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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
APRIL 1970
DIVISION OF PRODUCTION
AND
HANFORD PLANT ASSISTANCE
PROGRAMS
May 1970

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AEC RESEARCH & DEVELOPMENT REPORT

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Classified by G. E. King

C-65, Plutonium Production
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PACIFIC NORTHWEST LABORATORY

MONTHLY ACTIVITIES REPORT

APRIL 1970

Division of Production
and
Hanford Plant Assistance
Programs

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

May 1970

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By Jennie Maley, 2-17-94.
Verified By J. E. Savely, 2-23-94

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

ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Summary

Program I

The pickup and location of hydrogen in Zircaloy-2 samples exposed to reactor coolant in the rear shield area of KW while in galvanic contact with aluminum, anodized aluminum and stainless steel was found to be consistent with previous data of a similar nature. The rate of increase in base metal hydrogen was found to be dependent on the galvanically coupled material and the existence of a surface hydride layer.

Graphite samples from the GEH-13-418 capsule have been examined for dimensional and weight changes after receiving an additional exposure of 2.48×10^{21} nvt ($E > 0.18$ MeV). Turnaround from contraction to expansion occurred in certain samples.

Program III

Larger scale equipment was designed to permit retention studies when steam and krypton are released together.

An investigation of wind sensors used on the 100-N Tower indicates that intermittent errors in output may be ascribed to the sensor rather than to the time constant in the electronics.

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ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Program I - Basic Production

Low Temperature Hydriding of Zircaloy

(B. Griggs, Corrosion Research and Engineering Section)

Samples of K Reactor Zircaloy-2 tubing in several treated conditions were exposed for up to 14 months in galvanic contact with aluminum, anodized aluminum and stainless steel sample holders in the downstream portion of several K West reactor tubes. The objective of the experiment is to examine the hydrogen pickup and distribution in the Zircaloy samples. The samples included grit blasted; sanded (as received); case hydrided; case hydrided and stripped anodically in 3% HNO₃; case hydrided, stripped and etched in HNO₃-HF solution; and two samples of in service hydrided reactor tubing. The used reactor tubing samples had base metal hydrogen contents of 70 to 140 ppm while all the remaining samples contained base metal hydrogen in the range of 7 to 16 ppm.

Exposure of samples for 14 months to service conditions showed substantial (20 to 250 ppm) hydrogen pickup by samples in 6063 aluminum alloy holders, much less hydrogen pickup (1 to 20 ppm) by samples in anodized 6063 aluminum alloy holders and no hydrogen pickup by samples in 300 series stainless steel holders. Metallography of the samples qualitatively confirmed these analyses. Hydrogen pickup was qualitatively related to the extent of corrosion of the anodized holders. Hydrogen analyses of the samples after removing 5 to 8 mils from each surface (base metal) showed the samples in aluminum holders increased from 1 to 4 ppm/mo in hydrogen, samples in anodized holders increased from 0.3 to 2 ppm/mo and samples in stainless steel holders increased from 0.2 to 1.2 ppm/mo if they had surface hydrogen concentrations. Samples cut from used reactor tubing had much larger scatter of results both before and after test. In general, these scattered data do not conflict with the results obtained on the other samples. Several of the low base metal hydrogen samples were exposed in an argon atmosphere for 7 months at about 110°C and in static water for 5.5 months

orientation. While the transverse control samples show contractions that agree reasonably well with previously established dimensional change curves, the coated samples have contracted approximately twice as much as would be inferred from length change curves of parallel TSX graphite. This is difficult to understand, because silicon carbide expands under irradiation and its effect, if any, should be to retard the contraction of graphite. Sample ends on the coated graphites are poorly finished and this could contribute to some error in the length measurements. Measurements on the coated samples are, however, reproducible.

All samples were weighed before and after the GEH-13-418 capsule irradiation, and weight losses were small, generally <0.1%. The GEH-13-419 capsule is now under irradiation with the same sample loading, and will be discharged in April after accumulating an additional exposure of $\sim 3 \times 10^{21}$ nvt. At that point it appears that the only samples worthy of additional irradiation will be the TSX and TSGBF samples at 600-700°C. The remaining samples will have turned around and their expansion rates will have been established. It is also apparent that the silicon carbide coatings are not retarding the contraction of the graphite as was hoped.

In view of the fact that the N-Reactor graphite will likely operate at higher temperatures when the reactor is converted to power-only operation, it would be desirable to core several samples from the reactor stack and irradiate them at elevated temperatures. Future stack distortions under projected operating conditions could then be predicted from the results of the cored samples irradiations. These cored samples along with the 600-700°C TSX and TSGBF samples mentioned above could probably be accommodated in future instrumented capsule irradiations in the ETR.

Program III - Environmental and Regulatory Technology

Noble Gas-Water Partitioning Studies

(L. C. Schwendiman and J. Mishima, Air Pollution Chemistry Section)

A ten foot column and related equipment was designed and ordered fabricated. This column will permit experiments to be

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tendency toward localized corrosion seems to increase with decreasing molecular weight. If the system contains no CS-SS weld junctions, it would be much more economical to use NTA because the equivalent weight (200) is only half that of DTPA (400). NTA decontaminated carbon steel very effectively giving factors >100 in 2 hr. There seemed to be slightly more tendency for the films to spall rather than dissolve.

Corrosion of Inconel Heat Exchanger Tubing

(D. A. Jones, Corrosion Research and Engineering Section)

This study investigated the stress cracking of 304 stainless steel which has been observed in the N-Reactor steam generators, and also the possible cracking of Inconel used for retubing in these steam generators. It was found that sensitized 304 stainless steel, either from the steam generators or from typical commercial heats, will stress crack in water and steam at 170°C. Although failures were somewhat more frequent in the tubing from the steam generators, the investigation has demonstrated that the primary cause of failure is sensitization in either steam generator or externally purchased control heats of 304 stainless steel. The final report has been completed, and a draft submitted to DUN for approval.

ETOG Code

(K. B. Stewart, Theoretical and Neutron Physics)

The latest version of ETOG was imported and has been converted for use at Hanford. The current version has improved accuracy in the generation of resonance region cross sections and the elastic scattering (Pn) matrices. The improvement in accuracy results in increased execution time. Options generally used at Hanford are GAM I (modified to HRG) and MUFT 4 (modified for HAMMER). These options are local and are in the process of being included in the latest version of ETOG.

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ASSISTANCE TO ATLANTIC RICHFIELD HANFORD COMPANY

Summary

Program I

Further laboratory experiments with the slag and crucible dissolver solution nitric acid concentration measurement assembly, which uses sensors for determination of conductivity and specific gravity, confirmed system accuracies; planned tests will now be conducted under simulated process conditions at the 324 Building.

An extension of work reported earlier shows that addition of small concentrations of fluoride to Purex scrub solutions is beneficial in overcoming the deleterious effect of DBP on zirconium behavior.

Program II

Arrangements for the campaign reprocessing of irradiated neptunium target elements in the 325-A High Level Radio-chemistry Facility have been completed. Included for this purpose is a new dissolver (in C-Cell) and a new product isolation column (in A-Cell).

Program III

Several samples from the Rocky Flats fire were processed preliminary to determining the quantity of deposited plutonium.

Modifications to more accurately simulate ion movement have been made to equations in the Radionuclide Transport Model describing time-dependent movement and concentrations of macro-ions.

A study was made of the corrosiveness of Purex dissolver solution as a function of the Al/F mole ratio. The data indicate the $\text{Al}(\text{NO}_3)_3$ content of Purex dissolver solution may be decreased appreciably from the present flowsheet value with no serious adverse effect on dissolver corrosion.

Corrosion of 304L weldments exposed to sodium tungstophosphate solutions at 50°C was evaluated as a function of solution concentration. Concentrated solutions preferentially attacked the weld metal. However, no preferential attack occurred in concentrated solutions containing minor amounts of nitric acid.

Hot cell tests of cesium removal from Redox alkaline waste supernate were initiated during the month. Initial results compare favorably with prior laboratory data.

A hot cell test of sulfate precipitation purification of B Plant strontium product was completed. Analysis of the samples is in progress. Visual indications are that the test was quite successful.

Further laboratory studies of zeolite ion exchange purification of strontium have revealed a serious decrease in capacity with reuse of the exchanger.

In solid waste studies, bibliographies dating from 1960 were prepared on Refuse Disposal, Refuse Incinerators, and Industrial Wastes - Radioactive Materials. Source of the abstracts was the Engineering Index.

Calibration and testing of equipment to be used in the hot cells for thermal conductivity measurements on Tank 108 SX sludge were completed. Hot cell measurement of the thermal conductivity of actual 108 SX sludge will be started next month.

The following lettered items pertain to the Sr-Cs packaging program.

- A. Compatibility testing of SrF_2 has been extended to 6 months, with results generally in agreement with those

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than in basalt or sand because of salt's higher thermal conductivity and thermal diffusivity.

- I. The revolving tray, impact consolidation prototype has been completed. Twelve cans have been loaded with the new prototype.
- J. Thermal and mechanical testing of prototype cesium waste capsules was initiated.

Work performed this month strongly implicates dissolved solvent (TBP-NPH) as the material which poisoned the platinum catalyzed hydrazine reduction process.

A platinum catalyst was found to rapidly destroy hydrazoic acid, which would be present in the carbonate wash from the organic treatment system if hydrazine were used in the Purex 1B system.

Centrifugal contactor runs testing Purex 1B column hydrazine-stabilized U(IV) flowsheets were continued. The runs continue to be plagued with mechanical problems and excessive oxidation of the reducing agent. Design changes are being made which it is hoped will solve these problems.

Testing of a laboratory-scale, continuous-flow slurry reductor for preparing U(IV) by platinum-catalyzed reduction of uranyl nitrate with hydrogen was initiated. Preliminary results indicate that the concept, which involves continuous filtration of the U(IV) product, is sound but that the rates of U(IV) production are about 4-fold lower than those obtained in batch slurry reactors at ORNL.

Process Technology

Basic design for an on-tank neutron source-detector assembly for monitoring ^{239}Pu and ^{235}U in a Purex feed stream (tank D-5) is proposed. Further evaluations of concept validity and cost factors are continuing.

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Critical experiments and subcritical exponential experiments were continued for determining the effect of fixed neutron poisons (boron-containing steel and glass Raschig rings) on the criticality of plutonium nitrate solutions. Experiments were performed on a water-reflected 24-in. diameter

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ASSISTANCE TO ATLANTIC RICHFIELD HANFORD COMPANY

Program I - Basic Production

Acidity Sensor - Slag and Crucible Dissolver

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

Measurements of conductivity and specific gravity of the process stream should determine the concentration of the nitric acid solutions in the slag and crucible dissolver to an accuracy of $\pm 0.5M$.

Laboratory calibration and testing on the combined sensor assembly showed capability of measuring the specific gravity from 1.0 to 1.36 and the conductivity from 0.03 to 0.9 mhos/cm over the expected acidity range of 0.1M to 6.0M. Efforts during the reporting period centered on final preparation of the assembly and on fabricating associated acid-resistant piping and supports for on-line installation on a simulated process line. Based on the successful results to date in the laboratory, the system will be moved during the next reporting period to the 324 Building for additional tests under appropriate conditions.

Zirflex Tonic Effect

(J. L. Swanson, Applied Chemistry Section)

Earlier work demonstrated the effectiveness of small amounts of fluoride in the scrub solutions in increasing the Zr DF after the Zr was extracted from feeds containing uranium carbide reaction products (which make a portion of the Zr behave in an unscrubbable manner). Recent work has shown fluoride to be similarly effective when dibutyl phosphate (DBP) is the contaminant that perturbs the solvent extraction behavior of Zr. With $3 \times 10^{-4}M$ DBP in the organic phase, addition of $3 \times 10^{-4}M$ fluoride to each of four scrub solutions gave a tenfold increase in the Zr DF.

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A special device for resuspending plutonium-contaminated particles from the surface of samples was designed and fabricated. The device will be used to introduce particles into a cascade impactor for making size distribution measurements of the re-entrained particles.

Radionuclide Transport Model

(M. H. Karr, Earth Sciences Section)

Modifications were made to equations in that portion of the transport model describing time-dependent movement and concentrations of macro-ions. These modifications reflect recent developments in the soil-waste reaction investigations and should result in a more accurate simulation of ion movement. In particular, the change from linear chemical equations to a quadratic relationship has more closely simulated the reactions. The algorithm for computer solution of the macro-ion portion of the transport model was coded. This program is presently being debugged using data collected on Hanford soils in the soil-waste reaction investigations.

Coding of the modifications to the PST (Partially-Saturated Transient) flow model has been completed and debugging and testing are in progress. The modification should reduce the dependence of the solution convergence on the time step increment.

Field Measurement Methods

(M. H. Karr, Earth Sciences Section)

Consideration was given to methods of physically modeling unsaturated (partially saturated) flow of water through soil. The original proposal was to develop a system using two gamma sources of different energies which would enable monitoring of water content and bulk density with time. An alternate procedure is being considered which will measure water potential with time by measuring the electrical conductivity of a porous block embedded in the soil. The latter method is much less costly and may prove satisfactory for verification of the PST flow computer model. Transducers are being constructed and tests should be completed on the transducers by the end of May.

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function of solution concentration of Ca^{+2} , H^{+} , Na^{+} , and K^{+} . A preliminary experiment demonstrated that a small column technique would not be appropriate for the sorption measurement.

Program IV - Waste Management

Alpha Scintillating Glass

(O. H. Koski, Analytical Chemistry and Instrumentation Section)

The status of the sensor optimization study is as follows. A dozen sensors are to be optimized before program termination. Three are complete, eight are in process, and six were returned to the vendor for remanufacture. With anticipated yield through the process of seventy-five percent, the goal of 12 acceptable sensors is possible.

As a supplement to laboratory investigation of alpha measurement systems, a field study of three instrumentation loops was conducted in cooperation with ARHCO personnel. Both laboratory and plant tests showed that preamplifier input and output cable lengths and preamplifier selection might result in a factor of three variation in gain but were not significant with respect to the large difference between plant and laboratory observations.

Laboratory examination of plant stream samples which were taken at the approximate time of the investigation is expected to resolve the difference.

Treatment of Z-Plant Organic Waste

(R. E. Lerch, Applied Chemistry Section)

A report, BNWL-CC-2487, Regeneration of Saturation Carbon Beds by Gas Purging at Elevated Temperatures, was completed.

Hanford Z Plant Dissolver Studies

(J. Dunn, Chemical Engineering Section)

Dissolution improvements in treatment of incinerator ash for recovery of plutonium are being studied. Quantitative

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solution composition (initial) of $0.1M Na_3PW_{12}O_{40}$ - $0.25M HNO_3$. No preferential attack occurred.

Ion Exchange Removal of Cesium from Redox Alkaline Supernatant Solutions

(J. R. Lundquist, Applied Chemistry Section)

Redox waste alkaline supernatant (diluted 1 part to 2.4 parts water) was received during the month and studies are currently underway using this as a feed solution for loading Zeolon ion exchanger. Five percent breakthrough occurred at 28 column volumes using this feed, compared to 24 column volumes using a synthetic feed. Elution studies using the plant feed showed that 14 column volumes of eluant ($3.0M (NH_4)_2CO_3 + 2.0M NH_4OH$) were required to remove 95% of the cesium. Additional runs are in progress.

Purification of Strontium by Two Cycles of Sulfate Precipitation
(L. A. Bray, Applied Chemistry Section)

A demonstration of the purification of strontium was completed during the month using B-Plant solvent extraction product (5-1). The feed contained $0.05M Sr$ and $0.24M$ metal ion contaminants and had been produced using HEDTA as complexant (citric acid is normally used). The test consisted of two cycles of sulfate precipitation followed by carbonate metathesis. The strontium product was converted to fluoride and fired at $800^\circ C$. Strontium fluoride product samples have been submitted for spectrochemical analysis. Aqueous samples have also been submitted (ARHCO 222-S Analytical Laboratory) to determine the material balance of the overall test and to determine the decontamination factors for the metal ion impurities present in the feed. No physical or chemical behavior was noted that differed from the testing program with simulated solutions.

Purification of Strontium by Ion Exchange
(L. A. Bray, Applied Chemistry Section)

Additional laboratory studies have been completed to demonstrate the feasibility of purifying B-Plant solvent extraction

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90-95°C for 24 hours, the thermal conductivity was 0.486 BTU/(hr)(ft²)(°F/ft) at 25°C. After heating the sample to 160°C for 24 hours and cooling it to 25°C, the thermal conductivity was the same as the dried, finely ground material. Thermal conductivity measurements on TK-108 SX sludge will be started next month.

Strontium-Cesium Waste Packaging Program

The following tasks are part of an effort to provide a design basis for the Hanford Waste Management Plant, in which ¹³⁷Cs and ⁹⁰Sr will be consolidated in high integrity packages for long-term storage in water-cooled basins.

Compatibility Studies

(H. T. Fullam, Applied Chemistry Section)

The six-month tests of 316L stainless steel with non-radioactive SrF₂ have been completed and the couples evaluated. The results confirm those observed in the three month tests. Essentially, no attack was found in couples tested at 400°C. All of the couples tested at 800°C show attack and interaction zones of up to four mils were found in some couples.

Part of the six-month radioactive test couples have been evaluated. The complete results will be available shortly and will be reported in the next progress report.

Decontamination Studies

(H. T. Fullam, Applied Chemistry Section)

Work continues on the removal of SrF₂ from welded surfaces. Water spraying at 5000 psi using a garnet abrasive (-36 mesh) effectively reduced the contamination to a non-detectable level. However, the metal surface was badly pitted and eroded by the treatment. It is doubtful if such a process could be used in the packaging plant. Preliminary investigation of ultrasonic cleaning using 0.1M HNO₃-0.1M H₂BO₃ indicates that this cleaning procedure may have some promise for cleaning welded surfaces contaminated with SrF₂. The cleaning

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Cesium Ion Exchange

(G. Jansen, Chemical Engineering Section)

The 0.15M $(\text{NH}_4)_2\text{CO}_3$ -0.10M NH_4OH solutions used to scrub potassium and sodium from cesium during purification of cesium by inorganic cation exchange have a calculated initial pH of 9.4 with 42 percent of the ammonium ionized. As the solution passes down the column the ammonium ion is replaced by cesium, potassium, and sodium, and the pH can rise to a maximum of 13 because of replacement of the weak base ammonium by the strongly basic alkali ions. The increase in pH causes the ammonium ion to associate so that it does not remain an effective scrub. For example, if 60 percent of the total ammonium content of the solution is replaced by alkali ions, the pH is 10.54. Although the total ammonium content is 0.16M, only 5% is ionized so that the effective ammonium ion content for scrubbing is only 0.008 molar. To obtain the high observed scrubbing efficiencies the bulk of the scrubbing may be done by the cesium displaced near the top of the column. Because of the complex multi-ion interaction in the column the total effects of ammonium ion association cannot be determined until column breakthrough calculations, now underway, are completed.

CsCl Melt Cast Studies

(L. K. Mudge, Chemical Engineering Section)

A process for casting molten CsCl into a series of primary containers is being developed for use in the projected Hanford Waste Management Plant. Techniques for filling a series of containers have been developed, and the equipment is being tested to determine operating parameters and equipment corrosion performance.

Corrosion of the Hastelloy C-276 dryer-melter was determined ultrasonically after each run. After operation of the dryer-melter for about 130 hr for evaporation to dryness of a pH 4 CsCl solution (100 to 120°C) and for about 30 hr for melting the CsCl (600 to 720°C), corrosion of as much as 0.011 in. was measured in the area of the liquid-vapor interface. No corrosion of a 304 stainless steel vessel was detectable by this technique after a similar operating exposure.

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Additional Al_2O_3 trays ordered have not been received. The vendor is having difficulty in fabrication. Scheduled delivery is May 11, 1970.

Interim Basin Storage of Packaged Wastes
(G. Jansen, Chemical Engineering Section)

Heating experiments were conducted in a 1 ft wide by 4 ft long Lucite mockup of the waste capsule interim storage basin. The simulated strontium capsule was a steel can 16.5 in. long and 2.88 in. in diameter containing a 1 KW full-length heater surrounded by steel balls. The capsule was hung vertically in the center of the basin about 4 inches above the floor. The water inlet and outlet were directly opposite each other on the midplane of the capsule. The main water flow was across the capsule with a single large eddy circulating in the upper part of the basin. With a flow rate of 18.2 gpm/ft of basin width, the temperature rise at the midplane surface of the can was 4.6°C , and with 9.2 gpm/ft the temperature rise was 10.7°C . The temperatures at the ends of the can were appreciably lower than the midplane temperature. The effect of water depth on the temperature was less than 1°C in the range 36 inches to 72 inches. The maximum can surface temperature is expected to be on the vertical surface somewhat above the can midplane.

Direct Burial of Packaged Wastes
(G. Jansen and L. G. Niccoli, Chemical Engineering Section)

Calculations of maximum transient temperatures were made with FORTRAN V program DIBUW to include the situation of direct burial of radioactive wastes in salt. Temperatures were compared for salt (thermal conductivity = $2.5 \text{ Btu}/(\text{hr})(\text{ft}^2)(^\circ\text{F}/\text{ft})$), massive basalt ($0.65 \text{ Btu}/(\text{hr})(\text{ft}^2)(^\circ\text{F}/\text{ft})$) and basaltic sand ($0.35 \text{ Btu}/(\text{hr})(\text{ft}^2)(^\circ\text{F}/\text{ft})$). For example, a planar system of parallel, deep-buried 3 ft diameter tunnels spaced 25 ft apart and containing a single isotope with a half life of 30 years with a heat generation rate of $280 \text{ Btu}/(\text{hr})(\text{ft of length})$ would reach a maximum temperature rise of 185°F in 18 years in salt. The maximum temperature rise would be 533°F in 23 years in basalt, and 768°F in 27 years in basaltic sand.

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observed with the second sample of plant solution that contained poison.

Runs using a glass reactor failed to show any apparent reason for the poisoning--no precipitate was observed and the solution continued to wet the catalyst.

Saturation of dilute uranium solutions (like ICU) with 30% TBP-NPH prior to use as feed also led to poisoning of the catalyst. As with the other poison, catalytic activity was restored by washing with 2M HNO_3 .

Platinum Catalyzed Reduction of U(VI)
(G. L. Richardson, Chemical Engineering Section)

A laboratory-scale continuous flow slurry reductor is being evaluated for the preparation of U(IV) by platinum-catalyzed reduction of uranyl nitrate solutions with hydrogen. The reductor is made from a 600 ml gas wash bottle with a 10 micron glass-frit Buchner funnel installed on the side near the bottom for continuous filtration and removal of the U(IV) product by vacuum. The hydrogen is introduced through a 10 micron gas-diffuser frit near the bottom of the vessel and exhausted to atmosphere at the top. Agitation and heating are supplied by a magnetic stirrer-hot plate. A temperature well is inserted through the side to monitor the temperature (35°C in all tests to date). Feed is pumped in through a side inlet near the bottom.

A limited number of runs indicate that the concept is sound. An unknown amount of platinum deposited on the filter immediately after startup but there seemed to be no tendency for increased buildup with time. Most of the platinum appeared to remain in solution as expected. The rate of reduction (moles U(IV) produced per min per kg Pt) was somewhat lower than expected and was apparently affected by the concentration of platinum in the slurry and the solution residence time in the reactor. The maximum rate obtained with excess H_2 present was 0.66 moles U(IV)/(min) (kg Pt) at a Pt concentration of 5.4 g/l and a residence time of 37 min. The rate was decreased

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in the organic treatment system. A method of destroying the azide ion thus present in the carbonate wash is being sought. Recent experiments have disclosed a promising approach; on adding hydrazine and contacting with platinum catalyst, rapid reduction was achieved (at 50°C).

Alternative Purex Reductant - Uranium(IV)
(D. G. Bouse, Applied Chemistry Section)

Centrifugal contactor runs were continued to test flowsheets proposed to demonstrate the use of hydrazine-stabilized uranium(IV) as a reductant for plutonium in the Purex IBX (partitioning) column. Initial Purex plant testing of the uranium(IV) flowsheet is now in progress.

The last three centrifugal contactor demonstration runs were plagued with mechanical difficulties which resulted in higher than usual plutonium losses (2 to 6% versus < .5% in previous runs). Considerable aqueous entrainment in the organic product (IBU) was also encountered.

Oxidation of U(IV) has been greater in centrifugal contactor runs than was found in pulse column runs. A run was made to determine if Fe(II) was also oxidized to a greater extent in the centrifugal contactors. Analysis showed that only 13.4% of the Fe(II) introduced in the extractant (IBX) stream reports to the aqueous product (IBP) as Fe(II) vs. ~ 50% in the Purex plant. In a run with U(IV), no increase was noted in the U(IV) concentration of the end streams as a result of doubling the IBXU flow rate.

Hydrazine utilization is apparently greater in the centrifugal contactors than in the pulse column. With a feed containing .0001M HNO_2 , 27% of the initial N_2H_4 was found in the IBP. This percentage was found in a pulse column run with the feed containing .001M HNO_2 .

The two six-stage centrifugal contactors are being modified to accommodate a belt drive system and variable stirrer speed control. Attempts will also be made to exclude air from the units. Additional tests will be made when modifications are complete.

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operator, the source beam would be tilted down to permit balancing out the gamma-plus-stray-neutron net output, and up to produce the "fissile content" reading. A similar array mounted below the source could in like manner assay the "fissile content" near the bottom of the tank, indicating any enrichment or deficiency in that area. Possibly, the shape of the square wave leading edges might roughly indicate the ratio of ^{235}U to ^{239}Pu , owing to the greater yield of delayed neutrons from ^{235}U .

Consultation with physicists will be included in future studies to determine the least operable source intensity for this application, and to arrive at approximate cost estimates for such a source.

Organic Flowmeter Test

(O. H. Koski, Analytical Chemistry and Instrumentation Section)

Calibration checks of a one-inch thermal flowmeter show that short term repeatability is good but an apparent one-sided drift of the calibration occurred during the last month. The drift is characterized by a percentage change in flow, with the total shift during the month being on the order of ten percent. Since the thermal flowmeter is sensitive to changes in viscosity, thermal conductivity and heat capacity as well as surface conditions, the cause of the shift is unknown.

Converter Evaluation

(O. H. Koski, Analytical Chemistry and Instrumentation Section)

Evaluation of a commercial converter modified to function as a conventional plant rotameter readout has proceeded concurrently with the tests of other flow meters and has proven the conversion of rotameter flow to millivolt levels with accuracies of 1%. However, the adoption of the instrument to current and voltage values comparable with plant experience is required before process use is advisable. Also, the conversion of the output signal to levels suitable for process control instrumentation is desired. The stability of a commercially

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performed, a light leak was found which may have contributed to the cyclic drift which was observed during field testing of the original monitor last year. The monitor was also checked for instability with temperature change and no effects were observed.

The laboratory evaluation of the monitor is nearly complete, and the monitor is being prepared for field test. Field testing is planned for early May following the calibration of the field-prepared monitor.

Corrosion Studies

(R. F. Maness, Corrosion Research and Engineering Section)

Titanium specimens joined to 304L stainless steel by the DuPont Detabond process (about ten mils of tantalum used as an intermediate material) were exposed to boiling 65 wt% HNO_3 and to simulated Purex-type high-level waste solution for about 700 hours. Titanium (and Ta) are only attacked slightly in these solutions, whereas stainless steel corrodes at rates of about one and five mils/mo, respectively. No preferential attack has occurred to date in the bond area. The specimens will be subjected to a bond test and to a metallographic examination after another few hundred exposure hours.

Hydrazine-Stabilized Ferrous Nitrate

(R. E. Lerch, Applied Chemistry Section)

A report, BNWL-CC-2486, Iron(II) Nitrate: Its Preparation and Storage, is in publication.

Reprocessing ZPPR Scrap

(G. F. Schiefelbein, Applied Chemistry Section)

Studies of dissolution flowsheets for the recovery of burned ZPPR scrap (68% UO_2 -28.6% PuO_2 -3.4% MoO_3) were continued. Dissolution rate studies were conducted on Rocky Flats "fast" ZPPR scrap using a Plutonium Reclamation Facility (PRF) flowsheet (boiling 15.5M HNO_3 -0.65M HF). About 98% of the UO_2 and 85% of the PuO_2 present were dissolved after 15 minutes.

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Plutonium-Uranium Partitioning

(D. G. Bouse, Applied Chemistry Section)

Mixer settler runs were continued to test flowsheet modifications aimed at improved CC column partitioning of uranium from plutonium in the Plutonium Reclamation Facility (PRF). The successful coextraction of plutonium and uranium from ZPPR scrap solution was again demonstrated in a run made to produce feed for additional CC column studies. Three runs were made last month to test a proposed "top of the CC column" flowsheet. In these runs, the extraction column product (CAP) is introduced at the top of the column, a CCXF stream (1.2M hydroxylamine-0.15M HNO₃) is introduced six stages from the top, and a CCX stream (0.25M-0.15M HNO₃) is introduced at the bottom of the column. Uranium to plutonium ratios for the three runs were 2.5, 1.25, and 0.4, respectively. Uranium concentrations in the product from these runs were 9 g/l, 4 g/l, and 2.8 g/l, respectively. With the addition of a proposed mixer-settler unit in the PRF a uranium concentration of 4 g/l in the CC column product (CCP) is acceptable. Plutonium stripping was excellent in all runs.

Another top of the CC column run of the same type but with the hydroxylamine concentration of the CCX at 0.05M and the CCXF at 0.1M was completed. Analyses are pending. Additional CC column runs are in progress.

U(IV) Preparation for Purex Plant Test

(G. L. Richardson, Chemical Engineering Section)

A Purex Plant test of the use of U(IV) nitrate as plutonium reductant in the IBX (1st cycle partition) column has been completed with generally satisfactory results. The U(IV) was prepared in the 324 Building pilot plant facility by an aluminum reduction technique. Preparation of the first three batches was described last month. Only one of these was used; the first batch, prepared with hydrazine as holding reductant, was discarded and the third batch, after a long delay in getting the test underway, had very little U(IV) left in it and could not be used. Two additional batches were

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Soil-Chemical Parameters

(R. C. Routson and R. J. Serne, Water and Waste Management Section)

A study was continued to measure a series of soil-chemical parameters for a group of Hanford wells as a function of depth. Parameters which will be measured include: 1) moisture content-moisture potential relationships at 15, 1.5 and 0.33 bars tension; 2) pH in a saturated paste and in 0.01M CaCl_2 ; 3) cation exchange capacity (CEC); 4) mechanical analysis; 5) CaCO_3 content; 6) field moisture content; and 7) porosity. The CaCO_3 equivalent content as a function of depth for wells 1, 2, and 3 was reported.

Travel Time Calculations

(M. H. Karr, Earth Sciences Section)

Work began on digitizing the groundwater potential, transmissibility and aquifer bottom contour maps on a 1000 foot grid as part of an annual report of travel times from various facilities in the 200 Areas to the river. These periodic investigations will incorporate the latest improvements in potential maps, transmissibilities, and in calculation techniques.

Confined Aquifer (Surface)

(M. H. Karr, Earth Sciences Section)

Twelve wells that penetrate into the Beverly Member aquifers of the Ellensburg Formation were pump tested.

About half the wells tested showed evidence of a leaky aquifer. Geologic evidence also suggests this possibility. The sand-fill in some wells is permitting flow between aquifers, thus minimizing the value of some wells. The tests also are identifying wells with broken tubes and mislabeled tubes.

Tests to date consistently show that the upper part of the Beverly Member has a low transmissibility, and involves very low flow rates and small volumes of water.

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The hydroseismic recording system was maintained in continuous operation during the report period in well E33-12, north of B tank farm. The operation of the electronics was very satisfactory for the entire period and no maintenance other than changing charts was required. Numerous surface waves from teleseismic events (greater than 100 km distance) were observed routinely. This system is scheduled to be moved to well W11-1, 200-W Area.

A differential pressure detector has been designed and is now being built for installation on artesian wells. The purpose of this detector is to improve the sensitivity of the hydroseismic system to local seismic events by taking advantage of the shorter period pressure response.

Drilling - C Farm

(M. H. Karr, Earth Sciences Section)

High radiation levels of some of the drilling samples from a near surface leak at C tank farm required temporary suspension of drilling until better handling procedures could be established. New procedures were agreed upon with ARHCO monitoring, special handling equipment was obtained, and drilling is expected to resume this week.

Continuous Core - 116TX Tank

(M. H. Karr, Earth Sciences Section)

All of the equipment necessary to obtain a 20 ft continuous core of high level waste in the 116TX tank has been received from the manufacturer. Special handling and radiation control equipment required has been fabricated. Drilling is expected to start as soon as the work has been completed at C farm.

The Effect of Fixed and Soluble Poisons on the Criticality of Solutions*

(R. C. Lloyd, Critical Mass Physics Section)

Critical experiments and subcritical exponential experiments

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

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indicating k_{∞} to be less than unity. Preliminary values of bucklings are: $\sim -0.0032 \text{ cm}^{-2}$ at a concentration of 270 g Pu/l and $\sim -0.001 \text{ cm}^{-2}$ for the 400 g Pu/l solution.

The analyses of these recent data are under study and further experiments are under way.

Development and Modification of Computational Procedures
(L. E. Hansen, Critical Mass Physics Section)

A version of the KENO Monte Carlo criticality code that permits the use of different geometrical configurations within an array is now on hand from ORNL. This version of the code was modified to permit compilation under the new CSCX monitoring system on the UNIVAC 1108 computer. The code has also been successfully absolved, but no sample problems have as yet been run. Sample problem data are currently being compiled, however, and testing of the operational aspects of this version of the KENO code are proceeding.

This "mixed box" version significantly increases the flexibility of the geometry specifications that can be accepted by the existing versions of the KENO code, or more appropriately, by the BNW versions of the KENO code (i.e., SUKENO and SUKENO/ALBEDO). There will, however, still be restrictions on the geometrical arrays that can be specified. Perhaps the most stringent requirement will be that each box within the array must have a cuboidal outer boundary, and the exterior dimensions of each box must be identical.

The SUKENO code, and the version of SUKENO that was modified to permit the use of specular reflection at the exterior boundaries of arrays, SUKENO/ALBEDO, have been changed to read the necessary cross section data in the DTF format. This makes possible the use of existing DTF cross section decks, or data punched by the GAMTEC-II code, as input directly to the two versions of the SUKENO code. These data must not exceed 20 groups, with 6 downscatter groups including in-group scattering.

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BNL-1400

TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Summary

Radiation Protection

Special dose evaluations were made on a Hanford employee who was exposed to highly concentrated liquid waste. The Hanford Portable Radiation Monitoring Instrument Manual is nearing completion.

Environmental Evaluations

A duck killed on the 100-K trench on April 2 contained ^{32}P , ^{65}Zn , and ^{51}Cr concentrations of 0.04, 1.7, and 0.2%, respectively, of the concentrations found in the duck taken from this location on December 29, 1969. Seventy-two waterfowl, all somewhat contaminated externally and internally, have been collected from the B Swamp since the B-Plant strontium release. A deer shot near 100-N did not contain excessive radionuclide concentrations. On April 28, significant radiation levels were detected near the 100-N trench, which was found to be dry, the radiation source being the trench bottom which normally is shielded by about three feet of water. Analysis of the Columbia River above 100-F has been delayed due to the intensive waterfowl and mammal sampling effort.

Airborne radioactivity concentrations near the north fence of 200 East Area increased again. Regional fallout was slightly higher than one year ago. In general, environmental radioactivity measurements indicated no unusual sources of exposure to the population or environs.

Dosimetry Technology

A temperature of 60°C and a 3 to 5 day holding period appears to be most promising for quick annealing of the Basic Dosimeter. The prototype housing and dosimeter card are on order for the Multipurpose Dosimeter. Rough samples of the dosimeter card made of Cycloc plastic were received for

River temperatures increased to about 9.0°C in mid-April. New populations of larval midges developed in the three artificial channels held at 9.0, 11.5, and 14.0°C, but not in the warmer channels. The species composition differs from the populations established earlier during the winter. A simple apparatus for determining the upper incipient lethal limit of macroscopic river invertebrates was developed.

Waterfowl Census

The 21st Canada Goose nesting survey was begun. Initial population counts indicate a 25% decline in territorial pairs, with Locke Island nearly unproductive.

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BNW-1408

TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Radiation Protection

(H. V. Larson, Radiation Protection Department)

In cooperation with contractor personnel, special evaluations were made of the external radiation doses incurred by several employees of a Hanford contractor exposed to highly concentrated liquid waste (primarily $^{144}\text{Ce-Pr}$) while preparing to unload a bowling ball shipping cask. The initial evaluation indicated skin doses to parts of the hands in the range of 500 to 2000 rem for one employee. Whole body doses were well below prescribed limits, based on dosimeter results, the maximum measurement being 2.6 rads including 0.1R. The final dosimetry statement has not yet been prepared, pending additional measurements.

As a result of other reported radiation occurrences, 40 special bioassay samples were scheduled and 18 in-vivo counter examinations were made. An employee who incurred a Pu nasal contamination of 2000 d/m at a ruptured hood glove indicated an expected deposition of less than 10% of the MPBB. Other potential Pu intakes were significantly lower. Whole body counts for several employees who incurred beta gamma and nasal contamination up to 10,000 counts per minute in several different occurrences indicated that depositions were less than 1% of the MPBB for all detectable nuclides. One BNW employee incurred a localized skin dose of 1.8 rem while wearing a contaminated lab coat obtained from a bin of freshly laundered clothing.

Labels have been designed for use on all portable pool instruments leaving the calibration facility. The labels, on order, will provide a statement of the calibration date and place for the calibrator's initials and should serve to remind field personnel of calibration due dates as well as psychological uplift of attitudes towards instrument calibrations.

A purchase requisition has been written for approximately 20 beta-photon survey instruments to initiate the cutie pie replacement program.

turned over to BNW for analysis. Initial measurements indicated the bird to contain less radioactivity by at least a factor of 10 than the 100-K trench duck taken on December 29, 1969. Analysis of the muscle confirmed this, at least for the major radionuclide contributors to dose. The concentrations of ^{32}P , ^{65}Zn , and ^{51}Cr were 0.04%, 1.7%, and 0.2%, respectively, of the concentrations found in the December 29, 1969, duck.

Seventy-two waterfowl, primarily coot, were collected from B Swamp (216-B-3) between March 23 and April 21. The exterior, gizzard, gizzard contents, and, on some birds, the muscle were examined with a GM survey instrument for contamination. While thirteen waterfowl collected from B Swamp prior to the B Plant strontium release had no detectable external contamination, all collected since that release have had easily detectable contamination, both external and internal. Samples of selected B Swamp waterfowl collected both before and since the release are being analyzed for ^{90}Sr and gamma emitters. Concentrations of ^{90}Sr up to 20 pCi/g have been measured in muscle tissue. Consumption of one pound of muscle at this concentration would result in a bone (skeleton) dose less than 100 mrem. The dose from other radionuclides should be considerably less.

Samples of muscle, bone, liver, rumen, and thyroid from a deer collected on March 26 near 100-N did not contain unusual radionuclide concentrations. The thyroid contained 140 pCi/g of ^{131}I , probably resulting from drinking 100-N trench water. Except for this, all radionuclide concentrations were less than 10 pCi/g. The concentration of ^{239}Pu in the liver was less than 0.7 pCi/g.

While test well sampling in the vicinity of the 100-N trench on April 28, Environmental Monitoring personnel observed unusually high radiation background levels. They traced this background to its source, the N trench, which was discovered to contain no water. This situation was reported immediately to DUN Radiation Practices Engineering, and a fairly extensive survey was begun to determine if contamination had spread

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Studies continued with quick anneal procedures for the basic dosimeter. An annealing temperature of 60°C with a 3 to 5 day holding period continued to show promising results.

The order for the prototype housing, to be used with the multi-purpose dosimeter, was placed. Cyclocac samples were received and are being tested for identification punching.

A relative photon response curve was developed with TLD-700 ribbon behind 40 mils of tin. This could be a very useful tool for certain dosimetry purposes. The response indicated a very sharp cut-off between 90-100 keV and it essentially attenuates all the dose from energies less than 70 keV. Recent fast neutron dosimetry with TLD-600 and -700 ribbon was demonstrated with exposures from Pu fuel plates. Very good agreement was noted with the earlier results obtained using the TEPC. Scatter condition exposures were also made to note if routine calibrations would satisfy these conditions. Routine calibration from PuF_4 in a fast and moderated spectra was used for all evaluations and was found to be very satisfactory.

Several exposures to 1.38 MeV neutrons were conducted with a mockup of the neutron portion of the multipurpose dosimeter to determine the effects of the amount of moderator behind the dosimeter. Separate exposures were made with 1, 2, 3, 4, 6 and 8 inches of moderator. A size simulating the human trunk was chosen since most of the previous exposures were conducted on a polyethylene sphere about 9 inches in diameter. The 1.38 MeV neutrons closely approximate the average energy of PuF_4 neutrons, and the carbon target used to produce the neutrons also has a significant gamma yield. A buildup in both the gamma and neutron response was observed as the amount of moderator increased.

With one inch of moderator, the neutron response is 40 percent of the response with adequate moderator. The response is up to 85 percent with two inches of moderator and 92 percent with three inches. After four inches, no additional increase in response is observed. At the same time, the gamma

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the Richland Operations Office authorized a trial study. October 1967 was selected as the period of study. During this time, according to unclassified records, the river below the operating areas showed thermal modifications of from 1 to 3°C as a result of flow changes. The study is not complete because it was designed to make only a preliminary determination of the potential cost of raising the minimum flow below the Priest Rapids project by increments of 10 kft³/sec to as high as 66 kft³/sec, using power demands for the selected week. Such costs are interesting because they related to the continued dialog on similar Federal projects such as the Ben Franklin Dam, the Columbia River Navigation Extension project, and the siting of direct-cooled power reactors on the Hanford reservation.

Development work on dispersion models to simulate the reactor effluent mixing process was continued using a combination of analytical methods and field data. The optical imaging system has been used to produce real time information in both space and time using fluorescein dye as a tracer. This new information in combination with data previously collected in this program has been fitted to a Taylor type two-dimensional dispersion relationship using an adaptation of the LEARN-LIKELY code. In this manner values of the dispersion coefficients in the lateral and longitudinal direction can be accurately estimated from the field data on a point by point basis or on an averaged basis as the investigator requires. A universal model for the entire river reach from mile 380 down to mile 354 has not yet been achieved; some portions are better represented than others indicating that additional parameters must be included in order to obtain better overall representation.

Good progress has been made in the development of the mathematical representation of the transport of dissolved gas related to supersaturation in the Columbia River. A system of simultaneous differential equations has been developed and the physical programming of the test model is in progress. The system will represent gas transport rates as a function of sample depth for a range of bubble sizes. These in turn are related to the energy of the hydraulics in the dam tailrace.

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Thermal Effects Study
(W. L. Templeton, Ecosystems Department)

AEC-1, Performance of Thermally Shocked Young Salmonids
(M. J. Schneider, Aquatic Ecology Section)

The ability of juvenile rainbow trout, Salmo gairdneri, to escape predation following thermal shock continued to be examined. Juveniles were subjected to a thermal shock of 22°C for 100, 25 and 12 minutes and immediately placed in a tank containing adult predator fish, S. gairdneri. Twenty-five test fish and 25 control fish (not subject to thermal shock) were used in each test. The effects of shock are difficult to identify at this relatively low temperature. Previous studies have indicated that juveniles exposed to higher shock temperatures were more susceptible to predation. Statistical analysis will be necessary to identify the lower limit of temperature effect.

A series of 5 sham tests was performed in which the test fish were exposed to their acclimation temperature of 15°C. Following exposure, the fish were combined with an equal number of control animals, and the entire group was then exposed to predation. It appears that both groups were preyed upon with equal intensity.

The second group of prey fish is nearly depleted, and a new supply has been obtained. A total of 2,075 fish are currently being acclimated to 15°C. While this acclimation period is in progress, these fish will be cold branded to facilitate identification. Thermal resistance tests of the second group of prey fish were recently completed. This group of fish had experienced an infestation of parasitic monogenea (Gyrodactylus sp.) and may have been weakened. The thermal resistance studies indicated, however, that they were actually more resistant than the original group.

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of any gross external symptoms. Gas from blisters forming on the operculum and posterior to the eye has been analyzed, and the results are tabulated here.

<u>Sample</u>	<u>Fish #</u>	<u>O:N</u>
Air Standard		1:3.6
Blister, operculum	1	1:4.4
Blister, operculum	2	1:4.1
Blister, postorbital	1	1:7.2
Right eye	3	1:6.4
Left eye	3	1:9.0

This ratio of oxygen to nitrogen in the operculum blister is very nearly that of air. The ratio for gas behind the eyeball is relatively higher in nitrogen. As more afflicted fish become available, additional analyses will be run.

AEC-6, Hanford Chinook Salmon Population Studies
(D. G. Watson, Aquatic Ecology Section)

Preliminary draft of a summary report of the salmon surveys for the period 1947-69 has been completed. Statistical analyses of the survey data are being re-evaluated. This may suggest further analyses that may be appropriate to the explanation of population fluctuations in fall chinook salmon spawning in the Columbia River near Hanford.

AEC-8, Effect of Temperature on Fish Diseases
(M. P. Fujihara, Cellular and Molecular Biology, Biology Department)

Coarse fishes sampled at the Snake River, White Bluffs, Wenatchee and Keokuk sites were free of C. columnaris isolates, but antibody titers were observed in 38, 30, 43 and 57% of the fish respectively. Average titers were 1:18 at the Snake River and White Bluffs, and 1:28 at the Wenatchee and Keokuk sites. The higher percentage and magnitude of antibody titers in fish at the upper Columbia River sites were associated with the longer seasonal columnaris exposure at Wenatchee

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extensively upon periphyton. The species composition of the April midge populations appears to differ from populations which appeared in February and March (primarily Diamesa sp.), but individuals have not been studied taxonomically. Few planarians and Gammarus, organisms intolerant of cold temperatures, have appeared in the channels since last fall.

The initial 48 hr TM₅₀ tests with midge larvae (Diamesa sp.) were conducted with water from the channels, which permit a maximum upper limit of 10°C above ambient. A simple apparatus for exposing macroscopic river invertebrates to temperature increases up to 25°C above ambient was developed.

Since population levels of most invertebrates in the central Columbia are minimum during the winter, representative test organisms were sought elsewhere. Ostracods and mayfly nymphs, tentatively identified as Baetis bicaudatus, were collected from spring sources on the Snively Ranch and established temporarily in laboratory aquaria. At the present, one 48 hr TL₅₀ test with B. bicaudatus at temperatures of 9.0, 11.5 (control), 14.0, 16.5 and 19.0°C has been completed. Mortalities on these organisms are underway.

Waterfowl Census

(R. F. Foster, R. E. Nakatanti and T. P. O'Farrell,
Environmental and Life Sciences Division)

Population Dynamics of Hanford Wildfowl

(Wayne C. Hanson, Terrestrial Ecology Section)

The 21st Canada Goose nesting survey was begun and 115 nest histories initiated. About 190 territorial pairs were present within the study area, representing a 25% decrease in the population. Locke Island, which usually produces 35% of the nesting, was almost a total failure. This, presumably, resulted from absence of the 1967 age class of geese that would normally nest on the island this year. Coyotes destroyed most of this cohort three years ago.

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