also calculates the T matrix as would be used by THERMOS for the given boundary conditions. A typical 30-region case takes about 12 seconds running time of which only about 0.2 to 0.3 second is used for the collision probability and T-matrix calculations.

The advantage of incorporating THESEUS as a subroutine of THERMOS looks quite promising. If THESEUS was used as an additional collision probability subroutine that could be called at the user's option, a significant savings in running time

for the THERMOS cases in which a THESEUS collision probability calculation is adequate may well be possible. An estimated 70 to 80% savings might be realized for a 30-energy, 30-region problem.

Mission 7 - Increased Reactor Irradiation Space

Ni-U Bonding

(W. E. Gurwell, Fabrication Development Unit)

The formation of the intermetallic compositions NiAl_3 , Ni_2Al_3 , Ni_5U and NiU_6 by arc melting followed by annealing or forging was previously described. X-ray diffraction results were received this month. The compounds NiU_6 and Ni_2Al_3 were found to be relatively pure. The forged NiAl_3 compound showed one moderately strong line not associated with its orthorhombic cell. The patterns from both the annealed and the forged buttons of Ni_5U are completely unidentifiable. The annealed Ni_5U button was probably contaminated during annealing. One more attempt will be made to obtain an identifiable pattern from the forged Ni_5U button by using annealed filings.

Laboratory work conducted concerning Al-Ni-U bonding will be summarized in BNWL-CC-1328. The report will include the above work as well as electron microprobe work on a thickened hot die sized bond and the fabrication of duplex aluminum tubing.

Aluminum Alloys

(J. C. Tverberg, Primary Fabrication Unit)

A fine distribution of second phase particles in the Al-Fe-Ni alloys, under evaluation for resistance to ledge and groove corrosion, is essential if corrosion resistance is to be obtained. Normal casting methods result in large second phase particles that tend to accumulate along the grain boundaries. In order to overcome this problem, a direct chill casting apparatus was built and is in the process of undergoing process evaluation. In this device the molten aluminum is poured at 700°C into a pour basin that meters the flow of aluminum

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into the chill section. The aluminum freezes into a thin shell along the jacket, which is cooled by a water spray while it is being withdrawn into a tank of water. This allows the second phase structure to be quenched into the matrix in a uniform distribution. The apparatus has undergone extensive redesign from that of the original unit. A water actuated cylinder has replaced the air operated withdrawal shaft. This provides smoother operation of the withdrawal and a more positive starting action. Both the chill wall and the starting plug have been redesigned and will be made of copper. Using the original aluminum mold and steel starting plug a thin shell of aluminum ten inches long was made before the molten aluminum melted through; however, the procedure could not be repeated. One of the unsuccessful castings was examined metallographically and a fine second phase distribution was observed next to the chill. This indicates that the direct chill apparatus will be successful once the operating procedures are established.

Fuel Coating Corrosion

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

Sample fuel elements were discharged August 17 from an out-of-reactor test loop (TF-18) for examination. The loop operates on once-through reactor process water at 130°C and 35 gpm. Three nickel plated samples showed no evidence of corrosion after two months' exposure in the Zr-2 process tube. Three nickel plated fuel elements with defected plates from both the aluminum and Zr-2 process tubes showed no corrosion at the defect after 30 days' exposure. Sample fuel elements with a calcium aluminum oxide coating were completely devoid of special coating after three and one-half months' exposure. A dull gray surface with uniform corrosion had resulted.

2.1% Enriched Uranium Separate Target PCTR Experiments

(D. F. Newman, Heavy Moderator Reactor Physics Unit)

Several perturbation measurements were made in the super-cell, consisting of 2.1% enriched uranium fuel cells and 2.93 wt% lithium (42.8% Li⁶) in aluminum target cells in a 3-1 ratio, to determine the reactivity changes due to limited water loss in the target and fuel. Results of these measurements indicate

that water loss in the target alone will decrease reactivity, but that water loss in the fuel alone will slightly increase reactivity. Absolute neutron multiplication factors will not yet be obtained for the perturbation results until the sensitivity to a spectral mismatch is checked by calculation. Non-linear relative differences are readily apparent in measuring the relative masses of copper necessary to compensate for the change in reactivity, ΔM . Dry perturbations in the matched spectrum of the wet lattice appear to be more linear than the wet perturbations in the matched spectrum of the dry lattice.

Results of the perturbation measurements follow:

Mass of Copper Required to Compensate for Coolant Perturbation

Measurements in Matched Spectrum of Wet Lattice

<u>Lattice Condition</u>	<u>ΔM, 5x5 Test Array</u>	<u>ΔM, 6x6 Test Array</u>
Dry Target, Wet U_1 Wet U_2 (Wet buffers)	- 252 g	- 164 g
Dry U_1 , Wet U_2 , Wet Target (Dry buffers)	- 4 g	59 g
Dry U_2 , Wet U_1 , Wet Target (Wet buffers)	18 g	85 g

Measurements in Matched Spectrum of Dry Lattice

Wet Target, Dry U_1 , Dry U_2 (Dry buffers)	201 g	192 g
Wet U_1 , Dry U_2 , Dry Target (Dry buffers)	34 g	62 g
Wet U_2 , Dry U_1 , Dry Target (Dry buffers)	- 6 g	20 g

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A blocking effect due to neutrons incident on the target coolant water being reflected away from the target material is indicated by the above results. The change in reactivity, Δk , due to loss of coolant water in the target cell alone is estimated to be 12 ± 3 mk.

2.1% Enriched Uranium Separate Target Calculations

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

Infinite medium multiplication constants have been calculated for graphite moderated 3-1 supercells in a 6x6 array of cells and compared to PCTR experimental values. Each supercell contains three cells of 2.1 wt% enriched uranium fuel and one target cell of 2.93 wt% lithium (^{6}Li) in aluminum at a 7.5 inch square pitch; k_{∞} values for both the complete 6x6 array and the central 2x2 supercell were calculated using the 2-group diffusion theory code WHIRLAWAY in essentially 2-dimensional geometry. Cross sections were obtained from TEMPEST, THERMOS, and HRG. In the first model the outermost 20 cells (target and driver cells alike) of the array were considered completely homogeneous while the central 4x4 cells were allowed to have some heterogeneity--two regions, a graphite region and a region containing the driver or target element, coolant, etc.

The k_{∞} values of 0.9699 and 0.9943 were obtained, respectively, for the whole reflected 6x6 array and the central 2x2 supercell as compared to a measured value of 1.0035. The calculated infinite multiplication constant of the central supercell was determined by taking the ratio of total production to total absorption. The difference in the overall k_{∞} and the central supercell k_{∞} was found to be caused by the outer 20 homogeneous cells which had a k_{∞} of 0.8993. This discrepancy was reduced, but not completely eliminated, by replacing the homogeneous outer cells by cells identical to the inner ones in the model. With this model the values of 0.9995 and 0.9975 were obtained for the infinite multiplication constants, respectively, of the whole array and of the central supercell. These values are in better agreement with the measured value.

Additional 3-dimensional calculations are being performed to investigate the effect on the multiplication of removing different lengths of the central supercell from the 6x6 array.

Separate Large Bismuth Target PCTR Experiment

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

The 5x5 array of bismuth overbore and KVE supercells in an 8-inch graphite lattice was constructed in the PCTR test cavity on June 1 and the experimental measurements have been completed. Measurements in the cells with and without coolant indicate both lattices to be subcritical.

In order to achieve an acceptable condition for a spectral match it was necessary to invert the asymmetrical array of supercells and remove the thermal column shutter. Six Lucite rods were changed in specific driver locations to increase the subcadmium activity along the bottom row of fuel cells. The matched condition was achieved and the specific flux measurements were then obtained.

Relative reaction rate measurements in all cell components of the wet supercell were made with bare and cadmium covered foils in separate irradiations. Normalization between these separate irradiations was done by the use of the PCTR external thermal column. Maximum use of the RIDL-Nuclear Chicago automatic foil counting system was made due to the large number of foils irradiated in such a large supercell. Foil counting data were processed by computer code APDAC-III to correct for variations in foil mass, room background, and decay during counting. Fission foils irradiated in the separable fuel elements were counted and analyzed to obtain values for δ^{28} .

1.25% Enriched Uranium-Thoria Target Supercell

(R. M. Fleischman and A. D. Vaughn, Heavy Moderator Reactor Physics Unit)

The infinite medium neutron multiplication constant, k_{∞} , for a 3-1 fuel-to-target ratio supercell of 1.392 in. OD (a

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reduced fuel diameter from the original experiment) 1.25% enriched uranium and thoria target was determined for the wet condition (water coolant in process tubes) and for the dry condition (no coolant water). Values for k_{∞} are 1.04183 ± 0.00546 and 1.08024 ± 0.00524 for the wet and dry cases, respectively.

Mission 8 - Nuclear Safety

Reactor Effluent Disposal

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

The 1301-N crib received increased concentrations of tritium during the latter part of July which should be detectable in wells and springs in the area. A sampling program was started to intercept the pulse at several wells and springs. Results should augment previously determined waste travel times and provide more exact information on the distribution of waste as a function of time, path and distance at this particular site.

Mission 10 - Columbia River Studies

Water Treatment Variables

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

The effectiveness of activated silica as a coagulant aid was evaluated under three different conditions of water treatment. Use of these waters as reactor coolant in no instance provided significantly beneficial effects in the concentrations of effluent radioisotopes. It is thus felt that further experimentation with the use of activated silica for this purpose is unwarranted.

Completed data verified a nearly universal increase in effluent radionuclide concentrations during the period that 1 ppm SiO_2 was applied to water that had been pretreated at pH 6.6 and -5 mv zeta potential with sulfuric acid and alum. Although no measurements were made, it is reasonably certain that decreased efficiency of parent isotope removal resulted

from this treatment which in turn contributed to the additional radioisotope production.

Water treatment conditions were altered to provide neutral zeta potential at pH 6.6, and 1 ppm of activated SiO_2 , aged for only 1.7% of gel time, was applied to the water. Effluent activity values decreased to the approximate levels that were previously obtained with the same concentration of activated silica that had been aged for approximately 25% of the gel time. Some marginal benefits in effluent activity were obtained but were not of sufficient magnitude for the treatment to be considered for process application.

These various treatments were those considered to be most efficient on the basis of laboratory experimentation. Therefore, no further evaluation of activated silica is anticipated.

Deionized Water Facility

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

Data were collected for a full fuel cycle to determine the steady state concentrations of effluent radioisotopes from two tubes supplied with deionized water, since the conversion to the stainless steel transport system. As was previously postulated, the concentrations of the various nuclides were reduced from those obtained through use of the original carbon steel system, verifying the presence of a reservoir of impurities contained by the carbon steel corrosion product.

With the establishment of steady state conditions in this system, a program will soon be initiated in which the deionized water will be reconstituted to process water by suitable additions of various salts. It is anticipated that the effects of specific ions on the film deposition and retention process can be evaluated by their exclusion from the additives to the deionized water. Due to the solubilities of the salts involved, the constitution of process water will not be duplicated; instead, a compromise has been developed in which the sodium or nitrate salts of the various constituents will be employed. The conductivity and pH of the solution will be maintained at specified values.

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Process Technology

Be-Zr Braze Alloy Process Development

(R. G. Nelson, Primary Fabrication Unit)

A study has begun with the objective of producing high purity outer tube braze rings. This will be accomplished by starting with high purity elemental metals (Zr, Be, Sn, Fe, Ni, Cr) and consolidating and casting these materials using the vacuum-consumable arc process.

Nine primary ingots were melted and currently are being threaded for assembly into the second melt electrode. The machined primary ingots are 2 11/16 in. diam by 9 in. long. Melting was performed using 31 to 26 volts 1800 amperes at an absolute pressure of 5 to 15 microns during melting. Furnace pressure before and after melting was 0.5 micron. The rate of rise before and after melting was 0.2 to 0.6 micron per minute over a five minute test. This test is made with the furnace isolated from the pumping system. The low rate of rise indicates no system leaks and is insurance against contamination of the metal during melting. Arc power on time for each ingot was about five minutes. Attempts to electron beam weld the primary ingots together failed due to circumferential weld bead cracking, thus necessitating a threaded joint.

A test mold designed to cast a 24 in. long tube of outer ring geometry is being built. Initial casting should take place about August 30-31.

Single Pass Decontamination Studies

(A. P. Larrick, Water Reactor Corrosion and Chemistry Unit)

Laboratory corrosion tests to determine the corrosiveness of the inhibited phosphoric acid full-reactor single-pass decontamination process planned for the end of September have continued. Stress corrosion cracking did not occur on stressed U-bend samples of 17-4 PH stainless steel in the 900-H, 1025-H and 1100-H conditions, 416 stainless steel, or 347 stainless steel after seven hours exposure to 10% inhibited phosphoric acid at 71°C. Silver solder on samples of A212CS soldered to

347SS exposed at the same conditions was not attacked but small pits to 1 mil deep were observed on the carbon steel. Carbon steel exposed to concentrated, inhibited phosphoric acid at 93°C did not exhibit localized corrosion after one-half hour (the anticipated time that certain components will be exposed at these conditions during the reactor decontamination) but pits 1 mil deep were observed after eight hours on both plain steel samples and steel samples welded to stainless steel. Localized attack was not observed after twenty minutes in 93°C, 10% acid nor after twenty minutes in 76°C, 20% acid, both possible off-standard conditions. Localized attack was not observed in room temperature, 4% acid after 856 hours exposure--conditions typical of those expected in the decontamination waste storage tank where the spent solutions will be retained. Maximum corrosion during the above tests was 1.33 mils and occurred on carbon steel/stainless steel weld samples in the 93°C, concentrated phosphoric acid test. No corrosion occurred on Zr-2 samples exposed during the twenty minute tests.

Corrosion was also monitored during the 14-tube single-pass inhibited phosphoric acid demonstration decontamination conducted in N-Reactor the end of June. Samples were exposed twenty minutes to 10% acid at approximately 158°F. Samples exposed were A245 carbon steel crevice and noncrevice, A212 carbon steel welded to 304 stainless steel, AMS 5616, Zr-2 (etched, autoclaved, and crevice samples) and Zr-2 coupled to carbon steel. Corrosion penetrations calculated from weight loss data were low (0.062 mil maximum) but more pitting occurred than was predicted from the laboratory tests. Most of the carbon steel crevice samples had pits 1/2 to 1 mil deep by 1 to 3 mils diameter in the crevices and the CS-SS weld samples had pits to 20 mils long, 1 to 2 mils wide and 1 to 2 mils deep on the carbon steel side of the weld. In addition, a 1/8 in. wide row of smooth bottom pits 2 to 3 mils deep was found along the entire length of the samples; this pitting appeared to occur at a liquid-air interface when the pressure tube was partially full during some stage of the decontamination.

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Half-Plant Dichromate Test

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

A plant test employing 1/2 ppm of dichromate in the coolant to one-half of B Reactor and 1 ppm to the other half of the reactor is being checked for corrosion effects by electrically measured probes in six rear nozzles, samples installed in spacers, and several dummy fuel elements placed out of flux in the number 9 position. Two of the dummy fuel elements were replaced after about 24 days of exposure and the removed specimens examined. No evidence of ledge or groove corrosion was apparent.

Corrosion of 8001 and 1100 aluminum alloy as estimated by the electrically measured probes indicated rates between 0.08 and 0.2 mil/month. The 1/2 ppm dichromate portion of the test showed slightly higher rates than the 1 ppm side.

Fuel Cladding Corrosion

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

A test employing samples of 8001 and 1100 aluminum alloys exposed to reactor process water at various temperatures and flow rates with externally applied potentials has been started. Two seven day runs have been completed, employing potentials of 0, 0.05 and 1.0 volt, temperatures of 100 and 130°C, and flow rates of 25 and 50 ft/sec. In the test at 100°C and 25 ft/sec, the anodic samples which had been autoclaved before test severely pitted at 0.5 and 1.0 volt. Only the 1.0 volt anodic sample pitted when tested in the etched condition. The test at 130°C did not show any localized corrosion.

Low Temperature Hydriding of Zircaloy

(B. Griggs, Water Reactor Corrosion and Chemistry Unit)

Examination of the downstream fifteen feet of process tubes 2577-KE-2 and 2571-KE-2 for hydriding is in progress. These tubes contained low resistance anodized spacers and spacers with a "Sanford Hard Coat," respectively, for six months' exposure. Analyses of rings from the downstream five feet of

2577-KE show a low level of hydrogen starting at 12 ppm at six inches and decreasing to 8 ppm at sixty inches. Even though these low levels may be inherent in the as-installed tube, the uniformity of the slope of hydride concentration versus distance is similar to severely hydrided tubes. The possibility of isolated hydride particles is being investigated metallographically. Tube 2571-KE showed a scatter of hydrogen content in the rear five feet from 8 to 10 ppm. Analyses of the five to fifteen foot region are in progress.

Measurements of potentials and current flows between Zircaloy-2 and 6063 aluminum in reactor process water at temperatures up to 160°C were made. The potentials generally were lower at the higher temperature but fell in the range of 400 to 600 millivolts. Current density increased with temperature to about 40 microamps/cm². In a ten day run at 120°C, current density started at 40 microamps/cm² and held in a range of 2 to 4 microamps/cm² for the rest of the run. Potentials of about 400 mv were constant throughout the run. Hydride measurements of the Zircaloy-2 cathode are in progress.

Water Filtration Mechanisms

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

Twenty polyelectrolytes selected previously by coagulation tests are under evaluation as filter bed conditioners. Each is being compared with Magnifloc 990 for headloss, effluent turbidity, bed penetration, and run length on two laboratory filters. Polyelectrolyte concentrations of 0.03 ppm are being added to alum coagulated water just prior to filtration. Filtration is being carried out at 8 gpm/ft². Three filter runs are being made with each polyelectrolyte. Magnifloc 990 is used for three filter runs between the use of each of the twenty polyelectrolytes.

Analytical Instrumentation for N-Reactor

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

The recorder for the Parametrics hygrometer was installed. Additional work is required on the switching box for satisfactory operation. The Greenbriar chromatograph will not be ready

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for inspection until approximately September 1.

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

Developmental projects centering on personnel, miniature dose rate and dose monitoring devices, both with and without meter indication and alarm signalling features, are being actively investigated. Specific effort is being devoted to the development of solid state detectors (diodes) for gamma detection for incorporation into the planned miniature instruments. Liaison effort is now in progress to aid in determining the exact requirements and preferred operation characteristics.

ASSISTANCE TO ISOCEM

Summary

Mission 1

Continued study of means of catalyzing the nitrous-nitric acid oxidation of neptunium(V) has shown that the yield of the rate-accelerating material obtained on acidification of the aci-form of 1-nitropropane is increased as the nitric acid concentration is increased.

A method for the determination of low concentrations of alcohols in Purex diluent is being developed.

Mole for mole, Zr(IV) ion is markedly more effective than Al(III) for suppressing corrosion of 304-L stainless steel by HNO_3 -HF solutions.

Nitric acid-sulfuric acid systems show promise as a rapid test for susceptibility of 300 series stainless steels to intergranular attack.

Interfacial tension measurements of the UNH-hydrocarbon diluent-TBP- H_2O system were made at various temperatures.

Excellent gamma and X-ray spectral measurements were obtained on eight samples from the Waste Reclamation Facility using a cooled, lithium-drifted (SiLi) diode detector and associated electronics instrumentation.

Mission 3

Several scouting runs were made in the 324 Building pulse column facility to demonstrate the PAMEX process for separating americium from the rare earths. "Cold" rare earth behavior was followed by Ce^{144} tracer and americium by Am^{241} tracer. The results were generally discouraging. Typically, the americium split almost equally between the aqueous and organic raffinate streams in the stripping column with either DTPA or

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NTA used as the americium complexant. Since the distribution ratio was favorable for americium to strip completely, the failure to do so has tentatively been attributed to poor kinetics.

Mission 5

During the extraction of polonium from irradiated bismuth by fused NaOH, antimony and zinc transferred nearly quantitatively to the NaOH while silver remained in the bismuth.

Mission 6

Preparations for the laboratory use of fluorine are nearly complete.

Mission 8

Eight experiments were completed to develop and check apparatus and procedures for measuring plutonium releases from slow evaporation processes and for preparing specimens of plutonium nitrate for higher temperature releases. Uranyl nitrate hexahydrate was used as a stand-in in these initial experiments. The quantity of uranium released and carried in the airstream away from the containment vessel was less than 10^{-4} wt%, the detection limit.

The study to characterize particles carried in the A-Tank Farm vapor vent line and the CR vault exhaust was completed. Particles carrying radioactive material in the CR vault exhaust air were much larger than anticipated.

Neutron decay constants were measured for far subcritical systems of slightly enriched uranyl nitrate as a function of geometric buckling. The measurements provide further insight into the feasibility of using pulsed neutron source experiments on in-plant criticality assessments.

Data from previous experiments performed with PuO_2 -polystyrene compacts having an H/Pu ratio of 5 and containing 11.5 wt% Pu^{240} were analyzed yielding values for the following:

critical or material buckling, critical thickness of infinite slab reflected with Lucite, and the extrapolation length for an infinite slab reflected with Lucite.

Mission 9

Electrolytic oxidation of cerium(III) to (IV) was found to proceed well in zirconium and titanium equipment.

Shielding calculations were made using recently published mass absorption coefficients.

Instrumentation improvements were made in the flame velocity measurement apparatus to prevent spurious signals to the probes.

Periodic washing of the D2EHPA-TBP-NPH B-Plant solvent with dilute sodium carbonate solution to remove radiation-produced M2EHPA appears to be an adequate way to maintain strontium extraction capacity.

Process Technology

The last batch of Pu^{238} product was reprocessed to remove contaminants.

Alpha radiolysis experiments on plutonium(IV)-nitrate solutions yielded preliminary "G" values for total gaseous products.

The second and final resin pump modification resulted in a lowered resin attrition rate of approximately 2.8 ml/hr. The attrition rate with the original pump was about 65 ml/hr and about 3.8 ml/hr with the first modification. The final modification also had better pumping characteristics than either of the first two configurations.

A computer program to describe the expanding sphere model of hot spot formation was completed.

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ASSISTANCE TO ISOCEM

Mission 1 - Basic Production Mission

Neptunium Chemistry

(J. L. Swanson, Chemical Separations Unit)

The yield of the rate-accelerating material obtained when HNO_3 is added to a basic solution containing the aci-form of 1-nitropropane and NaNO_2 was found to increase as the HNO_3 concentration is increased. In one set of experiments, the yield was ten times greater when 15.7M HNO_3 was added (giving 11M HNO_3 at 40°C) than when 6.3M HNO_3 was added (giving 4.5M HNO_3 at 28°C).

In these experiments, 12.5 ml of HNO_3 solution was added to 5 ml of 0.1M NaOH + 0.1M 1-nitropropane + 0.05M NaNO_2 and the mixture was then diluted to 25 ml. Samples were adjusted to 3.1M HNO_3 , NaNO_2 and Np(V) were added and the rates of Np(V) oxidation were determined, allowing comparison of the extent of conversion of the nitropropane to the rate accelerating material.

Rapid disappearance of the rate-accelerating material was observed in 8M HNO_3 . In 3M HNO_3 , however, little or no destruction occurred in several hours.

Purex Diluent Studies

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

As a result of discussions with Isochem personnel on normal paraffin hydrocarbon specifications, it was determined that a test for high molecular weight alcohols at the 0.05% concentration level was necessary. Since it is difficult to detect alcohol below the 0.2% level by infrared spectrophotometry, a procedure was devised to concentrate the alcohol fraction for infrared analysis. Initial checks of the method with dodecanol and decanol spikes in cleaned NPH have shown complete recovery at the 0.05% level.

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The procedure involves absorbing the alcohol from a 100 ml sample of NPH on a column of silica gel (washed with methanol and oven dried at 120°C), washing the column with silica-gel-cleaned cyclohexane, eluting the alcohol with methanol or other volatile polar solvent, evaporating the eluate under vacuum, and mixing the residue with 2 ml of the cleaned NPH. Prior to infrared analysis, the mixture is checked for complete removal of the wash and elution solvents by gas chromatography.

Corrosion of 304-L Stainless Steel by HNO_3 -HF-ZrO(NO₃)₂ Solutions
(R. F. Maness, Materials and Process Chemistry Unit)

The corrosion resistance of 304L stainless steel was determined in boiling HNO_3 (2 to 10M)-HF(0.01 to 0.5M)-ZrO(NO₃)₂ solutions at Zr/F mole ratios varying from 0.5 to 2. Results to date indicate that, mole for mole, corrosion is about one-tenth as great when Zr(IV) rather than Al(III) is used as the fluoride complexing agent. A synthetic Purex 1WW solution, simulating that produced during Zirflex processing (0.33M F) and containing 0.5M ZrO(NO₃)₂, corroded 304L stainless steel at a rate of about two mils/month. This is about half the rate obtained when 1.0M Al(NO₃)₃ was used as the fluoride complexing agent.

Test for Susceptibility of 304L Stainless Steel to Intergranular Corrosion

(R. F. Maness, Materials and Process Chemistry Unit)

Sensitized and as-received 304L specimens were exposed to $\text{H}_2\text{SO}_4 + \text{HNO}_3$, $\text{H}_2\text{SO}_4 + \text{CrO}_3$, and $\text{H}_2\text{SO}_4 + \text{HNO}_3 + \text{CrO}_3$ systems. Preliminary results indicated that a single 24-hour exposure to the $\text{H}_2\text{SO}_4 + \text{HNO}_3$ system will suffice to determine the degree of susceptibility to intergranular attack.

Fundamental Mass Transfer Studies

(P. C. Owzarski, Engineering Analysis Unit)

Alterations to the equipment for single drop extraction were made. The direct current variable-speed motor has been a source of "down time" problems. Installing it outside the hood on the floor has finally resulted in a vibration-free drop.

A drop-weight apparatus for measuring liquid-liquid interfacial tension has been set up. Values for the UNH-hydrocarbon diluent-TBP-H₂O system are being measured as functions of UNH concentration in an equilibrium system at various temperatures. This will aid in an analysis of driving forces in interfacial turbulence. A suspected turbulence driving force in transfer of UNH from organic to aqueous phase is its density gradient at the interface which causes convective instability.

Electrodeless Conductivity

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

An electrodeless conductivity system is required for service as an HAW acidity monitor. To fulfill this requirement a commercial system is being evaluated and, in addition, a laboratory conceived system is being developed.

The commercial system has been characterized with respect to accuracy from 23 to 70°C at nitric acid concentrations from 0.5 to 3M. The accuracy is about 7% at 0.5M; 1% at 1M; 3% at 1.6M; 2% at 2M; and 5% at 2.6M HNO₃. The source of error appears to be caused by oscillator stability rather than by the temperature compensating device which attenuates the oscillator output. The application of the commercial system is restricted to specified cable type and length and therefore may present problems in plant installation.

The laboratory system is being developed to circumvent problems associated with the commercial system, in particular, ease of installation and calibration, lower initial cost and reduced size seem attainable with the laboratory device. The design of the driven and output toroids, pulsing system, cabling variations and signal conversion system have been under study. Operation with cable lengths to 100 ft have been shown to have only a slight effect on signal output. In addition, operation to solution loop resistances in excess of 500 ohms or the equivalent of operation down to acid concentrations of 0.001M appear to be easily achieved.

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Canned Motor Pump Evaluation

(J. Dunn, Process Systems Development Unit)

The Hayward-Tyler canned motor pump being tested as candidate replacement unit for Chempumps failed because of excessive bearing wear. The pile grade graphite (CS6BF) bearings were worn and scored up to 30 mils on the diameter and up to 60 mils on the lower thrust face during the 936 hour test run on water at 32 gal/min and 50 psig. New bearings were installed and the unit was subjected to additional testing. An evaluation report is being drafted.

Zirflex Dissolver Studies

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

A full-scale test was made using 7.5M NH_4F boiling solution for one hour followed by seven hours with 5.5M NH_4F . This alternative to straight 5.5M NH_4F zirflex decladding showed no perceptible improvement. Further tests are planned to evaluate 7.5M NH_4F solution for the full eight-hour decladding period.

Effect of DBBP Contamination on 1C Column Performance

(G. L. Richardson, Engineering Development Unit)

The use of dibutylbutyl phosphonate (DBBP) in the Purex Plant to recover neptunium and plutonium from high-level waste is being actively considered. The consequences of accidentally mixing this solvent, which has a high affinity for uranium, with TBP were explored briefly in the pilot plant 1C pulse column. The solvent in these studies contained 24% TBP and 5% DBBP in NPH diluent.

The uranium loss in the 1C column was increased from less than 0.002% to about 0.1% when operating at 60°C and an aqueous-to-organic flow ratio of 1.04. The loss was reduced to 0.025% by increasing the flow ratio to 1.17. The maximum stable operating frequency at the test conditions (volume velocity about 1300 gal/hr-ft², sum of flows) was also lowered from 85 to 76 cycles/min, a relatively minor effect.

Separations Instrumentation

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

The developed gamma and X-ray spectral measurement instrumentation, which employs both solid state circuitry and a Si(Li) detector, was used to measure the spectra of eight samples received from the Waste Reclamation Facility. Low energy spectra, measured with the liquid nitrogen cooled, lithium-drifted silicon detector that has a 1.0 keV resolution (FWHM) at 17 keV, demonstrated strong, well-resolved photopeaks at energies of 13.95, 17.7, 20.9, 26.4 and 59.6 keV in all eight samples. Standard samples of Am²⁴¹ and Pu²³⁹ are being prepared to use as reference information in evaluating the data.

Additional measurements, being made with the complementary solid state germanium detector system, will provide information relative to higher energy spectra (0-600 keV) of these samples. The information ultimately provided by these investigations should lead to operational instrumentation that will significantly enhance the ability to measure low level concentrations of Pu²³⁹ and Am²⁴¹.

The planned alpha counting and gamma spectrometry investigations are temporarily delayed pending installation of specific in-line sample cells. In these systems, the intensity of the alpha or gamma emissions is used to measure the Pu²³⁹ concentration in the CCW process line. Because of the gamma background problem at the hood location, both alpha and gamma detection instrumentation will be evaluated to determine the better approach for final application. Tentatively, the alpha counting detector will be a cerium-activated glass scintillator with an attached multiplier phototube. The gamma detector incorporates one in. diameter Na(Tl) crystals of various thicknesses from 0.25 in. to one in. in an effort to determine the optimum size. When completed, the system will be employed to measure Pu²³⁹ concentrations over a range of 10⁻² to 5 grams per liter.

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Mission 3 - Transplutonium

Pilot Plant Demonstration of the PAMEX Process
(G. L. Richardson, Engineering Development Unit)

A solvent extraction process for separating americium from cerium and most of the other rare earths was described. The process involves co-extraction of americium and rare earths into a di(2-ethylhexyl)phosphoric acid (D2EHPA) solvent from a complexed feed, followed by selective stripping of the americium from the rare earths by a carefully-buffered complexing solution. The process has worked well in laboratory batch extraction studies but attempts to perform the separation at the Strontium Semiworks pulse column facility were generally unsuccessful.

Several scouting runs were made in the 324 Building pulse column facility to test various flowsheet concepts for use in the SSW to purify an americium crude obtained during the recent Shippingport fuel processing runs made in the Redox plant. The pulse column geometries simulated those of the SSW and feeds were prepared from SSW reagents. "Cold" rare earth behavior was followed by Ce^{144} tracer and americium by Am^{241} tracer.

The results were generally discouraging. Typically, the americium split almost equally between the aqueous and organic raffinate streams in the stripping column with either DTPA (diethylenetriaminepentaacetic acid) or NTA (nitrilotriacetic acid) used as the americium complexant. Since the distribution ratio was favorable for americium to strip completely, the failure to do so has tentatively been attributed to poor kinetics. The performance was improved only slightly by increasing the operating temperature from the ambient 30 to 50°C. Cerium decontamination was generally adequate; in most runs, less than 1% of the cerium was found in the americium product.

Mission 5 - Other Isotopes

Corrosion of 304L Stainless Steel by Thorium Nitrate Solutions
(R. F. Maness, Materials and Process Chemistry Unit)

The corrosion resistance of 304L stainless steel was determined in non-fluoride-bearing $\text{Th}(\text{NO}_3)_4$ solutions. The objective was to determine the feasibility of concentrating, in stainless steel equipment, acid (ca. 2M HNO_3) solutions containing about 1.5M $\text{Th}(\text{NO}_3)_2$ to 4M $\text{Th}(\text{NO}_3)_4$. Initial, mid-point, and terminal solutions at the boiling point corroded 304L at rates of about 0.06, 0.1, and 12 mils/mo., respectively. Exposure periods were 150 to 200 hours. Coupons of 304L exposed to $\text{Th}(\text{NO}_3)_3$ solution during concentration from 1.5 to 4.0M $\text{Th}(\text{NO}_3)_3$ (90 exposure hours) corroded at an average rate of about one mil/month. Attack in all cases was intergranular.

Extraction of Polonium from Molten Bismuth with Fused Sodium Hydroxide
(W. W. Schulz, Materials and Process Chemistry Unit)

A statistically designed experiment was completed with the present batch equipment to determine the effects and possible interactions of the three main process variables--contact time, contact temperature, and weight ratio of NaOH to bismuth. Mathematical analysis of the data from these experiments is only partially complete. Indications are, however, that over the range of variables tested (30, 45 or 60 min. contact at 400, 500, or 600°C at NaOH/bismuth ratios of 1, 2 and 3) only contact time significantly affects removal of Po^{210} from the bismuth phase. In most of these experiments 99+% of the Po^{210} was removed from the bismuth phase, demonstrating again the feasibility of this technique for separating Po^{210} from metallic bismuth.

The experiments just referred to, as well as all previous fusion experiments, were performed with bismuth metal containing only about 3 to 6 mCi Po^{210} /gram bismuth. The feasibility of the separation scheme at much higher Po^{210} concentrations was demonstrated this month with a freshly irradiated bismuth slug. In this latter experiment, a 2.0 gram sample of bismuth

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containing 90 mCi Po^{210} /gram bismuth was contacted 45 min at 500°C in a graphite boat with 4 grams of NaOH. Over 99.8% of the Po^{210} transferred to the NaOH phase, a result in excellent agreement with that obtained at lower Po^{210} concentration.

The bismuth and NaOH phases obtained in the experiment just referred to were gamma energy analyzed to learn something of the decontamination potential of the NaOH fusion process. Of the identifiable gamma emitters, 99+% of the Sb^{124} and Zn^{65} transferred to the NaOH phase while essentially all the Ag^{110} remained in the bismuth phase. Some Co^{60} also appeared to be present in the bismuth fraction.

The excellent decontamination realized for antimony and zinc is very encouraging. Recent calculations indicate the desirability of maintaining very low concentrations of these two impurities as well as of silver, cobalt and tantalum in the recycled bismuth to avoid subsequent accumulation of neutron activation products which complicate handling the bismuth metal. In this connection also, on the basis of known chemistry, tantalum, if present, might be expected to report to the NaOH phase.

Extraction of Polonium from Bismuth by NaOH
(J. Dunn, Process Systems Development)

A continuous dissolver for processing 1.4 lb/hr of polonium-containing NaOH was designed in support of a study concerning polonium processing at Z-Plant. Information indicated that the extraction of polonium from bismuth by molten caustic may be "kinetics limited." The percentage extraction per contact is high--in the order of 99+% but the residence time is also high--about 1/2 hour. If this is the case, column extraction may not be feasible and a different contactor will be required.

Contact by dispersion and agglomeration of mercury (as a stand-in for molten bismuth) in water was demonstrated in a 1 inch diameter x 36 in. long column packed with ceramic Intralox paddles.

Mission 6 - Enriched Fuel Processing

Fluoride Volatility

(W. H. Yunker, Chemical Separations Unit)

Construction of the fluorine handling facility and transfer line was completed. A few small changes, a complete operational test and installation of metering equipment need to be made before fluorine will be available for laboratory use. It should be available in September.

Mission 8 - Nuclear Safety

Atmospheric Dispersion of Radioactive Particles

(J. Mishima, Particulate and Gaseous Waste Research)

A series of experiments is being performed to evaluate the release rate of plutonium from plutonium nitrate during evaporation of solutions and subsequent heating of the dried residue. Initial experiments have so far utilized uranyl nitrate hexahydrate (UNH) as a stand-in for plutonium nitrate to permit development of equipment and procedures which will insure containment of the plutonium. Since the evaporation phase of these experiments will be performed in a hooded enclosure, rather than a gloved box, extra precautions are being exercised. The dried residue will be heated in a gloved box to measure release rates. A second incentive for working with uranium in initial experiments is to determine its similarities to and differences from the behavior of plutonium.

The final four UNH runs were completed using an apparatus which allows air to sweep across an evaporating solution held in a shallow stainless steel cup. Uranium in the containment vessel, the condenser, and on a downstream filter is determined. A low air velocity of 10 cm/sec was used, with evaporation proceeding at 22 and 50°C. The results of these experiments are shown in the table following.

The fraction released under the last four run conditions was lower than for the first four experiments in which 100 cm/sec air was used. No detectable amounts of uranium reached

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UNH RUNS IN SLOW-DRYING APPARATUS

(3 ml of 291.986 grams uranium per liter solution)

Run No.	Temp, °C	Air	Duration, min	Air Velocity, cm/sec		Weight Percent Uranium Found in			
						Containment Vessel Wash	Condenser Wash	Filter Drying	Filter Cooling
U-1	95	Room	67	100	10 ⁻⁴ wt.% in condensate particle generation only	0.013	0.0008	0.0002	
U-2	50	Dry	225			0.0028	0.0001	0.0002	
U-3	220	Dry	75			--	--	--	--
U-4	RT	Dry	18 hr			0.0097	<10 ⁻⁴	<10 ⁻⁴	
U-5	90	Dry	120	10	~ 10 ⁻⁴ wt.% in 3cc condensate	0.0028	0.0008	<10 ⁻⁴	<10 ⁻⁴
U-6	50	Dry	240			0.0009	<10 ⁻⁴	*	<10 ⁻⁴
U-7	50	Room	225			0.0056	<10 ⁻⁴	<10 ⁻⁴	<10 ⁻⁴
U-8	50	Dry	225			0.0003	<10 ⁻⁴	<10 ⁻⁴	<10 ⁻⁴
U-9	RT	Dry	14 5 hr			<10 ⁻⁴	<10 ⁻⁴	<10 ⁻⁴	<10 ⁻⁴

* Lost in analysis.

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the filter or the condenser. Very small amounts of uranium were found in the containment vessel for other than room temperature, presumably from small droplets breaking at the surface. The 100 cm/sec air evaporation entrained a measurable amount of uranium.

Characteristics of Aerosols in Gaseous Effluents

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

Study of the airborne particles in effluent from the A-Tank Farm vent system was completed. The sampling and characterization of particles carried in the hot saturated air stream posed special problems in sampling. The removal of samples required near isothermal conditions in the sampling probe and throughout the collectors used. As earlier indicated, this was accomplished by placing all external parts of the sampling system in a heated, insulated shroud, effectively an oven, which was kept at near the effluent gas temperature. Since an exact duplication of gas temperature could not be assured, samples were taken at shroud temperatures of 91.5, 97.5, and 106°C. The indicated particle size distribution did not vary significantly with temperature. It is thus believed that the sampling method did not materially alter the particle sizes by either evaporation or condensation. The mass median diameter indicated by these samples was approximately 2 μ .

The particle loading was estimated from measurement of sodium leached from membrane filter samples. A loading of 0.4 grain/1000 ft³ was calculated on the assumption that the particles consisted of 8N NaNO₃ solution.

In a related study, aerosol samples were taken from the CR vault ventilation discharge at a point just upstream from a filter bank recently installed. The radioactive material present was found to be associated with particles whose mass median diameter was estimated to be about 12 μ , a somewhat unexpected result because of the relatively large particle size indicated. Visual examination of impactor stages and the membrane filter sample showed some particles to be present as

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large as 200 μ . One of these selected at random was found to read 5000 c/m on a VGM. These results suggest that large particles comprise the bulk of the mass loading and that the radioactive material present is associated with larger particles.

The particle loading proved to be too low to be measured by the gravimetric method used. For a four hour sampling period and a flow rate of 2 cfm, the total mass collected was less than 1 mg. This would correspond to a loading of < 0.003 grain/ft³.

The sampling arrangement which could be installed within the time, space, and radiation limitations was far from ideal, especially for large particles. The results are consequently indicative rather than definitive yet do give some insight into the nature of radioactive airborne material in this waste stream.

Pulsed Neutron Source Experiments

(S. R. Bierman, Critical Mass Physics Section)

Pulsed neutron source experiments were performed for determining the prompt neutron decay constant (α) of slightly enriched (2.26 wt% U²³⁵) uranyl nitrate over a range of geometrical bucklings. The H/U ratio (total U) for the slightly enriched UO₂(NO₃)₂ hydrogen-moderated mixture was 11.2. The value of k_{∞} for the material had previously been determined from PCTR experiments to be 1.029. The largest configuration, a 21 in. cube, contained only a small fraction of the critical mass. The decay constant for the 21 in. cube of uranyl nitrate was 3569 sec⁻¹. The decay constant for a 14 in. x 14 in. x 21 in. assembly was 5320 sec⁻¹. The data from measurements on two other intervening sizes have not yet been analyzed. These measurements are preliminary for the purpose of exploring the feasibility of using the pulsed neutron source technique for reactivity determinations pertaining to in-plant criticality assessments.

Criticality Studies of Plutonium Compounds in Undermoderated Range*

(S. R. Bierman, Critical Mass Physics Section)

Data from previous experiments performed with plutonium-plastic mixtures have now been analyzed, yielding values of critical buckling and extrapolation length. The experiments are part of the basic criticality studies for determining the critical dimensions and masses of undermoderated Pu systems and the effect of Pu²⁴⁰ on criticality. The fissile material was in the form of PuO₂- polystyrene compacts wrapped with 7 mil MMM #471 tape for contamination control. The H/Pu ratio was 5 with the Pu²⁴⁰ isotopic content being 11.5 wt%. The assemblies were comprised of Lucite reflected rectangular prisms. The average stack density of Pu over the cores was ~ 2.17 g/cc.

The material or critical buckling was determined from measurements on four different core configurations as $19,600 \times 10^{-6} \text{ cm}^{-2}$. Making use of constant buckling conversions, the extrapolation length for an infinite slab reflected with Lucite was found to be ~ 8.25 cm. The critical thickness for a Lucite reflected infinite slab with the above fuel composition was determined to be ~ 6.5 cm.

Mission 9 - Waste Management

Cerium Oxidation

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

Electrolytic oxidation of cerium was previously shown to work well in glass equipment but not in stainless steel. Experiments completed this month show that use of zirconium or titanium equipment gives results comparable to those obtained with glass. Presumably, zirconium or titanium equipment could be used for plant-scale application of the process.

* Work also funded in part by Division of Reactor Development under Applied and Reactor Physics.

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Other experiments were aimed at exploiting the stainless steel-cerium(IV) reaction for possible application to the dissolving of stainless steel fuels.

Basic Shielding Data

(H. H. Van Tuyl, Fission Product Chemistry Unit)

Calculations of low energy buildup factors for iron and lead were made with recently published mass absorption coefficients. These data and other low energy buildup factor data will be included in a forthcoming report.

Attenuation factors for water, normal concrete, magnetite concrete, iron, lead, and lead glass shields were calculated using the recently published mass absorption coefficients. Calculations were made as a function of photon energy and shield thickness. The calculations were extended to obtain the dose rate at ten feet from the kilocurie point sources of important fission products as a function of shield thickness with each of the above shield materials.

The data have been incorporated insofar as necessary in the recently completed chapter of the Waste Management Technical Manual.

Hydrogen-Oxygen Safety

(W. E. Willingham, Engineering Analysis Unit)

Instrumentation problems still exist in the work.

A glow wire was installed in place of the spark plug to eliminate spurious signals to the probes. Also, new ionization probes were constructed in an effort to obtain good signals to the counters.

Additional help on the instrumentation problem is being brought in in an effort to solve this problem.

Inspection of Underground Waste Tanks by Gamma Scanning
(J. Dunn, Process Systems Development)

A report was prepared describing the development and application of a remotely positioned gamma scanner for detecting concentration of sludge in underground waste storage tanks. The scanner employs a shielded plastic crystal, photomultiplier tube and count rate meters together with a rotatable indexed support tube. Several scans of the 103-A tank were made to measure activity levels in various areas of the tanks. Definitive analysis of the data proved to be difficult; however, it was concluded that no large concentration of gamma emitters remains in the tank following the sludge removal operation by sluicing.

Demonstration of Purex Condensate Recycle
(G. L. Richardson, Engineering Development Unit)

Pilot plant pulse column studies were continued to demonstrate the feasibility of using condensate from the first and second cycle uranium concentrators (1UD and 2UD) as makeup water for the 1CX and 2EX streams in the Purex Plant. Although the visual performance was unaffected by the use of the condensates, the uranium loss in the 2E column increased by more than 10-fold when 2UD was used as 2EX. There appeared to be no adverse effect, however, when either the 2UD or the 1UD was used as 1CX. This anomalous behavior is being investigated further in laboratory studies. The main differences between the 1C and 2E column flowsheets were the higher uranium and lower nitric acid concentrations in the 2E column feed.

B-Plant Evaporator Studies
(W. J. Van Slyke, Engineering Development Unit)

Tests using dilute sulfuric acid produced measured df's (based upon the pH of the condensate) ranging from 150 to 3500, depending upon the boilup rate. A PAW batch evaporation test indicated that good processing rates can be expected up to a concentration factor of approximately eight but will drop sharply at higher concentration factors. Experiments are in

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progress to examine the evaporation characteristics of a typical sludge processing composition.

B-Plant Solvent Quality

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

Previous studies have established that the strontium extraction capacity of a D2EHPA-TBP-NPA solvent decreases by a factor of about two upon irradiation to about 10^8 rads. Recent experiments have shown further that the strontium extraction capacity of these irradiated solvents can be largely restored by washing them with ethylene glycol or dilute NaOH or Na_2CO_3 solutions. Such washes remove M2EHPA (mono(2-ethyhexyl) phosphoric acid), a primary radiolysis product present to the extent of 0.03 to 0.05M in the irradiated extractant.

Direct proof of the adverse effect of M2EHPA on D2EHPA extraction of strontium was obtained. These experiments involved measurement of the strontium and cerium(III) extraction capacity of extractants of the composition 0.29M D2EHPA-0.0 to 0.025M M2EHPA-0.2M TBP-NPH. These extractants were prepared from as-received NPH and highly purified D2EHPA and TBP. The M2EHPA used to prepare these extractants was supplied by the Chemical Research Section who isolated it from irradiated D2EHPA; it contained greater than 95 wt% M2EHPA. In these experiments, the strontium distribution ratio (E_a^{O}) from synthetic IAF solution decreased to about one-half when the M2EHPA concentration was in the range 0.04 to 0.06M. The cerium(III) E_a^{O} initially decreased slightly with increasing M2EHPA concentration, passed through a minimum, and then increased markedly. The latter behavior reflects, apparently, the greater affinity of M2EHPA over D2EHPA for trivalent rare earths as observed earlier at Argonne.

Cyclic irradiation testing of a D2EHPA-TBP-NPH extractant was continued. The current irradiation experiment is designed to evaluate the effects on physical and chemical properties of the extractant of periodic washing with 0.6M Na_2CO_3 solution to remove accumulated M2EHPA and periodic addition of virgin D2EHPA and TBP. In this experiment the extractant, originally

0.31M D2EHPA-0.2M TBP-NPH, is irradiated while in intimate (stirred) contact with a sequence of aqueous solutions simulating a solvent cycle in the current B-Plant flowsheet for processing PAW solution. Irradiation times are chosen to approximate the solvent irradiation dose expected (approximately 13.3 watt/hr/liter/cycle) in plant operation.

The first 15 cycles of the current irradiation test were completed. The solvent was washed with 0.6M Na_2CO_3 after the 3rd, 9th, and 15th cycles; M2EHPA was not detectable after carbonate washing. The D2EHPA concentration of the extractant was adjusted to 0.32M from 0.292M after the 9th cycle; the carbonate washed 15th cycle solvent is 0.317M D2EHPA. The strontium extraction capacity of the carbonate washed solvent after 15 irradiation cycles is about 80% of its original value. Minor changes have occurred in other solvent properties, density, viscosity, and cerium(III) extraction capacity, but all are well within tolerable levels.

It is planned to make a further addition of D2EHPA to the solvent and to continue the cyclic test for another 12 to 15 cycles. Tentatively, it appears, however, that adequate solvent quality can be maintained by periodic removal of M2EHPA and small adjustments in D2EHPA concentration.

Settling Rates of Water-Leached Purex Waste Sludge
(W. W. Schulz, Materials and Process Chemistry Unit)

As part of the overall Hanford Waste Management Program, high pressure water streams will be used to remove sludge stored in the Purex Waste tanks. Subsequently, the undissolved solids will be allowed to settle from the leach liquor and will be further processed to recover associated Sr^{90} . Knowledge of the rate at which the undissolved solids settle is required to establish suitable plant operating time cycles.

Eight-gram portions of sludge from Tanks 101-A and 104-A were each leached 30 min at 80°C with about 16 ml of water. The resulting slurries were held at 80°C for 24 hours and the volume of settled solids noted periodically. The terminal

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settled solids volume was reached after about 5 hours in each case. However, the settled volume of the solids from Tank 101-A was about twice that of the solids from tank 104-A (63 versus 36%). The centrifuged volume percent of both solids was about 23%.

Process Technology

Np-Pu²³⁸ Processing

(T. S. Soine, Chemical Separations Unit)

The plutonium product from the February and March tubes has been reprocessed to remove some cerium contamination and shipments to Isochem have resumed. Total fission product concentration was reduced to 16 micro curies/gram Pu²³⁸.

Radiolysis and Pressure Buildup Over Plutonium Solutions

(J. C. Sheppard, Heavy Element Chemistry Unit)

Initial results of alpha radiolysis experiments with 0.47M Pu(IV) in 4, 6, and 8M HNO₃ yield preliminary "G" values of 0.1, 0.07, and 0.04, respectively, for total gaseous products. With a "G" value of 0.1, the rate of pressure increase in a sealed vessel nine-tenths full of a 200 g Pu²³⁹/l solution is 1.4 psi/day (at 25°C).

Purex Ion Exchange Resin Pump Studies

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

The second and final resin pump modification resulted in a lowered resin attrition rate of approximately 2.8 ml/hr. The attrition rate with the original pump was about 65 ml/hr and about 3.8 ml/hr with the first modification. The final modification also produced a higher pumping rate than either of the first two configurations. It did not require added fluidizing liquid at the pump inlet as did the previous pumps and will allow the Purex N-cell ion exchange facility to operate with greatly decreased "slipwater" and a much better operating efficiency. The ion exchange loop was returned to Isochem personnel.

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Underground Waste Storage Heat Transfer
(George Jansen, Engineering Analysis Unit)

A computer program to describe an expanding heat generating sphere was completed by Computer Science personnel. This idealized model describes a growing disturbance of the normal, percolation heat transfer mechanism in the sludge of a waste tank. Within the disturbance, heat is transferred by conduction only. The first tests of the program indicated that time lags as large as one day in the response of hot spots to corrective action can be attributed to thermal inertia of the hot spot itself.

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TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Summary

Environmental Evaluation

The concentration of diesel fuel in river water at 100-D has increased to approximately 10 times (0.7 ppm) the amount found in water at Priest Rapids. The concentration at downstream locations (100-F and Richland pumping plant) remains at approximately the same level detected at Priest Rapids.

A particulate release from the 325 Radiochemistry Building caused no significant problems in areas accessible to the general public.

A summary of predicted thermal and radiological effects of the proposed Ben-Franklin Dam was forwarded to RL-AEC on August 2, 1967.

Dosimetry Studies

Development work on a portable neutron survey instrument continued as did studies on the performance characteristics of thermoluminescent and radioluminescent glass materials in dosimetry work.

Personnel Dosimetry

The use of a resonance-tuned magnetics sensing device for locating heavy metal particles, such as plutonium in human tissue, looks promising. Studies of the X-ray beam spectra were conducted in the Calibrations Laboratory. Improvements in simulated lung tissue for use in phantoms will simplify calibration studies for lung counters.

300 Area Wastes Treatment

Analysis of effluent from a scavenging test in the 340 Building, previously reported, showed all radionuclides other than cesium-137 to be within crib limits. Current studies show

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that cesium-137 can be reduced to crib limits by the inclusion of 150 mg/l powdered Zeolon in the scavenging process.

Instrumentation Services

Approximately 1,700 hours of satisfactory operation have now been achieved with the advanced laundry monitoring system, and the formal report draft describing the system has been completed.

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TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Environmental Evaluation

(J. F. Honstead, Environmental Studies Section)

Evaluation of problems associated with fuel oil entering the Columbia River at 100-N continued. Analysis of river water samples from four locations (Priest Rapids, 100-D, 100-F and Richland pumping plant), indicated an increase in concentration of oil at 100-D from approximately 0.2 ppm to 0.7 ppm during the month. It is anticipated that the continued presence of oil at this level in the raw water at 100-D may adversely affect the taste of the finished water. Concentrations at the other locations remained at or below the detection limit, about one tenth of the concentration found at 100-D.

Radioactive Particulates in 300 Area

Radiation Monitoring personnel detected radioactive particulates (primarily Pm^{147} and Ce-Pr^{144}) on the roof and ground around the 325 Radiochemistry Building, including the adjacent sections of the 300 Area parking lots. GM measurements on the particulates in the parking areas and roadways were generally 2000-3000 c/m, with less than one particle per 200 square feet. Although special air samples and particulate traps have shown no evidence of movement, loose sand containing these particulates is being removed from parking and road areas.

Effects of the Proposed Ben-Franklin Dam

A mathematical study of the proposed Ben-Franklin Dam was made in response to "Request for Related Services - BNW-155"; an assessment of the predicted thermal and radiological effects of the dam was included.

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Personnel Dosimetry

(H. V. Larson, Personnel Dosimetry Section)

Accurate location of plutonium or other heavy metal particles within tissue is a necessity for efficient excision. Nondestructive Testing Methods Development was requested to study the feasibility of producing a highly sensitive resonance-tuned electromagnetic (eddy current) sensing device for locating heavy metal particles in human tissue. A probe was designed which appears to be capable of both wide area search and pinpoint accuracy. A prototype model of this probe was built and used to detect 1/16 in. long sections of 1/16 in. diameter rod embedded in cardboard. It was possible to locate the specimens very accurately and to obtain an indication of their size and electrical conductivity. Hastalloy C was used as a satisfactory substitute for plutonium in the testing and calibration phases of this development effort since its electromagnetic conductivity is similar to that of plutonium. Since this method does not depend upon determining the relative intensity of characteristic radiation, it is also applicable to the detection of non-radioactive electromagnetic conductors embedded in tissue.

A calibration study was started to determine the feasibility of evaluating the deposition of uranium in the human body using whole body counter examination techniques. Uranium and its daughters emit several energetic gamma rays that can be detected outside of the body when the material is inhaled or ingested. Uranium-235 emits a 185 keV gamma ray that permits direct estimation of the quantity of this radioisotope. The first daughter of uranium-238 (thorium-234) emits a 93 keV gamma

ray. The relative quantity of 93 keV gamma ray observed is dependent upon the time from separation. Thorium is removed in the fission product separation process and requires about 100 days to return to equilibrium. The recent history (less than 100 days) of the subject uranium is necessary to properly interpret uranium-238 deposition by in vivo examinations. Remab phantoms are being used to calibrate and determine the detection limits of both the lung and the whole body counters for uranium. A number of employees who have not been bioassayed for uranium for several years will be scheduled for lung counter examinations for uranium next month.

Study of various lung counting systems continues with the goal of selection of the best possible lung counting arrangement for determination of plutonium and americium lung burdens. Under consideration are the Maushart large volume proportional counter, multiple thin scintillator counter and a system composed of two 5 in. diameter by 3/8 in. thick NaI(Tl) scintillators. Lung fillers loaded with various amounts of plutonium and americium were fabricated from a non-flexible tissue equivalent plastic material for use with the Remab chest phantom in the counter study. A search for a flexible material easier to use has not been fruitful so a method of casting the lung filler in sections outside the Remab lung cavity was developed. Prior to this a given lung filler had to be chipped out of the cavity when changing to another radionuclide or activity level and was thus destroyed and lost to further use. Lung fillers can now be changed and re-used at will.

Pulse height analysis of the standard K fluorescence and filtered X-ray beam is in progress with the assistance of the Biophysical Sciences Section. A lithium drifted silicon detector is being used, providing resolution improved by an order of magnitude over conventional scintillation spectroscopy. A precise delineation of the X-ray spectra will allow more accurate dosimetry and answer questions regarding the purity of K fluorescence sources. The measurements will also resolve the question of whether the filters for the heavily filtered spectra have been correctly designed.

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Forty-eight Eberline E-120 geiger counters that are being considered as a possible type replacement for the present pool GM's were issued to the field. To date, satisfactory performance has been obtained, although several minor design and manufacturing problems have been noted.

There were 528 bioassay samples submitted for plutonium analysis during the month, including 43 from new employees who had either previously worked with plutonium or who were assigned to work with plutonium in their assignment at Hanford. Also, there were 7 new plutonium deposition cases confirmed by evaluation of bioassay data. These were not the result of recent depositions, but re-evaluations based upon recent bioassay data. Also, 136 samples were submitted for uranium, 155 for tritium, 30 for promethium, 12 for americium and 1 for carbon analysis. There were 4 whole body counter and plutonium wound counter examinations conducted as a result of radiation occurrence. Evaluation of the results of these examinations did not indicate internal deposition of gamma emitting radionuclides in excess of 1% of the applicable permissible limits.

Special external exposure evaluations were performed for 10 Hanford contractor employees; however, no exposure over permissible limits was revealed. Special occupational exposure summaries were prepared in response to requests from new employees of six former Hanford employees under provision of the Atomic Energy Commission regulation 10 CFR 20 and AEC Manual Chapter 0524.

Dosimetry Studies

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

Thermoluminescent Dosimeter

Following instrument modification, sensitivity of the thermoluminescent dosimeter was measured for circular and oblong LiF-Teflon discs and found to be about five times greater than for the old reader. Exposures to determine the complete exposure response of the TLD system were conducted. In addition to more sensitivity, the new phototube apparently has more dark current which requires additional attention for proper evaluation.

Exposures of several TLD discs on top of one another were made to uranium beta, 16 keV and 52 keV X-ray radiation to determine the response of such a system. If the response is desirable, such a system could be used to measure beta exposures within certain limitations.

Portable Neutron Survey Instrument

The 2 in. tissue-equivalent proportional counter was used to measure the absorbed dose from the PuF_4 and PuBe sources in calibrations. Long exposures were used to obtain good statistical accuracy in the energy loss event distributions. These were converted to dose distribution functions and integrated to obtain the total absorbed dose due to the neutron radiation. In both cases (PuF_4 and PuBe), the tissue-equivalent proportional counter (TEPC) yielded a dose 10% higher than the long counter. In earlier experiments the TEPC yielded answers 10% or more higher than the long counter when exposed to monoenergetic neutrons from the Van de Graaff accelerator.

Mossbauer Effect Study

The Mossbauer drive system arrived and was assembled. It was found that the drive system functions properly in the constant acceleration mode but does not work in the constant velocity mode. Communication with the factory initiated attempts to correct this deficiency.

Radioluminescent Glass Study

The radioluminescent glass study continued with the exposure of dosimeters to the filtered and fluorescent X-ray sources of Calibrations Operation. The dosimeters were exposed to doses of 100, 500 and 1000 mR. The preliminary results indicate that the general energy response curves obtained were essentially as expected.

Several values deviated greatly from the expected indicating that the calibrated energies may not be as assigned. A study for the gamma energy characterization of the filtered

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and fluorescent X-ray sources was started using a TMC photo-spectrometer and a ND-181 multichannel analyzer.

A set of glass rods exposed to Co^{60} radiation doses of 100 R to 10^4 R was read on the Toshiba reader. The reader response was found to be nonlinear, since the glass rods have appreciable radiation darkening at 10^4 R. In the table are the results and the corrections necessary to evaluate glass rods.

<u>Co^{60} Dose,</u> <u>R</u>	<u>Toshiba Reading,</u> <u>R</u>	<u>Correction</u> <u>Factor</u>
100	1.38	72.46
200	2.66	75.30
300	3.46	86.81
400	5.18	77.81
500	5.76	86.81
600	7.20	83.33
1000	11.25	88.89
1500	15.90	94.34
2500	23.55	106.15
3750	26.00	144.23
5010	31.90	157.05
10,000	57.60	173.61

Previously we have used the factor 72.4 posted by Toshiba for all meter readings. The glass is linear from 1 R to about 150 R.

Internal Dosimetry

The creatinine in urine experiment progressed through the collection of sample stage. Total twenty-four hour collections were made of urine from an individual exposed years ago to plutonium nitrate and whose urine theoretically yields relatively constant amounts of plutonium over reasonably long periods of time. Creatinine analyses were done in triplicate and triplicate analysis for plutonium-239 was requested.

Computer-obtained values were received for various parameters of mathematical expressions of theoretical urinary excretion of plutonium from chronic inhalation of plutonium nitrate and oxide aerosols.

300 Area Wastes Treatment

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

Analysis of the effluent from the scavenging test in the 340 Building showed all radionuclides other than Cs^{137} to be within crib specifications. A DF of 1.6 was observed for Cs^{137} and this resulted in a concentration of 7.1 $\mu\text{Ci/gal}$ in the supernatant liquid. Therefore, laboratory studies were undertaken to investigate methods of improving Cs^{137} removal in the scavenging precipitation process. It was found that the addition of 150 mg/l of powdered Zeolon, a synthetic zeolite, would increase the Cs^{137} DF to 4. Laboratory observations indicated that this modification did not alter the physical characteristics of the sludge.

As a result of these conclusions, a second scavenging test was carried out on a 10,000 gallon batch of laboratory wastes in the neutralization tank in the 340 Building. Operational details of the second test were similar to those of the first. Less than 500 gallons (5% of the initial volume) of sludge were produced in the second test. The remaining 9,500 gallons were removed from the tank as clear liquid which fell within crib limits. Suspended solids in this supernatant liquid were 2 mg/l. A Cs^{137} DF of 4 was observed in the second full-scale test.

Aquatic Biology Services

(H. A. Kornberg, Biology Department)

Incidence of exposure and antibody development against columnaris disease in adult blueback salmon migrants was examined at Priest Rapids and Rocky Reach Dams and at Azwell Spawning Channel on the Columbia River and at Tumwater Dam on the Wenatchee River. Field trips were timed to obtain samples during or near the peak of the run at each location. Additional samples were taken at Rocky Reach and Azwell near

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the end of the blueback run for comparison of columnaris exposure and antibody development within the same migration.

Numbers of adult bluebacks showing columnaris gill isolates and agglutinating titers, and titer magnitudes, increased at upper river sampling sites. Increases were presumably due to longer time intervals following initial exposure and additional challenge encountered during migration. Similarly, the increase in isolates and titers during the second sampling period at Rocky Reach and Azwell suggests that columnaris exposure of fish is higher as the disease becomes more endemic in the river system.

Instrumentation Services

Laundry Monitoring System

(E. M. Sheen, Instrumentation Section)

Proper operation of the advanced conveyor laundry monitoring system which monitors protective clothing for alpha, beta, and gamma emitting radionuclides, continued with approximately 1,700 hours of satisfactory performance to date. This system employs two stainless steel wire belt conveyors that transport the wearing apparel at a belt speed of 160 inches per minute between two large alpha-beta-gamma detectors that simultaneously monitor upper and lower surfaces of each item for alpha-beta-gamma contamination. Eight 5 in. diameter photomultiplier tubes are combined with eight 6 in. x 7 in. light pipes and dual-purpose scintillators to provide 1, 2, or 4 fully independent monitoring channels approximately 24, 12, or 6 inches wide, respectively, and to accommodate the various widths of miscellaneous garments.

The instrument reliably sorts items for further decontamination if they are contaminated to a level greater than the equivalent of 25,000 disintegrations per minute (11 nCi) of Ra DEF or 1,000 disintegrations per minute (0.4 nCi) of 5 MeV alpha emitters distributed over 100 cm² in each case. In routine operation, the belt speed is 160 inches per minute and the beta-gamma count rate circuit time constant is three seconds. If increased sensitivity is desired, it can be achieved by

reducing the belt speed and increasing the time constant of the count rate circuits.

Completion of the formal technical report draft and delivery of circuitry line drawings to maintenance personnel completes the project except for future maintenance assistance.

Meteorological Services

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

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

AUGUST 1967
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