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MONTHLY ACTIVITIES REPORT
OCTOBER 1968

Division of Production
and
Hanford Plant Assistance
Programs

by the
Staff of Battelle-Northwest
F. W. Albaugh, Director

November 1968

PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON

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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
DIVISION OF PRODUCTION AND HANFORD PLANT ASSISTANCE PROGRAMS
W. D. Richmond

DIRECT AEC SPONSORED PROGRAMS

Summary

Spot radiographs, per Combustion Engineering instructions, on the head-to-shell weld, No. 160-4, on steam generator No. 4B have been completed. No weld discontinuities in excess of specification limitations were detected.

Radiography on the 10-foot diameter head-to-shell weld, No. 160-4, on steam generator No. 4B, cell 4, 109-N Building, 100-N Area has been completed after stress relief. The weld complied with the requirements of the ASME Boiler and Pressure Vessel Code, Section III (Nuclear Vessels), 1968 Edition.

Whole-body counts were obtained on 506 children of the Fruitland School in Kennewick. These measurements will be related to foods the students ate as indicated by diet records.

It now appears that the relative merits of monitoring river temperatures at 140-F vs. Richland in relation to State standards will be more dependent upon the extent to which power levels may have to be reduced than upon the number of days out of the year when such reductions are necessary.
DIRECT AEC SPONSORED PROGRAMS

Assistance to Combustion Engineering

(C. B. Shaw, NDT Customer Applications)

Spot radiographs, per Combustion Engineering instructions, on the head-to-shell weld, No. 160-4, on steam generator No. 4B have been completed. These radiographs were taken prior to stress relief of the steam generator weld in locations 8-9, 9-10, 10-11, 25-26, and 26-27. These areas were requested for radiographic testing examination due to original welding difficulties. No weld discontinuities in excess of specification limitations were detected.

Radiography on the 10-foot diameter head-to-shell weld, No. 160-4, on steam generator No. 4B, cell 4, 109-N Building, 100-N Area has been completed after stress relief. Because of the weld thickness (3.750 inches), a Cobalt-60 isotope was used as the radiation source for radiography. The weld complied with the requirements of the ASME Boiler and Pressure Vessel Code, Section III (Nuclear Vessels), 1968 Edition.

Mechanisms of Environmental Exposure

(J. F. Honstead, Effluent Studies Center)

The whole-body counting program for elementary school children was completed in Fruitland School in Kennewick. A total of 506 children maintained 7-day diet records and were counted in the mobile whole-body counter. This represented 82.5% of the eligible children and one of the highest percentages attained during the program. The next elementary school to be approached with the whole-body counting study will be the Eastgate School in Kennewick. Fifteen classrooms in this school were visited and presented with the opportunity to participate in the diet study and whole-body counting project. The whole-body counting truck will be moved to Eastgate School about November 1 to begin measurement of these children.
ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Summary

Mission 1

Test electrodes for nickel plating are undergoing development related to geometrical effects and surface finish in preparation for the eventual autoclave evaluation procedures.

Design studies were resumed on an improved model eddy current tester to perform clad thickness tests on N-type coextruded fuel elements. This unit will replace the UE-2B testers which have become maintenance problems and exhibit a nonlinear thickness readout. The new design features 100% solid state electronics and a logarithmic output corrector for linearizing the thickness readout.

An analysis of several minor difficulties associated with N-Fuels clad-to-core and cap-to-core bond tests was conducted and corrective recommendations made.

Ultrasonic inspection of primary pipe welds was completed. One indication was noted. Further examination with radiographic techniques is in progress.

Further NDT inspection included radiography of standard boiler welds, header welds, and horizontal control rods.

A trip was made to National Lead Co. of Ohio to inspect quality control and NDT facilities and provide counsel and advice in NDT problem areas.

The editing program EDIT for the ENDF/B Neutron Cross Section Library has been successfully modified to treat all isotopes that have been publicly released to date.

Loading of the PCTR to perform measurements on 2.1 wt% 235U NPR tubular fuel in a 3.1 inch hexagonal pitch water modified lattice with boron in the water is in progress. Measurements
Fatigue life of the blanked end-spiders has been found to be equal to or better than those produced by the EDM process. A die has been designed to punch the weld projections in the end-spiders.

Mission 8

The construction of the experimental facility to study heat transfer during simulated loss-of-coolant accidents is currently in progress. A special method has been developed to weld Indonel sheathed thermocouples to Zircaloy to enable the accurate measurement of fuel and pressure tube temperatures during the experiments.

The initial studies on the metal-water reaction rate for Zircaloy-2 tubes in a flowing steam atmosphere were completed for temperatures ranging from 800-1200°C. The purpose of these experiments was to determine the equation of state for the corrosion reaction and to develop a method for determining the hydrogen generation heated in a flowing steam atmosphere. It was found that the parabolic rate law fits the data for tests performed at 1000, 1100 and 1200°C. The estimated accuracy of the experiments was estimated as ±2%.

A report, "Atmospheric Release of Noble Gas Isotopes from a Coolant Disposal System Following a Reactor Accident," BNWL-CC-1689, was issued.
room temperature. Hydride needles dispersed in the metal below the case were not attacked. The anodic oxide layer produced was removed by anodic treatment in standard HNO3-HF etch solution.

Photomicrographs of a commercially obtained, tetramethylsilane (TMS) coated sample, oxidized to 4.5% burnoff, showed several large cracks in the coating. A graphite sample was coated at PNL in the presence of gamma irradiation. The sample was then oxidized with 6000 ppm H2O at 900°C and, after 163 hours of oxidation, a protection factor of three, compared to noncoated samples, was obtained.

Tests for cyanogen were made on He-N2 mixtures irradiated in the presence of TSGBF graphite. A brown deposit was found and its identification as a form of paracyanogen is in process.

Experiments were performed on an electrically heated model of a K-Reactor fuel column to investigate the maximum temperatures attained during simulated front riser failures. These experiments were performed to extend previous front riser failure experimental results to shorter scram delay times and greater control rod reactivity insertions than considered in earlier experiments. Comparisons of these results of this experiment to earlier results showed that, at a given initial flow rate and power level, considerably lower test section temperatures were obtained with these shorter scram delays and larger control insertions.

The construction of an electrically heated model of a C-Reactor, one inch overbore fuel column is nearing completion. It is expected that experiments investigating steady-state and transient heat transfer in this fuel configuration will commence during the next report period.

River surface water temperature surveys were made with an aircraft-mounted radiometer and a Bofors IR camera. The latter surveys were made at several river flow rates. Data obtained in both instances were of good quality.
ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Mission 1 - Basic Production

Nickel Plating Studies
(R. W. Stromatt, Corrosion Research Section)

The effect noted last month resulting in a thicker nickel plate at the ends of the cylindrical electrodes has been mini-
mized by machining the electrodes so that the ends are hemis-
pheral in shape. In addition it was found necessary to smooth
edges left by machining. This care is necessary for our ele-
rodes because of their small surface area with respect to the
derge length; on fuel elements this problem would not be as
noticeable or important.

To contact the electrode, a 1/8 in. rod is screwed into the
electrode (this rod also transmits the rotary motion to the
electrode). The inside edge where the rod contacts the electrode
is not plated very well. Consequently, the plate would fail at
that point in any subsequent heat or autoclave tests. To pre-
vent this failure, an end-capping procedure was developed:

1. The rod is cut and filed off flush to the plated
electrode.

2. This end of the electrode is etched in dilute sulfuric
acid.

3. The end of the electrode is plated for a few seconds
at a high current density (c.d.) to ensure good throw-
ing power for the initial coverage. This is followed
by low c.d. plating to minimize hydrogen bubble forma-
tion at the crevice edges where the rod is screwed into
the electrode. If a hydrogen bubble sticks, a pit is
formed.

4. After about 30 minutes of low c.d. plating, the electrode
is removed and inspected. If the plate appears good,
the electrode end is plated at a conventional c.d. until
Parameter changes can also be made from the teletype through the use of DDT if run change cards are not ready or only a few parameters are changed.

The input parameters for the overall N-Reactor simulation (secondary system only) have been put on cards and the pot set calculation program is nearly completed. Calculations will also be made for the primary system but without automatic pot settings since this part of the problem is on the 1132 and the pots cannot be set from the PDP-7.

Clad Thickness Tester Redesign
(T. J. Davis, NDT Systems Engineering Section)

Design studies were resumed on an improved model eddy current tester to perform clad thickness tests on N-type coextruded fuel elements. This unit will replace the UE-2B testers which have become maintenance problems and exhibit a nonlinear thickness readout. The new design features 100% solid state electronics and a logarithmic output corrector for linearizing the thickness readout.

Routine Support N-Reactor Fuels
(T. J. Davis, NDT Systems Engineering Section)

An analysis of several minor difficulties associated with N-Fuels clad-to-core and cap-to-core bond tests was conducted and corrective recommendations made. These problems arose from incorrect transducer frequencies, improper control of receiver stability in the clad-to-core test, and end cap geometry changes in the cap-to-core test.

Plant Support Testing Service
(C. B. Shaw, NDT Customer Applications Section)

Ultrasonic inspection of primary pipe welds in 100-N, cell 4, on 15-inch inlet pipes to both steam generators has been completed. Welds adjacent to the primary pump in the cell bypass line were also inspected. One indication was noted in the girth weld adjacent to the 4B steam generator in the inlet
Code Development
(K. B. Stewart, Theoretical and Neutron Physics Section)

The computer program EDIT which is used to edit specific ENDF/B neutron Cross Section Library isotopes was modified when it was discovered that several isotopes could not be processed. The processing difficulties occurred because of dimensioning that was too large for one file but not large enough in another. The dimensions were scaled to allow processing of all ENDF/B isotopes that have been released for public use.

Examination of Fuel and Tube from KW-Reactor Incident
(J. W. Goffard, Fuels Development Section)

Radiometallurgical examination of fuel elements and the Zr-2 process tube from the June 1968 KW-Reactor instantaneous flow blockage incident has continued. It had been reported previously that the fuel was apparently protected from oxidation or hydriding by the intact layer of protective Al, Al-Si, U high temperature reaction products. Elements from positions 16 and 17, 20 and 21, and 41 have subsequently been observed to have suffered corrosion attack. Metallography of the uranium from an element from either position 20 or 21 suggests that the uranium beta phase (660-760°C) may have been reached following the incident and reactor shutdown. Similarly, fuel metallography of the element from position 35 indicates that the uranium gamma phase (>760°C) was attained.

Punched samples from 15 different locations along the Zr-2 process tube were examined metallographically. There were no indications that the Zr-2 beta phase temperature (≈850°C) had been attained or that the tube had suffered any detrimental hydrogen pickup. However, there were several sites where a thicker than normal surface oxide had developed on one side of the clad.

Study of Inner Bore Corrosion of Al Clad Elements
(J. W. Goffard, Fuels Development Section)

The K-5 Al-Si canned Al clad fuel elements from PTA 067 that were adjacent to a fuel failure and that display inner bore
Brazed End Closures
(R. R. Studer, Fuels Development Section)

An instrument to electrically detect the onset of sintering in the powder metal braze compacts is being developed. This instrument will be used to determine the optimum of sintering conditions in a parametric sintering study. Vacuum sintering of braze rings is under way. Sintering is being investigated in an attempt to improve the time required for melting and the uniformity of flow. The first approach will be to determine sintering conditions that result in maximum conductivity and minimum warp.

Mission 7 - Target Space Enhancement

PCTR Water Tank Measurements on 2.1 wt% 235U NPR Tubes
(D. R. Oden, Reactor Neutronics Section)

Loading of the PCTR to perform measurements on 2.1 wt% 235U NPR tubular fuel in a 3.1 inch hexagonal pitch water moderated lattice with boron in the water is in progress. Relatively large positive water loss reactivity effects observed are expected to be reduced by replacing the KVE fuel surrounding the NPR fuel with 2.1 wt% DUN fuel. Measurements to determine the degree to which spectral equilibrium exists between the central test cell and adjacent buffer region will commence upon attainment of an operable reactor loading.

During the past month, various driver fuel and flux modifier configurations have been loaded in an attempt to find a loading condition to allow measurements to be made in the unpoisoned lattice. It was found that even with the minimum practical buffer size (one ring of 6 elements surrounding the center cell), a loading condition could not be achieved which simultaneously satisfied safety criteria for minimum drivers loaded, minimum control rod system worth, and maximum allowable water loss reactivity effects.
End-Spider Support Development
(R. R. Studer, Fuels Development Section)

A second fatigue testing fixture for end-spider supports has been built and is being used to accumulate fatigue data on blanked supports. Present indications, based on two specimens tested at each increment of loading, is that the fatigue life of blanked supports is equal to or better than that of the EDM produced supports.

The design of a die for producing weld projections on the spider supports is in the detailing stage and will be submitted for construction before November 1.

Mission 8 - Nuclear Safety

N-Reactor Fuel Temperatures Following a Primary System Rupture
(R. E. Collingham, Thermal Hydraulics Section)

The purpose of this program is to experimentally study heat transfer in an electrically heated model of the N-Reactor lattice following a simulated loss-of-coolant accident. Of particular interest in these experiments is the investigation of transient radiative heat transfer between the fuel and the pressure tube.

The construction of the experimental facility is currently in progress, and work is being performed in developing specialized fabrication techniques and evaluating equipment designs. The development of techniques to install thermocouples in the walls of Zircaloy tubes is progressing satisfactorily. An Inconel wire, representing a sheathed thermocouple, was successfully welded, using electron beam welding, to a Zircaloy wafer. The wafer, in turn, was successfully welded into a recess in the wall of a Zircaloy tube, again using electron beam welding.

A mock-up of the evolved hydrogen measuring system to be used in the loss-of-coolant tests is being prepared. This is a displacement system in which the gas is collected by feeding it into the top of a vertical water-filled column. As the water is displaced, the liquid level goes down. A differential pressure transducer will measure the head change. The mockup will be used to check this technique under various operating conditions.
ZIRCALOY-2--STEAM REACTION RATES

<table>
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<th>Temp., °C</th>
<th>Effective Time Interval, min.</th>
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<tr>
<td>1200</td>
<td>0-80</td>
<td>( V = 10.25 t^{1/2} )</td>
</tr>
<tr>
<td>1100</td>
<td>0-80</td>
<td>( V = 5.7 t^{1/2} )</td>
</tr>
<tr>
<td>1000</td>
<td>0-120</td>
<td>( V = 2.75 t^{1/2} )</td>
</tr>
<tr>
<td>900</td>
<td>0-7</td>
<td>( V = 1.6 t^{1/2} )</td>
</tr>
<tr>
<td></td>
<td>7-60</td>
<td>( V = 1.07 t^{1/2} + 1.5 )</td>
</tr>
<tr>
<td></td>
<td>60-80</td>
<td>( V = 0.087 t + 3.9 )</td>
</tr>
<tr>
<td></td>
<td>0-5</td>
<td>( V = 0.96 t^{1/2} )</td>
</tr>
<tr>
<td></td>
<td>5-120</td>
<td>( V = 0.083 t + 1.6 )</td>
</tr>
</tbody>
</table>

(a) \( V \) is expressed as cm\(^3\)H\(_2\) (STP)/cm\(^2\) Zry-2; \( t \) = time in minutes

These equations result in hydrogen volumes from 5 to 10 percent greater than that predicted from the Baker-Just Argonne equations. This is apparently a function of the dimensional change and distortions of the large diameter, long tubes that would not be as easily discernible or as pronounced with smaller specimens. The effect of autoclaved surfaces is now under study to duplicate conditions expected during hypothetical loss-of-coolant accidents for N-Reactor.

Release of Noble Gases in Reactor Accidents
(A. K. Postma, Air Pollution Chemistry Section)

Experiments were designed to provide understanding of absorption and subsequent surface release of noble gases from water pools. Emphasis is to be placed on bubble formation and escape because transport by this mechanism represents the major path for prompt release of the fission gases.

A report, "Atmospheric Release of Noble Gas Isotopes from a Coolant Disposal System Following a Reactor Accident," BNWL-CC-1689, was issued.
Process Technology

Inhibition of Carbon Steel Corrosion in Filtered Columbia River Water
(A. P. Larrick, Corrosion Research Section)

Four inhibitor systems were investigated for their ability to control pitting corrosion and corrosion product tuberculation growth in filtered Columbia River water.

A long-chain organic polymer film dispersing material, Betz DE-287B, failed to control either pitting or tuberculation and is being dropped from further consideration.

Two other inhibitor systems, Betz Dianodic 187, a zinc-phosphate-chromate formulation, and Calgon TG-10, a zinc-phosphate formulation, controlled pitting corrosion on fresh steel surfaces for short periods of time at high velocity conditions simulating N Reactor single-pass cooling conditions. In this regard, these two inhibitors appear to be comparable with an alternate method of controlling pitting, namely, the use of catalyzed sodium sulfite which removes oxygen from the water. However, before their use in N Reactor can be recommended, it will be necessary to investigate their ability to control pitting on steel surfaces containing magnetite films typical of those formed in high temperature water such as those on N Reactor piping.

The Betz Dianodic 187 and Calgon TG-10 were primarily investigated for their ability to control tuberculation at conditions simulating K Reactor cross-tie line conditions. Both materials significantly reduced tuberculation, film weights, and uniform corrosion but did not completely control tuberculation. The catalyzed sodium sulfite presently being considered for the application thus appears to offer slightly better tuberculation control. However, it is expected that better tuberculation control could be obtained with the Betz Dianodic 187 and Calgon TG-10 if the concentrations of these inhibitors were increased. The concentrations could be raised considerably before the cost would become comparable to the cost of using catalyzed sodium sulfite.
K-React Zircaloy-2 tubing which had approximately 2 and 8 mil case layers. Metallography of the treated specimens showed all of the continuous case had been removed in about 20 minutes but separated hydride needles located directly beneath the case layer were not removed, presumably because they were surrounded by metal. Hydrogen analyses of tubes before and after treatment were 1880 and 200 ppm. Removal of the anodic oxide layer was accomplished by anodically treating the anodized specimen in standard HNO₃-HF etch solution at room temperature. It was found the anodized layer could be removed but details have not yet been worked out.

Current consumption during the anodic dissolution of the hydride in HNO₃ approximated the conversion of Zr to Zr⁺⁴; however, some flaking of the hydride layer occurred. Substantial gassing was observed on the anode during the hydride removal. Solutions used to remove case layers contained substantial quantities of fine white precipitate with some flakes of apparently unchanged hydride.

Other solutions tested for anodic removal of hydride layers were 1% KOH, 10% oxalic acid, and 15% HNO₃. Even though the hydride did not anodize exactly like the metal in these solutions, its dissolution was very slow if at all or the metal was attacked.

Coating Formation on Graphite
(J. K. Yoss and R. C. Giberson, Chemical Ceramics Section)

A tetramethyilsilane (TMS) sample coated by the Marquardt Company was oxidized with 6000 ppm H₂O at 1000°C. The sample was oxidized to 4.5% burnoff and metallographically showed several large cracks in the coating. Photomicrographs of similar samples, unoxidized, had smaller cracks in the coating. It is possible that the thermal oxidation was at a higher temperature than the temperature during coating and this may be related to the increase in crack size.
hydroxide was added to the still-frozen mixture and allowed to warm to hydrolyze any (CN)₂. Tests for cyanogen by formation of Prussian-Blue were negative, indicating a G[(+CN)₂] value of less than 3 for vapor.

Another run was made for 43 hours using the molecular sieve trap. Again tests for cyanogen were negative, both in the gas and on the contents of the molecular sieve trap. The recirculator was then removed from the gamma facility and visually examined. A brownish deposit was present starting about 5 inches above the heater, extending for about 6 inches and then tapering off fairly rapidly. Fusion of portions of this material in KOH still gave negative tests for CN, but this may not be significant since it has not been demonstrated that all forms of paracyanogen hydrolyze in KOH to give cyanide.

It has previously been demonstrated that paracyanogen is formed on the interaction of graphite and microwave-excited nitrogen. There is little doubt the deposit found here is some form of paracyanogen, but efforts to identify it are continuing.

C-Overbore Steady State Demand Curves
(D. E. Fitzsimmons, Thermal Hydraulics Section)

Construction of an electrically heated model of a C-overbore fuel column is nearing completion. This model will be used to investigate steady-state and transient heat transfer in this fuel configuration during single- and two-phase coolant flow.

Assembly and welding of the model components were completed, and the seven outer surface thermocouples were installed. The ceramic supports were attached to the outer surface of the models. These supports which are shaped to model the arch type self supports of the fuel are made of ceramic to provide electrical insulation between the heater rods and the process tube. The quantity and spacing of these supports matches that of a normal fuel column.
downstream hardware. Of primary interest in these experiments were the test section temperatures following a simulated front riser failure. The following ranges of parameters were considered:

- Front header-to-rear header pressure drop, 40-250 psi
- Power level, 1500-300 kW
- Scram Delay, 0.4-2.0 sec.

Comparison of these preliminary results with earlier results shows that, for a given flow rate and power level, considerably lower test section temperatures were obtained with these shorter scram delays and larger control rod reactivity insertions.

In these experiments, the heat rejected to the coolant from the graphite (5% of the total tube power) was simulated by two methods: adding it to the rod power and, more realistically, adding it through the process tube wall using an electrical heater strapped to the tube. Also, experiments were performed with no graphite heat simulation. The data for heating through the process tube wall agreed well with the data for no graphite heating simulation. Thus, these results indicate that graphite heating need not be simulated in similar future transient experiments. Preliminary analysis of this data indicates that graphite heating simulation through the process tube (or not simulated) rather than in the rod results in a 15% higher indicated allowable reactor tube power.

River Temperature Traverses
(J. R. Eliason, Water Resources Systems Section)

On October 1, 1968, several traverses of the Columbia River were made with the aircraft-mounted radiometer to check with temperature traverses being collected from a boat. The radiometric temperature measurements were recorded on magnetic tape and will be reproduced on a strip chart recorder for final analysis. In-flight observations of the radiometer output indicated a river temperature of approximately 17.4°C and
100-BC Trench Disposal
(D. D. Tillson, Water Resources Systems Section)

Monitoring of water levels and temperature patterns in the 100-BC Area affected by effluent disposal at the 107-C trench was renewed. Some minor problems with the continuous temperature logger presently prevents any assessment of the hiatus in effluent disposal during the summer months. Water levels in the wells nearest the effluent trench show direct response to the renewed disposal. Water level response in the periphery wells is masked by seasonal variations in the regional ground water system and do not as yet indicate any definite trend that could be attributed to the effluent disposal. A continuous temperature recorder was put back in service on the warmest spring along the river bank below the Disposal trench. The temperatures measured to date are at the same levels recorded during the previous disposal period and again indicate a very short holdup time and cooling effect of the system. Data from the previous test period are being compiled and prepared for publication in report form separate from the new test data.
ASSISTANCE TO ATLANTIC RICHFIELD HANFORD COMPANY

Summary

Mission 1

A flowsheet utilizing an acid fluoride strip solution has been developed for the recovery of plutonium and americium from the Plutonium Reclamation facility aqueous waste stream. Weldments of 304L, 316, Incoloy 825, and Hastelloy C-276 were exposed to solutions simulating those found in the strip column. Corrosion of Incoloy 825 and Hastelloy C-276 was slight, whereas 304L and 316 were attacked moderately, one to four mils/month.

The corrosion resistance of cylinders fabricated of 316 stainless steel by a powder metallurgy technique was evaluated in simulated Purex feed solution. Corrosion was negligible.

Corrosion of 304L was determined in sodium tungstophosphate solutions. End-grain attack was severe in a saturated (about 0.3M) solution at 25°C. However, attack was nil in solutions of process concentration (0.017M) at 40°C.

Two Savannah River type miniature centrifugal contactors (a single-stage model and a multi-stage unit) are being fabricated.

A new sample of Purex aluminum-clad dissolver solution demonstrated a 30-fold niobium DF improvement when spiked with Zircaloy-clad dissolver solution prior to extraction. Results to date suggest an alternate procedure for improving both zirconium and niobium decontamination.

Discussion of the problems involved in the continuous measurement of the H⁺ concentration in slag and crucible dissolvers led to establishment of detection requirements and the need for circumventing interference effects from other ions present in the dissolver.
Work continued on the development of a mathematical model of the Purex backup facility for nitric acid recovery. Preliminary predictions of plant performance correlate well with actual performance, indicating that existing literature data will be useful in predicting performance under different operating conditions, or with modified equipment.

Corrosion tests showed the feasibility of using a condenser system constructed of 304L during the dissolution of Zry-2 clad U-Zr alloy fuels in boiling acid-fluoride solution. The maximum measured corrosion rate within the condenser was 0.7 mil/mo.

Corrosion of 304L stainless steel and of Uranus S Alloy was evaluated in simulated sugar-killed Purex acid waste solutions. Variables were temperature and nitric acid, fluoride ion, ferric ion, and Cr(III) concentration. The results to date indicate corrosion rates >0.5 mils/month in all solutions. The corrosion resistance of 304L appeared to be slightly superior to that of Uranus S Alloy.

Vapor phase corrosion of Hastelloy N exposed to cesium chloride at 675°C was found to increase as a result of an air sweep of the vapor phase.

Laboratory settling rate studies were performed with a solids-containing sample of PAS from a current plant test. Addition of Purifloc C-31 resulted in a clear supernate.

Laboratory data indicated that periodic additions of small quantities of new plant solvent inventory may be an acceptable mode of operation. Eventually disposal of worn-out solvent will probably be required and would be facilitated by treatment with sulfuric acid or methanol to cause fractionation into a reusable NPH phase and a small-volume HDEHP-TBP phase.

Use of the LC column for solvent washing was also investigated during the month.

Conditions were established which permit satisfactory B-plant extraction of 90Sr from the waste solutions which will result from Purex processing of U-Zr alloy fuels.
Further calculations were made on thermal characteristics and economic consideration of cesium and strontium capsules.

Process Technology

In Purex process second and third plutonium cycle flowsheet tests, miniature mixer-settler runs were made to complete testing of modified 2B column operation. Use of laboratory-prepared (vice vendor-supplied) Fe(NO₃)₂ gave Pu losses of 2.4 to 2.6%, which still exceed the desired 2%.

A series of mixer-settler runs to test flowsheet modifications aimed at improving CC column performance has been initiated.

Activated carbon exhibited a high capacity for removing dissolved TBP, DBBP, and CCl₄ from CWW. Plutonium was also removed (DF > 4) but americium was not.

A study of the kinetics of the oxidation of Np(IV) to Np(V) in TBP-WP by HNO₃ is in progress. Preliminary indications are that the oxidation is very slow.

Laboratory tests of an ARHCO conceptual "heel-type" thorium dissolution flowsheet were completed and appeared to be highly successful.

In Zirclex process studies, efforts to define dissolver off-gas composition continued. Results were characterized by a lack of reproducibility.

In studies on the use of hydrazine-stabilized ferrous nitrate as a plutonium reductant, the half-life of ferrous nitrate in nitric acid is being measured as a function of storage temperature, acidity, and hydrazine content.

A multi-batch dissolution run is in progress aimed at determining the effect of a steady-state heel of solids on the dissolution of ZPPR fuel scrap.

Exceptionally good results have been obtained in all tests of the glass mockup of the 234-5 dissolver. Single component
Both transport theory (DTF) and multigroup diffusion theory (HFN) have been used to analyze the data on reflector savings for enriched uranyl nitrate solutions. Both transport theory and diffusion theory overestimate the measured reflector savings, but the DTF calculations provide the best agreement with experiment; the computed values are 0.1 to 0.7 cm larger than measured values which range between 4 to 5.2 cm.

Coulombic potentials are also being calculated for the new ground water map. These will be verified by comparison with field data.

Neutron moisture probe measurements in many 200 Area wells show essentially the same moisture content as measured in the wells three years ago. This is strong evidence of an equilibrium state.

Moisture probe measurements were made in the 108-SX tank to determine the moisture content of the sludge layer.

Soil samples from beneath the sludge in the 105-A tank were analyzed for the presence of Cs, Ce, Pr, Zr, Nb, Eu, and U.

Attempts to core the sludge in the 105-A tank were unsuccessful.

Horizontal crustal movement results were received from the USGS, which are being reviewed. This information is being correlated with geologic and field data.
ASSISTANCE TO ATLANTIC RICHFIELD HANFORD COMPANY

Mission 1 - Basic Production

Materials of Construction
(R. F. Maness, Corrosion Research Section)

A solvent extraction flowsheet has been developed for the continuous recovery of americium and plutonium from the Plutonium Reclamation facility aqueous waste stream. The flowsheet uses an acid fluoride strip solution. Weldments of 304L, 316, Incoloy 825, and Hastelloy C-276 were fabricated and exposed to solutions simulating those found in the strip column. Corrosion of all test materials was slight, 0.01 to 0.06 mils/mo., in two-phase systems, apparently due to preferential wetting by the organic phase. Weldments of Incoloy 825 and Hastelloy C-276 were only slightly attacked in aqueous solutions, whereas 304L and 316 weldments were attacked moderately (one to four mils/mo.). In addition, the weld metal of 316 weldments exposed to aqueous solutions was subject to preferential attack and a 304L specimen exposed to a chloride-bearing solution was subject to end-grain attack.

Cylinders fabricated of 316 stainless steel by a powder metallurgy technique are being considered for use as sieve plate spacers in Purex extraction columns. Their corrosion resistance was evaluated in simulated process solutions and in boiling 65 wt% HNO₃ solution. Corrosion in the nitric acid solution was severe. However, negligible corrosion occurred in the simulated process solutions at 50°C.

Miniature Centrifugal Contactors
(D. G. Bouse, Applied Chemistry Section)

A Savannah River Laboratory designed, single stage, miniature centrifugal contactor is now being fabricated for use in
Instrumentation Development
(D. P. Brown and W. G. Spear, Instrument Research and Development Section)

Discussion of the problems involved in the continuous measurement of the $H^+$ concentration in slag and crucible dissolvers led to establishment of the requirements, including an accuracy of $\pm 0.5M$ in the range of 1-5M or higher. Initial investigations indicate that a combination of measurement techniques will be needed, where the principal method might entail electrodeless eddy current conductivity determinations. The use of several techniques on a joint basis should materially reduce the interference effects from other ions present in the dissolver, thus enabling a legitimate determination of the $H^+$ concentration. The toroidal eddy current measurement method, using small coils, gives promise for an initial approach for investigation.

Mission 4 - Plutonium-238

Solvent Extraction Flowsheet for $^{237}$Np and $^{238}$Pu Separation
(D. G. Bouse and W. W. Schulz, Applied Chemistry Section)

A miniature mixer-settler unit has been installed in the 222-S Building 1E-cell to demonstrate proposed solvent extraction flowsheets for recovering and separating $^{237}$Np and $^{238}$Pu. Feed for these runs will be prepared initially from dissolver solutions obtained about six months ago from dissolution of irradiated NpO$_2$-Al target elements. This material contains about 2.5 g/liter of $^{237}$Np and 0.25 g/liter $^{238}$Pu and is about 1.0M in Al(NO$_3$)$_3$ and 3.7M in HNO$_3$.

In preparation for these runs, portions of the dissolver solution were adjusted to acidities in the range 2 to 6M HNO$_3$ and allowed to stand 24 hours at 25°C. They were subsequently contacted (10 min, 25°C, A/0 = 1) with 30% TBP-NPH. At equilibrium aqueous phase acidities of 3.5 and 6M HNO$_3$, 2.98% of the $^{238}$Pu and from 96.9 to 97.2% of the $^{237}$Np reported to the organic phase. At 2M HNO$_3$ in the aqueous phase, however, only about 87.7% of the $^{237}$Np extracted. Spectrophotometric analyses showed the higher acid feeds contained mainly Np(IV) while some Np(V) was present at 2M HNO$_3$. A desirable goal of
Mission 9 - Waste Management

Organic Waste Disposal
(G. E. Benedict, Chemical Engineering Section)

A study was made of disposal methods for Plutonium Rec- lamation Facility Solvents by means other than direct incin- eration; as a result of this study, the use of a fluidized bed calciner for 234-5 Building aqueous wastes has been pro- posed. The suggested calciner would be of the in-bed com- bustion type which could also be used as an incinerator- scrubber for TBP and DBBP bottoms from a CCl₄ still. It is hoped the P₂O₅ and HCl formed when TBP or DBBP burns and CCl₄ hydrolyzes will react with and be retained by the bed of mixed Al, Mg, Ca, and Na oxides.

This type calciner should be ideally suited to 234-5 Building aqueous wastes. Idaho Nuclear reports that nitrate ion is reduced to nitrogen gas by the fuel oil supplying the calcination heat. Therefore, no NOₓ gas recovery equipment would be needed.

Radioiodine Trapping
(L. A. du Plessis, Air Pollution Chemistry Section)

The conversion of ethyl iodide to ethyl nitrate and silver iodide on a smooth silver nitrate-coated cylindrical surface was measured following improvement in the experimental arrange- ment and method. At 89°C, with He as the carrier gas and at an ethyl iodide pressure of 43 torr, the degree of conversion of AgNO₃ to AgI was proportional to the square root of time and exposure. Deviations from the t¹/² dependence found in earlier experiments with a capsule of crystals of AgNO₃ were probably due to significant decreases in the reacting area with time. Complete conversion of small granules would account for the progressive decrease observed. The current series of experiments at relatively high organic iodide vapor pressure has served to establish that such compounds of iodide do react with silver nitrate at significant rates. Attention will now be given to removal of organic iodides under conditions more representative of the Purex plant streams expected to contain organic iodides.
A "Permasep" research permeator has been ordered from DuPont for comparison with the existing unit. The membrane in the permeator is comprised of very small diameter hollow fibers. Potential advantages of this type of membrane configuration suggest that such an evaluation would be worthwhile.

Movement of Radionuclides Through Hanford Soils
(R. C. Routson, Water and Waste Management Section)

A computer program is being developed which will predict radionuclide distribution and breakthrough as a function of influent volume and concentration of solution ions, the concentration of soluble components in the soils, the exchange and sorption properties of the soil solid phase, solubility product relationships of the known slightly soluble and insoluble salts of the soil solution phase, and soil to solution ratio. The initial-preliminary program is completed and soil parameter determinations and column verification for a Burbank Sand soil are being initiated.

Materials of Construction
(R. F. Maness, Corrosion Research Section)

Tests were made to determine the feasibility of utilizing a condenser system fabricated of 304L during the dissolution of Zry-2 clad U-Zr alloy fuels. Zircaloy-2 tubing was dissolved in boiling 1M HNO₃-1M Al(NO₃)₃-2M HF solution with the vapors condensed in a Teflon condenser. The terminal solution contained 3M fluoride and 0.5M Zr(IV). Coupons of 304L were placed in the condenser and one coupon was positioned immediately above the condenser. Ten dissolutions were completed to give a total exposure time of 40 hours. Corrosion rates of 304L coupons placed in the top, middle, and bottom of the condenser were 0.7, 0.1, and 0.0 mils/mo, respectively. The coupon immediately above the condenser corroded at a rate of 4.5 mils/mo.

Corrosion of 304L stainless steel and of Uranus S Alloy was evaluated in simulated sugar-killed Purex acid waste solutions. Variables were temperature (90°C and boiling), nitric acid concentration (0.5 to 1.5M), fluoride ion concentration
inventory until very high HDEHP and TBP concentrations are attained may be an acceptable mode of operation. For example, in one test, current B-Plant solvent was butted to 0.9M HDEHP and 0.5M TBP; the specific gravity of the extractant increased only 7 percent while the Sr $E_a$ increased about fourfold. If disposal of an inventory of worn-out solvent should become necessary, treatment of the extractant with either concentrated sulfuric acid or methanol may be desirable. Either reagent fractionates the solvent into a reusable NPH phase and an HDEHP-TBP fraction which must be discarded. A fivefold reduction can be achieved in the volume of organic material which must be stored or incinerated.

Since startup, the B-Plant has used the IC column to acid strip fission product rare earths from the extractant. Subsequently, the solvent is batch washed with a NaOH-sodium tartrate solution to remove radioactive yttrium and small amounts of inert impurities. Since there currently is little economic incentive to isolate the rare earth fraction, consideration is being given to performing the solvent wash step in the IC column. Batch studies conducted to simulate proposed column washing conditions demonstrate this mode of operation is entirely feasible.

Platinum-Catalyzed Reductions Using Hydrogen and Hydrazine (J. L. Swanson, Applied Chemistry Section)

The reduction of Pu(IV) in aqueous solution by H$_2$ in the presence of platinum catalyst has been confirmed. Bubbling an 8% H$_2$-Ar mixture through a tube containing 1/8 inch pellets of 0.5% Pt on alumina and a 0.005M solution of Pu(IV) in 2M HNO$_3$ + 0.02M sulfamic acid resulted in reduction of about 92 percent of the Pu(IV) in two minutes at room temperature. In a similar experiment with 0.009M N$_2$H$_4$ + 2M HNO$_3$ and pure Argon as the gas, all of the Pu(IV) was reduced within two minutes. The N$_2$H$_4$ reaction appears simpler for the aqueous phase reduction.

There would appear to be benefits from carrying out the reduction of Pu(IV) in the organic phase alone rather than in a two phase system. For this application, reduction by H$_2$
Cesium Precipitation from Acid Wastes
(J. A. Partridge, Applied Chemistry Section)

Analytical data from the hot cell test with PAW (September report) showed good DF values which are comparable to those of the former test. The DF for Sr 89 + 90 obtained during this test was 18,000. The Cs loss to the supernate was 0.3% percent.

This test concluded hot cell and laboratory studies of Cs-PTA precipitation and a report will be written.

Deep Storage of Hanford Wastes
(D. D. Tillson, Water Resources Systems Section)

A concerted effort was begun to obtain and analyze data that would pertain to deep storage of high level radioactive wastes. Numerous informal discussions were held with authorities in many fields that might be concerned with deep storage. A preliminary outline proposal was prepared and is scheduled for discussion with ARCO during the first week in November prior to going into the final draft proposal stage.

Fuel Element Crushing
(J. Dunn, Chemical Engineering Section)

Splitting of the combined inner and outer elements of N-type fuel with a press-operated piercing die required from 70 to 75 tons total pressure, approximately 30% of the force needed when crushing with die and platen.

In the most effective die and platen test, the tooling was damaged by the excessive pressure (250 tons) applied. With the piercing die, the stem end of the moving die was bent and the fuel element was cocked at an angle from the vertical. A redesign of the parts will provide a stationary piercing point over which a fuel element will be ejected from a supporting sleeve of sufficient rigidity to prevent lateral deflection.
a layer of dry soil a few feet thick immediately adjacent to the
tank is the only part of the soil that is important for periods
of change up to one month. The calculations assumed that tanks
already were near operating temperature. The equivalent thick-
ness of the layer of dry soil is determined by the total heat
generation rate within the tank, the moisture content of the soil,
and the temperature level within the tank. Use of this tech-
nique with the heat transfer data from the wet soil cube experi-
ment to determine the boundaries of the heat transfer system with-
in the soil surrounding the tank should greatly reduce the diffi-
culty in making transient thermal calculations for tank farms
with more than one tank.

Waste Packaging Studies

A. Cs-Sr Capsule Design Studies
   (L. K. Mudge, Chemical Engineering Section)

   Calculations on the thermal expansion of SrF₂ (linear
   thermal expansion coefficient of 20 x 10⁻⁶ in./in./°C)
   packaged in a 3 inch OD cylindrical cladding (linear
   thermal expansion coefficient of 11 x 10⁻⁶) showed that
   the SrF₂ could be compacted to 96 percent theoretical
density at room temperature and would increase to 100 per-
cent theoretical density if the package were increased in
temperature by 1000°C. Cesium chloride packaged at 71, 78,
and 80 percent of theoretical density at room temperature
in a 2 inch OD capsule would reach 100 percent theoretical
density at 645°C (CsCl(l)), 645°C (CsCl(s)), and 470°C,
respectively.

   The inner cladding of the SrF₂, or CsCl, capsule will
be at a higher temperature than the outer cladding at the
time of loading the inner into the outer clad. If the tem-
perature of 2-1/2 in. OD inner cladding is 545°C and the
outer cladding is at room temperature at the time of load-
ing, the ID of the outer cladding must be at least 0.01 in.
larger than the inner cladding OD at the same temperature
so that the inner clad will fit into the outer clad.
The use of the thermosyphon to prepare and concentrate cesium chloride solution does not appear to be practical. Stable operation of the unit with dilute acidic solutions was not attained. A variety of feed points and heater locations were tried but all were unsuccessful.

Small-scale pan dryer tests using glass equipment indicated that it will be difficult to obtain a granular CsCl from a simple pan dryer. As the chloride converts to the solid form it cakes up in front of the wiper blade and large agglomerates are formed.

C. Engineering Scale Flowsheet Studies
(L. K. Mudge, Chemical Engineering Section)

Three runs were completed on the precipitation and filtration of SrF₂. A KF solution was used to precipitate the SrF₂, resulting in an extremely fine precipitate (particles down to 1 μ in diameter as determined using Stokes equation for creeping flow around spheres).

In Run 1, the Sr(NO₃)₂ feed was neutralized to pH 7, KCl solution was added (5 percent excess) at 150°F, and the slurry was digested for 2 hr prior to filtration through an alundum crucible. Recovery of SrF₂ in the filter was poor since the filter was cracked during filtration. In the second and third runs, the pH was adjusted to pH 10; the resulting slurry of Mg and Fe hydroxide was centrifuged, and the supernate was then adjusted to pH 7 before precipitation of SrF₂ at 175°F. In the third run, the feed was added to the strike solution which resulted in a finer precipitate than was obtained by adding the strike solution to the feed solution. The SrF₂ slurry was digested for two hours at 175°F in both Runs 2 and 3. The filter was cracked again in Run 2 which made the yield poor. The yield was about 86 percent of stoichiometric in Run 3.

The digestion period had no effect on the product size as determined by settling rates, except in Run 3, where a significant increase in the settling rate was noted. The products from Runs 1 and 2 both had 2 percent volatiles
Another experiment was performed using graphite disks, obtained from Gulf General Atomic, which were coated with a pyrocarbon. These pyrocarbon samples were placed in the bottom of a reduction vessel and fired. After reduction, it was impossible to identify the disks. It appears that the pyrocarbon tested is not compatible with the environment.

Purex Process Second and Third Plutonium Cycle Flowsheet Tests
(D. G. Bouse, Applied Chemistry Section)

In completion of studies on flowsheet modifications suggested for the Hanford Purex plant second cycle strip (2B) column operation, a mixer settler run was made to again test the use of Fe(NO$_3$)$_2$ as the reductant in the 2BX stream. Ferrous nitrate for this run was prepared in the laboratory (instead of vendor supplied Fe(NO$_3$)$_2$ used in previous runs) by the dissolution of ferrous carbonate in nitric acid. To assure reagent stability, the solution was prepared and used at a temperature near 0°C and precautions were taken to exclude air during preparation. Composition of the 2BX stream for this run was 0.14M Fe(NO$_3$)$_2$, 0.15M N$_2$H$_4$ and 0.2M HNO$_3$. With no 2BS-HNO$_3$ (12.2M HNO$_3$) added to the system during the first four hours of operation, Pu losses were 2.4 percent. The 2BS-HNO$_3$ stream was introduced during the last three hours of operation and Pu losses increased to 2.6 percent.

Although waste losses for this run are the best yet achieved with Fe(NO$_3$)$_2$ '5 percent minimum for all previous runs', they still exceed the 2 percent waste loss required for continuous plant operation.

Charcoal Treatment of Z-Plant Waste
(R. E. Lerch, Applied Chemistry Section)

Investigation continues on loading of organic waste materials onto carbon beds from a high salt stream (0.25M HNO$_3$, 0.4-0.9M AlF(NO$_3$)$_2$, 5.5-6.5M total nitrate). Tributyl phosphate, DBBP, and CCl$_4$ adsorb onto activated carbon to the extent of 0.43, 0.61 and 0.28 liters of organic per liter of carbon, respectively. The loaded carbon bed ("carbon sorbate") containing either TBP
0.6M HF, three hours reflux time, and subsequent addition of 0.5M Al(NO₃)₃ to complex the fluoride. A total of 220 grams of oxide were charged in five consecutive dissolution runs. The final dissolver heel contained 3.2 grams of solids representing 1.5 percent of the total oxide charged, a negligible solids accumulation. This procedure will be repeated with a new lot of oxide in the near future.

Oxidation of Np(IV) to Np(V) by HNO₂
(G. F. Schiefelbein, Applied Chemistry Section)

A study of the kinetics of the oxidation of Np(IV) to Np(V) in 30% TBP-NPH by HNO₂ is in progress. The oxidation of Np(IV) to Np(V) could have important ramifications in Purex 3B column performance where separation of the neptunium product from thorium is sometimes less than adequate. If Np(IV) could be oxidized to the (V) state in the organic phase it would transfer to the aqueous phase providing effective decontamination from thorium. This chemistry also has long range significance to recovery of neptunium directly from HAW concentrate which also contains thorium.

Preliminary indications are that Np(IV) is very resistant to oxidation by HNO₂ in 30% TBP. A TBP solution 0.15M in HNO₃, 0.03M in HNO₂ and containing 4.2 g/liter Np(IV) was prepared, maintained at 40°C, and scanned, spectrophotometrically over a 72 hr period. No oxidation of Np(IV) to Np(V) was observed. Higher HNO₂ concentrations will be evaluated in the future.

Dissolution of Thoria Fuel Elements
(W. W. Schulz, Applied Chemistry Section)

A conceptual "heel-type" flowsheet has been proposed by ARHCO personnel for Purex plant dissolution of irradiated ThO₂ wafers. Laboratory tests of this flowsheet performed with unirradiated DUN ThO₂ wafers of current design were highly satisfactory. (The DUN elements are 2 1/8 in. long x 1.5 in. diameter and are made from National Lead Co. of Ohio Precipitation Process thoria powder.) The conceptual flowsheet involves dissolution (in boiling 13M HNO₃-0.1M Al(NO₃)₃-0.05M KF) of 59 percent of a charge of fresh wafers plus heel from a previous charge in
experienced in the CC column in partitioning uranium and plutonium satisfactorily. The base flowsheet employs hydroxylamine nitrate to reduce Pu(IV) to Pu(III) but, in most plant runs, considerable Pu(IV) was present in the exit aqueous product stream. A series of mixer-setter runs to test flowsheet modifications aimed at improved CC column performance has been started. These runs will use PRF solutions (obtained in Pu-only runs) spiked with the appropriate amounts of uranium. The initial run was made to test the basic CC column flowsheet. Results closely paralleled plant experience, i.e., about 25 percent of the plutonium remained in the organic phase and, of that stripped, 78 percent was as Pu(IV). Additional runs are in progress.

Structural Temperatures
(G. Jansen, Chemical Engineering Section)

Natural convection during tank startup appears to reduce the time for moisture to condense on the tank dome during tank startup to less than 30 minutes after hot supernate is introduced into the tank. This does not result in appreciable increased differences between dome and slab temperatures for thermal stress calculations over steady-state difference. However, the thermal gradients across the inner quarter of the concrete wall and dome are very high.

Modelling Purex Nitric Acid Recovery System
(D. Bolme, Chemical Engineering Section)

The conversion and recovery of nitric oxide as nitric acid requires two alternating steps; the oxidation of NO to NO₂, and the absorption of NO₂ in water to form nitric acid and NO. Either of these steps, or both of them, can be rate controlling although the literature indicates that the oxidation of NO tends to be rate controlling at atmospheric pressure, while the absorption of NO₂ can be rate controlling at pressure around 4 atmospheres absolute. Oxidation of NO takes place as a homogeneous gas phase reaction. There are substantial differences of opinion in the literature as to the mechanism whereby this reaction proceeds, but little doubt about the rate at which NO is oxidized, which is all that concerns us here.
Hydrogen-Oxygen Safety Studies
(W. Willingham, Chemical Engineering Section)

Initial runs in the annular container indicate that a much higher pressure is developed than in the straight cylinder. A higher range pressure transducer is being procured to enable extension of the experimental data.

Thorium Denitration Studies
(W. Willingham, Chemical Engineering Section)

A 5 liter glass vessel has been set up in Room 115 of the 324 Building for study of the proposed nitric acid removal procedure. Test runs are being made on H2O to check out the system.

Drawings for equipment setup and modifications for the jet test stand have been made and the necessary pieces of equipment are being located. Installation in EDL-102 of the 324 Building is planned.

Instrument Development
(D. P. Brown and W. G. Spear, Instrument Research and Development Section)

Planning discussions centered on the development of specific gamma spectrometry techniques applicable to the determination of fissile material in irradiated fuel elements received in casks from the reactor areas. Appropriate measurements made prior to insertion of the fuel pins in Purex dissolvers could materially enhance the nuclear safety of the operation.

Automated Fe(II) Determination in LBX
(J. L. Russell, Chemical Sensors and Instrumentation Section)

A tridentate chelating agent, 2,4,6-tripyridil-s-triazene (TPTZ), reacts specifically with Fe(II) under very simple conditions. The resulting intense blue-violet color permits Fe(II) to be measured colorimetrically at the parts per billion level. Tests were conducted to determine whether the TPTZ
Vessel Vent Off-Gas Chemistry  
(L. A. du Plessis, Air Pollution Chemistry Section)

Changes in the sampling train permitted a sample to be obtained from the vent system at Purex plant which showed none of the several gas chromatographic peaks obtained with the previous sample. The several peaks obtained initially were ascribed to evolution of volatile organic compounds from components used in the train. Only one peak, not yet identified, appeared in the more recent sample. It is not due to lower alkyl iodides. Further improvements in the sampling train and sample analysis were investigated; these included better trapping of N₂O₄ and an improved method for employing a larger aliquot of the cold trap sample in the chromatograph.

Hydrological Assistance  
(D. B. Cearlock and D. D. Tillson, Water Resources Systems Section)

To calculate the permeability distribution and travel times of the Hanford groundwater flow system at the groundwater table surface, it is necessary to construct a groundwater table potential map and a groundwater table flow path map. The groundwater table flow path map has been drawn. Travel times and permeabilities are presently being calculated along flow paths. The calculated data will be verified by comparison with field data on the present waste movement.

A group of wells surrounding the B and BX tank farm and well #11-45A on project ALE were logged with the 4 in. diameter neutron probe for comparison with previous neutron logs obtained on the same wells in 1965. The findings in all cases were practically identical both in relative moisture by volume and distribution in depth, except for the first 5-10 ft below surface. The indications are that the unsaturated zones in these locations are in an apparent equilibrium state. Calibration of the 4 in. neutron probe is presently under way and will provide a means of making quantitative measurements of moisture content within the type of monitoring system now in use.
drill with an 18 in. split-core barrel. Results from a core attempt, taken during September in the same tank, indicated special precautions would be required in order to hold the semi-liquid material in the core barrel after penetration of the relative hard surface layer and obtain a complete section. After coring through approximately 4 in. of the upper crust, however, the material lodged in the core barrel and prevented any recovery of the more liquid material underneath. No further core attempts are immediately planned.

Results were received from the U. S. Coast and Geodetic Survey on a simultaneous adjustment of six triangulation networks encompassing the Hanford Project and surrounding area to approximately 50 miles radius. This study was requested as an aid in determining if horizontal movement of the earth crust had occurred and could be detected using historical measurements. Initial analysis of the results fails to show any obvious patterns of horizontal movement that could be attributed to crustal movement, particularly along the controversial Olympic-Wallowa Lineation. Further detailed study of the results is in progress and will be incorporated with vertical crustal movement studies utilizing benchmark data.

Criticality Studies With Plutonium Nitrate Solution in Slab Geometry; Effect of $^{240}$Pu on Criticality*

A number of experiments were performed in continuation of criticality studies for determining critical thicknesses of plutonium nitrate solution of high $^{240}$Pu content in slab geometry. This geometry is obtained by means of a variable thickness slab-shaped vessel of 42-in. dimensions; its thickness is set prior to beginning an experiment, and cannot be adjusted by any means during an experiment. Thus, the variable thickness slab is in all ways identical in terms of the criticality measurements to any single vessel that might be built with the same thickness.

* Work also funded in part by Division of Reactor Development under Applied and Reactor Physics.
<table>
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<th>Critical Slab Thickness, cm</th>
<th>Experimentally Derived</th>
<th>Computed</th>
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<td>18.1</td>
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<tr>
<td>Infinite Bare Slab (Corrected for room return and effect of vessel wall)</td>
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<td>21.1</td>
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<td>Infinite Water Reflected Slab (Not corrected for stainless steel in vessel wall)</td>
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<td>12.6</td>
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<tr>
<td>Infinite Water Reflected Slab (Corrected for effect of vessel wall)</td>
<td>11.7</td>
<td>11.7</td>
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</table>

The results are very good for the water reflected slab, but indicate difficulty in treating the effect of the stainless steel grid support structure on criticality in the case of the bare slab.

**Reflector Savings for Enriched Uranyl Nitrate Solutions**
(C. A. Rogers, Physics Research Department)

Further analyses were made of the data for determining the reflector savings of slightly enriched uranyl nitrate solutions. The reflector savings were obtained from critical experiments with a rectangular parallelepiped core (30.9 x 30.9 cm) of PuO$_2$-polystyrene having an H/Pu ratio of 15. The values given in the table were found from the change in core length on adding uranyl nitrate reflectors to the ends.
Summary

Instrumentation Development

Completion of operational tests on the system at the Hanford Plant. Progress on the circuitry detail final report.

Development of the beta monitoring additions to the ITT/FSS Monorail Laundry Monitor focused on the design of a supplementary hand-held detector for use with portable instrumentation.

NDT Assistance to Battelle-Northwest

Magnetic particle examinations have been completed on 227 parts, approximately 85% of the total circuits and were removed from the drawing board to be automatically tested for 131 tubes. Wall thickness measurements were taken and compared with previous measurements. A group of 196, 1/2 in. OD steam generator tubes identified as Data Set III in steam generator No. 4B have been eddy current tested. There were 28 tubes with indications that were either dents or OD indications.

Ultrasonic testing of numerous pressure vessels in the switching transformer, in all but 300 Area substations, has been completed. The vessels' wall thickness measurements were consistent with previous testing.

Radiation Protection

The effect of a proposed new security credential on the response of the Hanford film badge is negligible. Tests on the" 

Thermal Effects Studies

The annual survey of salmon spawning between Richland and Priest Rapids was begun. Early observations indicated fewer nests in the vicinity of Midway than in recent years.

The location or movements of 298 adult salmon and steelhead trout carrying conjugate tags were recorded in the reach of the Columbia River in the vicinity of the reactors. The program is now virtually completed for the year.

Adult coho salmon males were significantly less tolerant to temperatures in the range of 26°C to 30°C than were adult chinook salmon males.

More of the adult sockeye salmon that entered the Columbia River in June of 1968 had residual antibodies for columnaris disease than did similar fish in 1967. However, the incidence of infection at the spawning grounds was less.

The annual census of wintering waterfowl utilizing the Hanford Reach of the Columbia River was begun. Compared to one year ago, the current population of both ducks and geese is substantially greater.
TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Instrumentation Development

(E. M. Sheen/W. G. Spear, Instrument Research and Development Section)

Laundry Scaler Table

Progress toward completion of an instrumentation report continued with preparation of a line drawing of the circuitry for the new scaler tables designed for the ITT/FSS Laundry Facility.

Laundry Monorail Monitor for 147pm Detection

Development of the low energy beta monitoring additions to the ITT/FSS Monorail Laundry Monitor focused on the design and partial fabrication of a supplementary light weight (hand held) detection probe. This unit, which uses a single 5 inch photomultiplier tube, a thin scintillation beta detector, and a battery-powered preamplifier, will operate with a cable-connected, commercial count rate meter and high voltage supply instrument.

Progress on the instrument maintenance/instruction report for the modified monorail system centered on completion of several line drawings for the developed circuits and detector assemblies.

Assistance to Battelle-Northwest

(C. B. Shaw, NDT Customer Applications Section)

Magnetic particle examinations have been completed on 227 lifting devices as part of preventive maintenance. Included in this inspection were miscellaneous hooks, shackles, eyelets, turnbuckles, bolts and slings. Of the parts examined, three pieces contained cracks and were removed from service.

Pressure vessels (19 total) were ultrasonically tested for Battelle-Northwest Landlord as required for the established routine maintenance program. Buildings checked this quarter
The field evaluation of the thermoluminescent non-radiation worker dosimeter was completed. In general, this dosimeter was superior to film for personnel monitoring of the non-radiation worker group. For the audit dosimeters used in the field study, an accuracy of ± 20 percent to the 95 percent confidence level was achieved over the exposure range of 100 to 1000 mR. An angular dependence study was made and showed that the prototype TLD dosimeter is quite good. Regardless of energy above 40 keV, the angular dependence is within ± 50 percent. However, in actual practice, the effects of exposure at very acute angles will be much less since the bulk of exposure will be at angles approximately normal to the plane of the dosimeter.

By contrast, angular dependence studies run on the Hanford film badge dosimeter showed pronounced over-response for energies below a few hundred kilovolts at angles smaller than about 20° as referenced to the plane of the film. An over-response by a factor of 10 was noted when the badge was irradiated edge-on with 43 keV X-rays. The over-response can be corrected by placing a small piece of lead tape on the edge of the film badge. This reduces the angular dependence to within a factor of 2 from the normal response for the worst possible condition. As with the TLD dosimeter, in actual practice this is much greater than would be expected since the bulk of the exposure to the badge would be from angles reasonably close to normal. Plans are being made to add lead tape to all radiation worker film badges.

Lung counter examination data suggest a lung deposition of 10 to 15 nCi of insoluble uranium for a plant employee. Some of this was incurred from a recent incident; however, most of the deposition appears to have resulted from chronic intake prior to the incident. Urinary excretion data do not indicate that soluble uranium is of concern in this case. The case itself is unique in that a base line was established just prior to the incident when a lung examination was made following a three-weeks vacation. This provides an unusual opportunity to collect data to test current evaluation models. Additional internal examinations will continue provided the lung burden is measurable and the employee is withheld from work with uranium.
Continued evidence of increased river shoreline contamination was seen at locations below the plant boundary. The average weekly measurement of gamma exposure rates (by 40-liter ionization chamber) at Richland during October, 1968, was 60 µR/hr compared to 50 µR/hr during October, 1967. At Sacajawea Park, the average for October, 1968, was 43 µR/hr compared to 29 µR/hr for October, 1967. Current levels are higher than in 1965 at Sacajawea, but about the same at Richland. Similar increases were observed in surface GM measurements.

Dosimetry Technology
(C. E. Newton, Jr., Bioengineering Department)

Personnel Dosimetry for Non-Routinely Exposed Workers

Efforts have been directed toward resolving the difference in response of TLD and film from the recent field study concluded at ARHCO. Two sets of experimental dosimeters with film, TLD ribbon and TLD Teflon were forwarded to the radiation analyst at the ARHCO 234-5 Building. The Teflon was placed over the entire iron filter and over one-half of the tantalum filter. The ribbon was placed just behind the tantalum filter. Theoretically, the Teflon should have indicated the greatest response followed by the film and ribbon which should have shown a similar response for the high gamma component. The film results for both sets of data exceeded the TL values. The readings from the Teflon were about 50 percent less (which agreed with earlier field studies) and about one-third for the ribbon.

Following the above study, calibrated exposures were made with X-rays and natural sources to note the angular effect on TLD and film in the personnel dosimeter. Exposures were made at the normal and in 15° increments through 90° from the normal. Two sets of dosimeters were used for this study. The second set was modified to note what effect 0.0065 inches of lead tape would have when placed on the edge of the dosimeter. The following table presents the relative response of various energies and angles normalized to a radium gamma exposure:
The experiment showed that the electrometer tube drift, complexity, and expense of the Harshaw 2000-B automatic charge integrator is not necessary. The converter, a Hewlett Packard 2212A volt-to-frequency converter, followed by digital counters and control logic, would be more stable and more economical than the Harshaw integrator, especially if several TL dosimeters are read simultaneously to save time.

Analysis of the energy response data reported previously continued and preparation of a report on this phase of the program started. Variations in sensitivity from one batch of TLD-700 ribbon to another have increased for the last two groups of dosimeters ordered. A batch received in June was 20 percent less sensitive than earlier batches. A batch received in August was about 30 percent less sensitive. The manufacturer was contacted and has told us that a remeasurement of the sensitivities of the batches concerned showed only a 10 percent variation. Our reader system continued to show a 30 percent difference. These differences make it necessary to keep the batches separate.

Thermally Stimulated Exoelectron Emission

Thermally stimulated exoelectron emission (TSEE) detection instrumentation was gathered, built, modified and assembled. What appeared to be glow curves were recorded on electronic and ink types of X-Y recorders. As amazing and detailed as they are, they appear to be meaningless. The next step is to purge the counting chamber of air by using propane as a counting gas.

The TSEE collage includes a Harshaw TL reader with its phototube housing removed so that the planchet can be used to heat the TSEE material to a continually measured temperature. An end window GM tube with the end window pane removed is placed window down on the Harshaw planchet drawer. A special preamplifier was built to electronically quench the GM pulse and to fit in a light tight can over the GM. The preamplifier was later modified to permit GM tube voltages as high as 7500 volts. No working 3000 to 6000 volt supply was located, so a 2800 volt battery pack was placed in series with the 0-3000 volt supply
incorrect, and the programmers freely admit that poor fits can occur and that "proper" interpretation is necessary to distinguish the good fits from the bad ones. The program has not been closely checked to eliminate defects and minimize running time, and the results are critically dependent on choice of resolution.

**Thyroid Counter**

The coincidence shield for the low background thyroid monitor has been completed and tests will begin shortly.

A thyroid phantom which permits $^{131}$I in the thyroid and the depth of the thyroid to be varied was also fabricated.

**Thermal Effects Studies**

(R. F. Foster, R. E. Nakatani and W. L. Templeton, Ecosystems Department)

**Effects of Fluctuating Temperatures on Juvenile Salmon**

(P. A. Olson, Aquatic Ecology Section)

The laboratory at 1706-KE was set up to perform the scheduled test on the effects of oscillating temperatures on salmon. The test should start early next month with chinook salmon eggs to be obtained from fish holding facilities at Priest Rapids Dam.

**Salmon Redd Census**

(D. G. Watson, Aquatic Ecology Section)

Aerial surveys were made of the Columbia River between Richland and Priest Rapids on October 2, 14, and 21. One salmon nest was observed on the first survey, 25 on the second, and 274 on the third. Very little spawning was noted in the Midway area upstream from the Vernita bridge. In recent years, particularly since the establishment of Priest Rapids Dam, this area has been very heavily seeded. Much of the spawning in this region in past years has been in fairly deep water. High river levels during this year's surveys may be a limiting factor.
Temperature Tolerance of Adult Salmon
(C. C. Coutant, Aquatic Ecology Section)

Precocious chinook and coho salmon males have been tested in the laboratory for thermal resistance at temperatures of 26, 27, 28, 29, and 30°C. Coho are significantly less temperature tolerant than are chinooks at the temperatures tested, both from equilibrium loss times and death times. Fifty coho and thirty-nine chinooks were used in the experiments. Further work has been curtailed by lack of available fish.

Effects of Temperature on Fish Diseases
(M. P. Fujihara, Cellular and Molecular Biology Section)

The study of C. Columnaris antibody production and disease incidence among adult sockeye salmon during upstream migration was completed. The sockeye migration moves through the mainstem of the river during June and July and is exposed to warmer temperatures than the spring run of chinook salmon. The percentage of sockeyes sampled at Astoria that had agglutinating titers was 25 in 1968 compared with 2.5 percent in 1967. Heavy exposure to columnaris in the river occurred initially at Bonneville Dam during passage through the fish ladders. Although the adult sockeyes appeared to have started their upstream migration with a higher percentage of residual titers, the incidence of exposure and infection at the spawning grounds was less in 1968 than observed in 1967.

Population Dynamics of Hanford Wildfowl
(W. C. Hanson, Terrestrial Ecology Section)

The annual aerial census of wintering waterfowl utilizing the Hanford reach of the Columbia River was begun. Two weekly counts showed an increase of ducks from about 33,000 to 40,000 and a shift in the resting area utilization in response to hunting pressure. The Canada goose population decreased from 5,300 to 3,000 birds; McNary Wildlife Refuge reported a concurrent increase in geese, suggesting that a segment of the population moved there. Compared to one year ago, the current population of both ducks and geese is substantially greater,
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