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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
DECEMBER 1969
DIVISION OF PRODUCTION
AND
HANFORD PLANT ASSISTANCE
PROGRAMS

January 1970

AEC RESEARCH &
DEVELOPMENT REPORT

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Classified by G. E. King

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(Special Distribution)

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MONTHLY ACTIVITIES REPORT

DECEMBER 1969

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Division of Production
and
Hanford Plant Assistance
Programs

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

January 1970

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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

Corrosion tests on Inconel-mild steel couples and stainless steel tubing, exposed continuously to water at 165°C, have been completed, and the samples are being processed. Intermittent steam-water exposures are continuing.

A report on recent studies of aluminum erosion-corrosion has been completed and submitted for comment.

Final circuit corrections were made on the ET-12 eddy current clad thickness tester; the instrument now appears to have excellent stability.

Program II

Initial experiments demonstrated that 40% of ^{85}Kr dissolved in hot water would be released when the water was delivered under a low head to a second vessel. During quiescent cooling 25% to 43% of the gas remaining in the water escaped.

In meteorological studies, data processing proceeded in the study to define the frequency of occurrence of streamline patterns. Streamline and trajectory analysis examples were submitted to DUN.

ASSISTANCE TO DOUGLAS UNITED NUCLEAR

Program I - Basic Production

Corrosion of Inconel Heat Exchanger Tubing

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

Corrosion testing has been completed on Inconel-mild steel couples and stainless steel tubing samples in a steam heated (165°C) autoclave. All samples in this test were exposed continuously in the water phase. Samples from three discharges (after 2 week, 4 week and 8 week exposures) are currently being inspected for stress corrosion cracking and general corrosion. A second set of tests is underway in which the samples are being exposed intermittently to the steam and water phases at 165°C. These tests will be completed in about 7 weeks.

N-Reactor Simulation

(J. D. Lodge, Process Simulation and Analysis Section)

Subroutines have been written for the steam generator, dump **condensors** and surge tanks of the N-Reactor secondary. These subroutines account for 75% of the subroutines that will be used to convert the all analog simulation of the overall N-Reactor system to a hybrid simulation.

ET-12 Clad Thickness Tester

(T. J. David and C. B. Perry, NDT Systems Engineering Section)

Final circuit corrections were performed on the prototype ET-12 eddy current tester. This project had been temporarily suspended to reduce the rate of fund depletion. The final circuit configurations have resulted in apparently excellent tester stability. This will be verified during forthcoming production line qualification of the tester.

The ET-12 was designed for replacement of the UE-2B testers which have become maintenance problems and possess non-linear readout. The ET-12 is a 100% solid state tester

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of N Reactor were submitted to DUN.

Process Technology

Low Temperature Zircaloy Hydriding

(B. Griggs, Corrosion Research and Engineering Section)

The ability to remove a surface layer of Zircaloy-2 and zirconium hydride platelets from K-Reactor Zircaloy tubes after removing the continuous hydride case anodically in 3% HNO_3 has been demonstrated for short sections of tubing by anodic etching in 3% HNO_3 -0.8% NH_4F . A recent test of the process by DUN on a 5-1/2 foot section of tube removed from 3667-C showed complete removal of the surface hydride concentrations. At 65 inches from the downstream end of the tube, the total hydrogen concentration was 206 ppm and the base metal concentration 74 ppm. Hydrogen concentration in an adjacent ring treated by anodic etching was 77 ppm. Similarly, at 4 inches from the downstream end the anodic treated sample contained 96 ppm hydrogen while an adjacent sample with three more mils etched off contained 99 ppm.

The effect of temperature, velocity, metal hydrogen concentration and current density on the etch rate of the anodic etching process is being determined. Initial results show the higher voltage portion of the process is only slightly affected by temperature and velocity but strongly affected by current density. The low voltage part of the process is strongly affected by temperature and velocity and less by current density. The two temperatures being tested are 5-10°C and 25-30°C. The velocity effects were qualitatively tested by directing a 0.1 inch jet discharging at 30 ft/sec at the anodic surface from about 1/4 inch. A 30 minute treatment with the high (10 to 50 volt) voltage process produced no visible effect while a subsequent 5 minute treatment at low voltage (1 to 2 volts) produced visible craters 0.1 to 3 mils deep.

The initial portion of the high voltage process is used to penetrate the anodic oxide left from the anodic 3% HNO_3 treatment. It was found that this process will also effectively

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decontaminated over a period of 2 hr at 85°C with a flow rate of 50 gal/min (~ 2.5 ft/sec). The activity was apparently all removed after 42 min at temperature. The solution readings were: at 12 min, 80% of maximum; at 42 min, 100%; at 72 min, 100%; at 102 min, 99.7%; and at 132 min, 94%. The low reading for the last sample indicates some redeposition. Samples taken to evaluate redeposition read ~ 1000 C/min for a 25 cm^2 sample surface which corresponds closely to 0.04% of the initial activity level of the spacer.

A more valid experiment would include provisions for feed and bleed essentially in the same ratio as N Reactor. The loop is being modified to provide this capability.

Coating Studies on TSX Graphite

(R. C. Giberson, Graphite and Polymers Section)

Samples of TSX Graphite coated by DUN have been furnished to BNW by DUN for oxidation testing.

The first set of samples was flat plates $3/16$ in. x 1 in. x 2 in. The samples were coated at a temperature of 545-550°C using hexamethyldisilane (HMDS). The helium flow rate was $300 \text{ cm}^3/\text{min}$. Total exposure time was 24 hours. The partial pressure of the HMDS was about 36 mm. Sample 2 had a net weight change of +0.0007 grams during the coating run and Sample 5 had a net weight change of -0.0009 grams during the coating run. It is, therefore, concluded that the samples underwent some oxidation during the coating process. Unfortunately, the size of the samples furnished precluded oxidation on a recording balance. Instead, both samples, along with a non-coated sample, were placed in a furnace and oxidized simultaneously, with the rate being determined by "before and after" weight measurements. The oxidation conditions were helium flow, $500 \text{ cm}^3/\text{min}$; H_2O partial pressure, 25 mm of Hg; graphite sample temperature, $890 \pm 10^\circ\text{C}$. The reaction rates are shown in the table which follows.

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1. 300 energy points are allowed in File 3.
2. Inelastic scattering cross sections are given in File 3,
 $MT = 5, 6, 7, \dots$
3. Energy dependence reduced neutron widths are given
in File 2.

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BNWL-12

ASSISTANCE TO ATLANTIC RICHFIELD HANFORD COMPANY

Summary

Program I

Partial assembly, in the laboratory, of the conductivity sensor portion of the nitric acid concentration measurement system, as being developed for use with slag and crucible dissolvers, allowed initial tests aimed at establishing sensor characteristics.

The effectiveness of complexants on the solvent extraction behavior of Zr from feeds containing uranium carbide reaction products is under study. Fluoride was found more effective than oxalate. Digestion of the carbide feed with H_2O_2 was also helpful in improving Zr DF.

The solid state spectrometer for use in monitoring Np and Pu in Purex HAW was completed. Its energy resolution at 18 keV is about 600 eV, not sufficient to resolve Np and Pu X-ray lines. Hence, prior separation of Np and Pu is required. Radioisotope sources being investigated for production of fluorescence X-rays are very weak compared to X-ray tubes. A 0.25 g ^{241}Am source produced fluorescence intensities weaker by a factor of 250 than produced by an X-ray tube. Minimum measurable amounts of U and heavier elements, using an X-ray tube source, were found to be about 25 $\mu g/ml$ in solutions ($10^{-4}M$) or 10 μg collected on a filter paper.

Program III

Detailed engineering design was ordered on the 242-B Aerosol Release Facility. Techniques for analyzing specimen samples received from the Rocky Flats Plant fire were studied to insure obtaining the most significant data.

The computer algorithm for solving the equations describing transient flow of water through partially-saturated soil is now working.

C-3, unlike Zeolon, was degraded by repeated use.

The regeneration of organic-saturated carbon beds is under experimental investigation. Regeneration appears to be a temperature dependent desorption process rather than oxidation-desorption. Some loss of capacity is observed on successive cycles of absorption and regeneration.

The corrosiveness of fluoride-bearing Purex high-level waste solutions was investigated using Zr(IV) rather than Al(III) to complex the fluoride ion present. The Zr/F mole ratio needed for effective corrosion inhibition was about 0.8. However, low 304L corrosion rates of one mil/month could be obtained in these aluminum(III)-free solutions only by also reducing the Fe(III) content to about 20 percent of the flow-sheet value.

Zirconyl nitrate was prepared from simulated Zirflex decladding solution by precipitating $Zr(OH)_4$ with KOH solution. The precipitate was filtered, washed and dissolved in nitric acid. Corrosion tests indicated product purity was poor. A similar product was obtained when NH_4OH rather than KOH was used to precipitate the $Zr(OH)_4$.

Weldments prepared by joining 304L to mild steel with 308L filler metal were exposed to "standard" and to Zirflex decladding solution and to neutralized Purex IWW solution, all at 50°C and as a function of pH (neutral to alkaline). No attack, general or preferential, occurred on any of the test specimens during a 1500 hour exposure.

A temperature compensation circuit designed for an electrodeless conductivity system is composed of a resistive temperature transducer, a resistance bridge amplifier, a function generator, a summing amplifier and a multiplier operating in the division mode. Output data include process temperature and compensated and non-compensated electrolytic conductivity.

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is being assembled. Waste packages filled with simulated material are being prepared for evaluation with the equipment.

- G. A computer program is being written for the determination of thermal conditions around strontium and cesium waste packages buried directly in the soil. The output will help determine when it is thermally feasible to store these isotopic sources in the ground.
- H. A circular feeding mechanism was designed for SrF_2 encapsulation; it will require much less space than the previous straight design.

Minimization of the amount of ammonia formed during the catalyzed reduction of U(VI) to U(IV) is under study. Results are currently somewhat ambiguous.

Centrifugal contactor runs continued aimed at testing use of hydrazine-stabilized uranium(IV) nitrate in the Purex IBX column. Control runs with ferrous sulfamate reductant duplicated plant experience very closely. Results of the U(IV) runs have not been fully resolved.

Process Technology

Experimental simulation of the top portion of the Plutonium Reclamation Facility CC column was successfully completed. Gas formation in the column was not found to be excessive.

Purification of current B-Plant strontium product to the degree desirable for encapsulation is under study. A precipitation process involving a sulfate product precipitation followed by a hydroxide by-product precipitation appears promising. Elements which must be removed include Ca, Mg, Cd, Mn, and Al.

Miniature centrifugal contactor studies of Pu stripping from 20% TBP- CCl_4 indicated successful operation at 75 ml/min (combined aqueous and organic) but flooding at 150 ml/min. It is hoped to partition Pu from U by co-extracting followed by preferential stripping of the Pu.

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A study was continued to measure a series of soil-chemical parameters for a group of Hanford wells.

All well logs have been examined to determine the bottom of the unconfined aquifer. Aquifer thickness may now be estimated.

The small diameter pump for use in piezometer tubes has been completed and is ready for field tests.

Construction of a thallium source shield is underway as part of the deuterium field test. A small diameter pump for sampling groundwater has been completed and is ready for field testing.

Extensive piezometer measurements have been made to help identify aquifer permeability and transmissibility. These have been correlated with geologic evidence.

Well 40-62 was pump-tested and is being used to relate the geophysical response of the water potential surface to permeability.

Six wells south of the 200-E Area were subjected to continuous neutron logging as part of a study related to soil moisture and percolation of rainfall.

A method was devised to install thermal conductivity probes in high level waste tanks to assist in the study of in-tank solidification.

Further experiments were undertaken for establishing the effect of fixed poison (boron-containing stainless steel Raschig rings) on the criticality of plutonium nitrate solution. Critical approach and exponential experiments were performed on an 18-in. diameter water-reflected cylindrical vessel containing stainless steel Raschig rings. The results confirm previous measurements made on a smaller vessel in that a large negative value for the buckling ($k_{\infty} < \text{unity}$) of the 275 g Pu/liter solution with Raschig rings was obtained. This

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Program I - Basic Production

Slag and Crucible Dissolver Acidity Sensor

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

Laboratory tests evidenced feasibility for measuring the nitric acid concentration of solutions in the slag and crucible dissolver using a combination of conductivity and specific gravity determinations. This approach should achieve the desired acid concentration accuracy of $\pm 0.5M$.

Partial assembly of the conductivity sensor allowed preliminary investigation of the response characteristics of this portion of the instrument. Receipt of special Teflon seals should allow completion of fabrication of the conductivity sensor and final assembly of the combined measurement system. Arrangements made for experimental installation at a 300 Area location will enable tests aimed at establishing the expected performance of the system.

Zirflex Tonic Effect

(J. L. Swanson, Applied Chemistry Section)

The effect of complexants other than fluoride on the solvent extraction behavior of Zr from feed solutions containing uranium carbide reaction products is under study. Addition of $0.007M$ oxalate to the feed solution resulted in a 14-fold increase in the extraction Zr DF but little or no effect on the subsequent scrub DF. In contrast, addition of $0.003M$ fluoride to the feed resulted in little or no effect on the extraction Zr DF but a greater than 20-fold increase in the scrub DF. When the complexant was added to each of the scrub solutions (but not the feed), a 20-fold increase in Zr DF was obtained with $0.001M$ fluoride while only a 2-fold increase was obtained with $0.001M$ oxalate.

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newest SiLi dodes on the market are claimed to have a resolution at the iron line of just under 300 eV. If the resolution varied with energy in proportion to the diodes we have, a new one should be expected to have a resolution of about 375 eV or less at 18 keV and thus be able to resolve Pu and Np lines. However, even with better resolution it is likely that Pu and Np must be separated prior to measurement with a SiLi spectrometer since plutonium is usually present at higher concentrations than Np.

A radioisotope source has been designed, utilizing 200 Ci of ^{147}Pm and a thin transmission foil of Sn for maximum excitation of both Pu and Np or a foil of Ag for discrimination in favor of Np. This source has not been fabricated pending tests with a less efficient ^{241}Am source now in hand. Fluorescence intensity measurements were made to determine the best orientation and best locations of source, sample, and detector, and a special holder with titanium tube collimators and lead shielding was built to hold both solid and liquid samples.

Preliminary measurements were made on $4.2 \times 10^{-3}\text{M}$ solutions of Th and U, elements similar in fluorescence properties to Np and Pu. The measurements were made with the ^{241}Am source-SiLi spectrometer system and also with an XRD-5 X-ray spectrometer. Fluorescence intensities were about 250 times greater with the XRD-5 system than with the ^{241}Am -SiLi system. Precision of the measurements was about $\pm 10\%$ with the ^{241}Am -SiLi system and $\pm 1\%$ with the XRD-5. Minimum detectable amount of uranium and other heavy elements with the X-ray source was determined to be about 25 $\mu\text{g/ml}$ in solutions and 10 μg on the surface of a small millipore filter. Minimum detection limits for the ^{241}Am -SiLi system have not been determined. Interest remains in the source-SiLi spectrometer system because it has a lower background than the X-ray spectrometer for U, Np and Pu peaks.

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system is to be used to make laboratory determinations of the hydraulic characteristics of soils and to provide laboratory data for verification of both the PST Model and the diffusion equation.

A series of instruments has been assembled consisting of thermocouple psychrometers and diode thermometers. The psychrometers are being used to determine the energy profile in the unsaturated soils on the Hanford Project. These measurements will provide invaluable information in assessing the extent of natural rainfall penetration.

All work on deep percolation of rainfall was concerned with the new well #12-B, located about 100 feet north of well #12-A. Soil samples were taken for tritium content analysis and duplicate water content measurements were made.

Conventional soil psychrometers at 5-foot intervals and soil temperature sensors at 10-foot intervals (except the first 10 feet which are 1, 2, 5, 7.5 and 10 feet) were connected into 2 multiple-conductor cables. The initial plan was to pull the well casing a small distance so the soil from that particular depth would fill back into the well. By repeating this operation, the well would be filled with soil psychrometer and temperature sensors at various depths.

However, considerable difficulty was encountered in attempts to pull the casing. It was finally decided to remove the instruments from well #12-B and use it as a monitoring well. A new well #2-B will be drilled near well #2-A. Then an attempt will be made to bury the sensors in the new well in the same manner as planned for well #12-B.

Soil-Waste Interrelationship

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

A two-level factorial design experiment was continued to measure the sorption of ^{134}Cs on a Burbank sand subsoil as a function of solution pH, (Ca^{++}) , (K^{+}) , and (Na^{+}) . Initial attempts to measure ^{134}Cs sorption on small soil columns proved unsuccessful due to the failure of the small columns

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*Computer Data Display System

(R. T. Jaske, Water Resources Systems Section)

A display editor was developed for the PDP-9/UNIVAC display. Basically it allows the user to edit machine language text via the Univac. Its major advantage is that the user can view 20 lines of text simultaneously.

The disc interface neared completion. Remaining tests include checkout of error detection circuit and write lock-out. Software development will be greatly enhanced through the use of the disc by eliminating large amounts of tape manipulations.

Program IV - Waste Management

Treatment of Z-Plant Organic Waste

(R. E. Lerch, Applied Chemistry Section)

A literature search on regeneration of organic saturated carbon beds revealed that passage of superheated steam through the bed was the most common method of regeneration. Passage of superheated (600°C-800°C) steam through a saturated bed results in essentially complete regeneration of the carbon. Possible regeneration of carbon beds saturated with TBP (tributyl **phosphate**)-CCl₄ or DBBP (dibutylbutyl **phosphonate**)-CCl₄ by means of superheated steam will be investigated if the necessary equipment can be assembled.

Regeneration of organic saturated carbon beds (TBP, DBBP, CCl₄) by air purging at elevated temperature is currently under investigation. Preliminary laboratory results indicate that the degree of regeneration goes through a maximum (80-95% of starting capacity) between 200°C and 300°C for both TBP-CCl₄ and DBBP-CCl₄ saturated beds. Below 200°C regeneration of the carbon is incomplete. Above 300°C degradation (i.e., burning or charring) of the carbon occurs and the reloading capacity drops off. Preliminary investigations using argon instead of air as the purge gas gave comparable results. This would seem to indicate that regeneration is

* In part also charged to the AEC Division of Reactor Development and Technology.

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with the phosphorus peaks making both the quantitative and qualitative interpretation of the chromatogram uncertain. The specificity of this detector for phosphorus compounds will alleviate this problem and produce more consistent results.

Derivatives of DBP - Many efforts have been made to produce a volatile derivative of DBP so that gas chromatographic techniques can be used for its evaluation. Efforts to make a **silyl** ether derivative have failed. Attempts will now be made to prepare the methyl ester derivative using diazomethane. The procedure that will be employed will closely follow that reported in ORNL-TM-2289.

Gas Chromatography-Mass Spectroscopy - The Perkin-Elmer GC-MS combination Model 270 has now been received on site. It is expected that the equipment will be in operation by the end of January. This should be of great assistance in identifying many organic decomposition products formed from chemical and radiation degradation processes. The only requirements that the compound must have for examination by this technique are volatility at 250-300°C and a molecular weight not exceeding 3000 mass units. Some information may be obtainable from polymers having molecular weights greater than that specified, and from non-volatile materials by using pyrolytic techniques coupled with the GC-MS combination. It is hoped that many compounds that have not yet been identified will be identified by this new technique.

Ion Exchange Removal of Cesium from Normal and Enriched Fuel Wastes

(J. A. Partridge, Applied Chemistry Section)

Cesium elution tests were conducted with the ABEDEM Ti-A resin comparing 5M NH_4NO_3 , 5M NaNO_3 , and 5M $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ as eluants. The results showed that NH_4^+ ion is superior to Na^+ ion as a Cs eluant with this resin.

In the first of these tests, Cs was loaded onto a 20 ml resin bed. The feed was simulated PAW-ZAW (see report for November, 1969). After the column had been washed with 1M

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4.5 (0.4M HDEHP-0.3M TBP-NPH), gave a Cd E_a° of 2.4. Therefore, Cd extracts almost as well as Sr with the present solvent extraction flowsheet and little purification would result from further cycles of solvent extraction.

A purification flowsheet is under development which involves sulfate precipitation, metathesis of the sulfate to carbonate, nitric acid dissolution of the carbonate, and final purification by hydroxide precipitation. Removal of the major impurities by sulfate precipitation is possible because of the high solubility of the Mn, Mg, and Cd sulfates. No carrier (Pb or rare earth element) is required to precipitate 95-99% of the Sr because of the high Sr feed concentration (ca 0.1M). With a fourfold dilution of the initial feed, sulfate precipitation (pH 1, 1M SO_4^{2-} , $< 8^\circ C$) will also provide a calcium DF of 7.

Because an additional DF from calcium may still be required, further purification of the strontium product has been considered using hydroxide precipitation.

Based on the relative solubilities of $Sr(OH)_2$ and $Ca(OH)_2$ in water, it appeared probable that the $Ca(OH)_2$ could be precipitated leaving high purity strontium in solution. To test this method, nitrate solutions containing Ca^{++} and Sr^{++} at a mole ratio Sr/Ca of 10 were made alkaline with NaOH. Strontium to calcium ratios in solution were measured as a function of pH and temperature. Acceptable mole ratios of strontium to calcium could be obtained at any temperature, but the highest purity strontium was obtained at elevated temperatures. Because strontium will precipitate at a high pH, careful pH control is necessary. It appears best to adjust the pH at room temperature, heat the solution to boiling and then cool. Several runs were made which would be typical of a plant process. Two of these are described.

A solution containing 0.50M Sr^{++} and .05M Ca^{++} was adjusted to pH 12.7, heated to boiling and cooled to room temperature. The supernatant solution contained 0.497M Sr^{++} and 0.00521M Ca^{++} . The strontium loss was less than 1%. Another solution containing 0.30M Sr^{++} and .030M Ca^{++} was

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A failure occurred in a line carrying Purex coating removal solution at or near a weld joining 304L stainless steel to mild steel pipe. To assist in evaluating the failure, weldments prepared by joining 304L to mild steel with 308L filler metal were exposed to aluminum and to zirconium coating removal solutions and to neutralized Purex IWW solution all at 50°C and as a function of pH (neutral to alkaline). No attack, general or preferential, occurred on any of the test specimens during a 1500 hour exposure period.

Electrodeless Conductivity Measurement

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

Temperature compensation for an electrodeless conductivity system is under development. The circuit involves two amplifiers and a multiplier operating in a division mode. One amplifier converts the resistance thermometer reading to a suitable voltage analog. A function generator on the amplifier output which is connected to the second amplifier is constructed so as to apply the first and second order temperature correction terms. The multiplier-divider combines the temperature terms with the uncorrected conductivity to provide an output referenced to 25°C. Process information available will then be temperature, uncompensated conductivity, and compensated conductivity.

Strontium Purification by Ion Exchange

(L. L. Ames and J. G. Adams, Water and Waste Management Section)

The two-level factorial design to study the probable interactions in a nitrilotriacetic acid (NTA)-sodium gluconate-current acid waste (CAW) system was completed. A statistical analysis of the dependent variables was undertaken which showed that, using column volumes to 10% Sr breakthrough, pH was significant at the 99% confidence level. The NTA and Na-gluconate concentrations were shown to be significant at the 95% confidence level. A comparison of the Sr loading curves of the pH 8 solutions with the loading curves of the pH 6 solutions indicates that, for this specific design, the

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B. SrF₂ Process Studies

(L. K. Mudge, Chemical Engineering Section)

Work has continued on flowsheet development for the conversion of a strontium nitrate solution to SrF₂ that is suitable for encapsulation in high integrity waste packages. The pH of the strontium nitrate feed may not affect the SrF₂ sintered density as was reported in November. Varying the feed pH in recent runs had no effect on the sintered density of the SrF₂. The recovery of Sr remained the same over a feed pH range of from 3 to 7.

Filtration rates were determined as a function of SrF₂ cake thickness. Additional runs at different pressure drops will be made to determine the constants in filtration correlations given in the literature.

■ Cesium Ion Exchange

(G. Jansen, Chemical Engineering Section)

Design parameters are being calculated for scaleup of laboratory experiments in the purification of cesium by cation exchange. A fixed bed of the inorganic exchanger, Zeolon H, is first loaded with cesium from feed solution, then scrubbed with 0.15M NH₄NO₃-0.15M NH₄OH and finally eluted with concentrated NH₄NO₃.

The total capacity of Zeolon H in contact with a feed of composition 0.1M CsNO₃-0.3M KNO₃-0.7M NaNO₃ is estimated at 2.57 meg/gram of water-washed air-dried exchanger. A selectivity of 23 for cesium over sodium and 4.3 for cesium over potassium was estimated for this feed composition although it is a strong function of cesium loading.

Decontamination factors during the loading step were estimated assuming equilibrium performance of the column. As the cesium loading was varied from 0.25 meg/gram (22% column utilization) to 1.16 meg/gram (100% utilization), the potassium decontamination factor increased from 1.7 to 4.3 and the sodium decontamination factor increased from

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waste package array. Time periods are chosen such that the major temperature peak for the point in question is included. The program will include capability to handle an array of 24 packages with up to 10 different isotopes in each package. Three subroutines have been written and debugged. Development of the main program is in progress.

F. Strontium Fluoride Densification

(K. R. Sump, Materials Development Section)

A circular feeding mechanism was designed incorporating most of the features of the previous straight design. The primary advantage is space saving. The 5 compartment tray has an OD of 13 in. and an ID of 8 in. Only four of the five compartments are loaded because one is always in the unloading position above the container.

The hopper and boat holder are designed to handle a 5 in. wide, 7 in. long, 2-1/2 in. high boat containing ~800 g of SrF_2 . The boat is slid horizontally into the boat holder. The assembly is rotated less than 180° during unloading and then returned. The tray is rotated by turning a drive rod in the center.

The above circular feeder design has been chosen as the best and detail drafting will be done followed by fabrication.

Platinum Catalyzed Hydrazine Reductions

(J. L. Swanson, Applied Chemistry Section)

Means of minimizing the amount of ammonia formed during the catalyzed hydrazine reduction of U(VI) to U(IV) are under study. One of the major problems has been the analysis for ammonia in these hydrazine-containing solutions. Results have been dependent on the oxidant employed to destroy the hydrazine; the reasons for this are not completely understood.

Alternative Purex Reductant - U(IV)

(D. G. Bouse, Applied Chemistry Section)

Centrifugal contactor runs were continued to test flowsheets

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Tests run with 20% TBP-CCl₄ organic and 0.2M HNO₃ aqueous show that a throughput of 150 ml/min (combined aqueous and organic) to be excessive for adequate phase separation in the laboratory centrifugal contactor which will be used. A flow rate of 75 ml/min (combined aqueous and organic) produced good separation and will be used in a stripping run.

Effect of Flocculants on Centrifuged Phosphotungstic Acid (PTA) Precipitates

(L. A. Bray, Applied Chemistry Section)

Tests have been completed using Magnafloc (990-N) and Purafloc (Dow-N-11) to increase the settling rate of Cs-PTA precipitates. Increased digestion temperatures were also tested to increase the particle size. Heating the feed (current acid waste) to ~50°C prior to and during the PTA digestion cycle gave the most beneficial results. When heated, the Cs-PTA settled completely in two hours as compared to ~12 hours at 30°C. No increase in cesium loss was detected at 60°C as compared to 30°C. Magnafloc (990-N) and Purafloc (Dow-N-11) are comparable flocculants. However, precipitated N-11 appeared to dissolve more readily in 2M NaOH. A 1 ppm addition of flocculant slightly increased the Cs-PTA settling rate. A 10 ppm addition of flocculant was comparable to digesting the solution at 60°C for two hours. Plant tests are in progress using the heating procedure.

Plutonium Reclamation Facility CC Column Studies

(E. J. Wheelwright, Applied Chemistry Section)

Experimental simulation of the top portion of the Plutonium Reclamation Facility CC Column was completed. The results can be summarized as follows:

- 1) Gas formation in the column is not excessive and should not disrupt operation when the nitric acid concentration is kept reasonably low (1.5M or less in the aqueous).

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produce any marked change up to 0.85M acid. A **stepwise** change occurred when the meter was exposed to concentrated acid. Recovery to the original conditions required several hours. The meter accuracy changed about eight percent in a manner related presumably to the wetting characteristics of the meter surfaces and not to the bulk properties of the fluid.

The change which occurs **in** the transition range from viscous to turbulent flow is abrupt with 25 .percent change in flow meter output occurring with a 5 percent change in flow rate.

Purex 2AF Stream Nitrite Monitor

(R. D. Dierks, Analytical Chemistry & Instrumentation Section)

A duPont 400 process photometer was received from the **manufacturer** at month end. The instrument was designed to monitor the nitrogen dioxide concentration in the gas phase of the Purex 2AF stream sampler to provide an index of the nitrite ion concentration in the 2AF solution. The instrument will be tested in a laboratory 2AF sampler **mockup** to assure its operation prior to plant installation and to prove the operation of the phase separator system.

A design for a stainless steel housing that will attach to the access door of the sampler pit (J-5-B) and completely contain the **duPont** photometer sensor, has been provided ARHCO personnel for implementation.

Ferrous Iron Monitor

(A. L. Jackson, Analytical Chemistry & Instrumentation Section)

The colorimetric reagent 2,4,6-tripyridyl-s-triazine (TPTZ) for the ferrous iron monitor is only slightly soluble in water and only marginally soluble in practical organic solvent systems. One marginal system, the ethylene glycol-water system, which had not worked on previous tests was re-examined and found to be satisfactory under certain practical conditions.

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with a jackleg between the column and the feed tank. The pump is self-priming, meets the head and flow characteristics required and does not introduce air into the CX Column when the flow is interrupted on the suction side of the pump.

Dissolver Filters

Removal of undissolved particulate matter from the effluent from the continuous dissolvers is necessary to promote process continuity and improve product recovery. Parallel filters have been designed and are being tested for their ability to handle a minimum of one full shift (8 hours) production without replacement. Two different porosities of all-polypropylene sock-type filters have been successfully demonstrated and further tests of other grade filter socks are planned.

Radioiodine Trapping

(L. C. Schwendiman and L. A. du Plessis, Air Pollution Chemistry Section)

This study was brought to a close and a document, L. A. duPlessis, Some Observations Concerning the Chemistry of the Purex Vessel Vent Silver Reactor, BNWL-CC-2378, was written.

Soil-Chemical Parameters

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

A study was continued to measure a series of soil-chemical parameters for a group of Hanford wells. The cation exchange capacity was measured for Well #1 and a portion of Well #2 as a function of depth.

Calculation of Transmissibility

(M. H. Karr, Earth Sciences Section)

Verification of the calculated transmissibility for the Hanford groundwater flow system is still being delayed due to difficulties in procuring the graphical digitizer.

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Transmissibility Measurements

(M. H. Karr, Earth Sciences Section)

One new well was cleaned and pump tested. This well, 40-62, is situated next to the BX telephone exchange between the 200-E and 200-W Areas. It is planned that this well will be used for further experiments to relate the geophysical response of the water potential surface to permeability.

A well pumping system was built from excess equipment that has been only slightly used and is in excellent condition. This system will provide the capability of running pump tests at almost any time without having to obtain the services of an outside contractor.

Field Measurements

(M. H. Karr, Earth Sciences Section)

Six of the twelve wells drilled by ARHCO south of the 200-E Area as part of a special study were logged with the continuous neutron logger. Well #12 was logged twice and the new well to be used for soil moisture measurements was logged prior to installation of special sensing devices. The results of logging on well #12 appear to be in excellent agreement with moisture content measurements taken from individual samples in the laboratory.

Measurements were also made in well #12 with an interval neutron logger. These measurements also were in good agreement with the other two methods. Because of the time involved in making the measurements, and because no increase in accuracy could be noted, this method was suspended.

There are still some problems with the logging instruments which necessitate almost constant maintenance in order to keep the system operable.

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larger vessel also give a large negative value for the buckling ($k_{\infty} < \text{unity}$) of the solution with Raschig rings and confirm the previous measurements. The plutonium solution is now being concentrated up to about 400 g Pu/liter to examine the effectiveness of these rings at higher concentrations or for changes in moderation. This vessel will then be replaced with a 2 ft diameter vessel containing borosilicate glass Raschig rings. Glass rings of two different sizes and several boron contents are available for these experiments. Data need to be obtained on self-shielding effects of the boron and the effectiveness of different ring sizes for different concentrations examined.

HFN Code Modifications

(R. D. Johnson, Critical Mass Physics Section)

Modifications have been completed to the HFN multi-group diffusion theory code. The results have been coded as a new version of HFN which is more accurate, more complete, and with somewhat less running time than the original version. When final checkout procedures have been completed, the code will be released for common usage in criticality calculations.

SUKENO Code Testing

(R. B. Kidman, Theoretical and Neutron Physics Section)

The SUKENO code was tested on the uranium dioxide sphere problem. Thus far, the results indicate that it is the input cross sections to SUKENO that have been causing unrealistic multiplication factors. For example, the 100 cm radius uranium dioxide sphere was calculated in three ways:

1. GAMTEC was carefully set up to generate proper cross sections which SUKENO subsequently used.
2. A preliminary set of Hansen-Roach cross sections was used by SUKENO.

The current 26-group cross section library used by W. W. Little was used in a 1DX run. The resulting multiplication factors were 1.08, 0.74, and 0.7, respectively.

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TECHNICAL ASSISTANCE TO THE HANFORD PROJECT

Summary

Radiation Protection

A much improved portable radiation monitoring low energy beta measuring instrument was assembled and tested. Several cases of medically-administered tracer materials have interfered with evaluations of potential internal depositions from Hanford sources.

Environmental Evaluations

Airborne radioactivity from fallout decreased somewhat from the last reporting period. Environmental radioactivity measurements indicated no unusual sources of exposure to the population or environs.

Columbia River Studies

Computer runs using the DISPERS version of the LEARN-LIKELY code continued. Dye tests were carried out to collect information on vertical mixing in the Columbia River between 100-F and the Hanford town site. Studies continued to eliminate the discrepancy between computed and observed temperatures between Umatilla and Warrendale for 1969 conditions.

Thermal Effects Study

Juvenile rainbow trout acclimated to 15°C require about one hour exposure to 26°C sublethal temperatures to exhibit significantly poorer escape performance during predation by larger fishes.

Active feeding by gulls below the thermal discharge at K Reactor diminished significantly during the month of December. No juvenile salmonids were found within stomach contents of sacrificed birds or were collected in the traveling scoop trap.

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TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Radiation Protection

(H. V. Larson, Radiation Protection Department)

The problem of low energy beta monitoring with standard portable radiation monitoring pool instruments appears to have largely been resolved by completion of a technical breakthrough and administrative adjustments. Currently, a pancake shaped thin mica window Geiger tube is used for field monitoring of low energy betas. However, this tube has numerous disadvantages including relatively low sensitivity, 3 to 6 month life-time, and high susceptibility to breakage. Although the repair of these detectors has been reduced to \$75 from \$125, the problem of cost is still very acute because of the high breakage. Several prototype low energy beta detectors were made using a thin NE-102 scintillator, optically coupled to a standard 6199 photomultiplier tube. The prototype units have vastly improved sensitivity, are far more rugged, and, in addition, cost only about one-third that of a new pancake GM tube. A meeting was held with Hanford contractors on December 23 to determine what course of action should be followed with respect to the Pool. Specific items discussed were this prototype probe, calibration and dissemination to the field of existing pancake probes, and procurement costs of new pancake probes. It was agreed that ordering of new pancake probes would be limited pending field evaluation of the prototype NE-102 detector. Furthermore, it was agreed that External Dose Evaluation would assume the responsibility for procurement, maintenance, and calibration of all pancake tubes now in service and that such calibration would be done according to a new system whereby both detector and count rate meter are individually calibrated.

Lung counter examination results for a Hanford contractor employee following exposure to airborne plutonium were abnormal. Unusual photopeaks, indicating the presence of a radionuclide which was not readily identifiable, interfered with the evaluation of the lung burden which might have been incurred from the incident. The responsible radionuclide was subsequently determined to be residual traces of ^{57}Co labeled vitamin B-12,

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Special external exposure evaluations were performed for 127 Hanford contractor employees, primarily as a result of requests by contractor radiation protection representatives. There were no exposures in excess of the applicable limits.

Environmental Evaluations

(W. L. Fisher, Environmental Evaluations Section)

Regional fallout levels decreased somewhat since the last reporting period and were about the same as those a year ago. The average airborne concentration of beta activity at the perimeter communities was 0.11 pCi/m^3 . This radioactivity appears to be unrelated to Hanford operations.

Airborne radioactivity concentrations near the center of the 200 East Area north fence continue to be somewhat higher than at other onsite sampling locations. After peaking at 11 pCi/m^3 during the week ending November 11, the average beta concentration remained steady during the next two weeks and then decreased to 2.3 pCi/m^3 during the week ending December 2. More recent data are not yet available.

A study of the Radiological Considerations of Opening the Columbia River for Recreational Purposes--Ringold to 100-F Area was published as BNWL-CC-2363.

In general, environmental levels of radioactivity were consistent with previous experience and indicated no unusual sources of exposure to the population or environs.

Columbia River Studies

(R. F. Foster, Environmental and Life Sciences Division)

Effects of Reactor Effluent on the Quality of Columbia River Water

(R. T. Jaske, Water Resources Systems Section)

Computer runs using the DISPERS version of LEARN-LIKELY continue. At the present time examination of data collected between 100-B and Ringold and 100-D and Ringold are being

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population groups will be included in the survey. These will be seafood production employees and their families, probably including the maximum consumers of seafood, and the students at the local rural high school. The potential size of these groups could exceed 400 and plans will be laid to permit actual surveys of from 200-300 people. The suggested location for the whole body center is the school grounds near Rockaway, Oregon. A coordinated public relations program will be an important part of the study.

Thermal Effects Study

(W. L. Templeton, Ecosystems Department)

AEC-1, Performance of Thermally Shocked Young Salmonids
(C. C. Coutant, Aquatic Ecology Section)

Tests continued to determine relative escape performance of 15°C acclimated juvenile rainbow trout from predation by adult rainbow trout. The rate of testing was reduced due to seasonally large size of the available prey and by erratic feeding performance of the predators. Tests at 26°C sublethal temperatures were conducted in December with thermal exposures of 16 min (4 tests), 32 min (2 tests) and 64 min (2 tests). At this time, it appears that exposures of at least 64 min duration are required at 26°C to increase vulnerability of shocked fish to predation.

AEC-4, Effect of Thermal Shock from Effluent Discharge on Young Salmonids

(J. A. Strand, C. D. Becker and C. C. Coutant, Aquatic Ecology Section)

Feeding activity by gulls below the thermal outfall of K Reactor diminished significantly with only one instance of typical feeding occurring on December 14. On that date, approximately twenty-four birds were observed circling the plume and periodically diving onto surface waters to obtain forage material. The prey species was not identified. On all other dates, less than four birds were seen in the vicinity.

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lower river temperatures. Columnaris exposure and infection at temperatures below 50°F have not previously been observed in the Columbia River.

Furunculosis exposure, infection and antibody production at White Bluffs was 37, 7 and 76% and 23, 4 and 92%, respectively, as compared to 33, 10 and 75% at the Snake River site and 39, 12 and 71% at the Wenatchee site. A decrease in exposure and infection was observed at all sites. The data, however, suggest a lag period between reduction in exposure of fish and a drop in antibody production.

Dermocystidium Disease - Dermocystidium infection was observed in 42% of 50 spring-run Chinook salmon mortalities examined at Wells Artificial Spawning Channel. In vitro culturing of the **pathogen** has been unsuccessful; however, studies will be initiated to transmit the disease through subcutaneous and intraperitoneal injection.

AEC-9. Effects of Increased Temperatures on Survival and Growth of Juvenile Steelhead
(P. A. Olson, Aquatic Ecology Section)

Juvenile steelhead exposed to approximate Columbia River temperatures and added increments of 3, 4 and 5°F since early July are now experiencing the normal decreasing seasonal temperatures near 53°F in the warmest lots. No mortalities have occurred since mid-November. While past mortality appeared to be entirely from columnaris disease, its occurrence was not correlated with relative temperature conditions. Mean mortalities for each temperature increment range from a minimum of 3.3% in the 5°F increment to a maximum of 14.7% in the 3°F increment. High temperature increments are favoring more rapid growth during this period of lower temperature.

AEC-10, Temperature Tolerance of Aquatic Insects
(C. D. Becker, Aquatic Ecology Section)

Base river temperatures in the control channel decreased from 12 to 9°C from November 20 to December 20. Temperatures in

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