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AEC Research and Development Report

PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
NOVEMBER 1972

Division of Production and Materials
Management and Hanford Plant
Assistance Programs

DECEMBER 1972

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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
NOVEMBER 1972

Division of Production and Materials Management
and
Hanford Plant Assistance
Programs

by the
Staff of Battelle-Northwest
R. S. Paul, Director

December 1972

BATTELLE
PACIFIC NORTHWEST LABORATORIES
RICHLAND, WASHINGTON 99352

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PACIFIC NORTHWEST LABORATORY
MONTHLY ACTIVITIES REPORT
DIVISION OF PRODUCTION AND MATERIALS MANAGEMENT
AND HANFORD PLANT ASSISTANCE PROGRAMS
ASSISTANCE TO ATLANTIC RICHFIELD HANFORD COMPANY

Summary

Program II

In studies on reducing the concentration of dibutyl phosphate (DBP) in degraded tributyl phosphate (TBP) extractant current efforts are on eliminating the use of ether in the diazomethane process for methylating DBP.

Program III

Documentation of the results of the "Radionuclides in Soils" program continued and is progressing as scheduled.

Monitoring of the lysimeters and instrumented wells continued.

Results from the verification of the Variable Thickness Transient (VTT) Model indicate that the transmissivity is the most sensitive input parameter and that the calculated transmissivity distribution should be improved in areas of poor model performance, although model performance is good over much of the project.

Documentation of the transport model was continued.

Program IV

Neutralization of the caustic in "salt cake" wastes with phosphoric inhibits the corrosion of mild steel by the solidified waste at temperatures of about 80°C.

A study was initiated to define the process parameters which affect the degree to which barium impurity can be removed from strontium during strontium fluoride precipitation.

Process Technology

Separations and waste processes instrumentation studies included development work on in-line gamma analysis, in-line alpha analysis, pH control of cesium carbonate neutralization, flowmetering applications to waste processes and suspended solids analysis in waste processing streams.

In Plutonium Reclamation Facility instrumentation studies, utilization of scrap Kynar in a moulding process has been successful in the production of gamma analyzer cell parts with good surface finish and low background buildup.

Flowsheet modifications were developed to reduce the amount of Na_2SO_4 required for sulfate precipitation purification of strontium solution obtained by solvent extraction treatment of Purex Acidified Sludge (PAS). Also, reuse of rare earth element carrier used in carrier precipitation recovery of strontium in the waste solutions from the above process was successfully shown in laboratory studies.

Work was directed toward the eventual augmentation of wind measuring systems on the Hanford Reservation.

The installation of the particulate air sampling system at the U-Pond area neared completion. Equipment is being assembled for erecting 2 cfm air samplers along the east side (B-C Zone 1).

A rough draft has been prepared of the document covering the finding from the study on the fractional release and size distribution of particles airborne during the burning of flammable materials containing uranium compounds. Work is in progress on the document, "Some Experimental Measurements of Airborne Uranium (Representing Plutonium) in Transportation Accidents."

Filter and cascade impactor samples of the particles in the 234-5Z Building stack are underway and are scheduled for removal at the end of this month.

Operation of all local seismic systems and maintenance of the United States Geological Survey micro earthquake network were continued.

ASSISTANCE TO ATLANTIC RICHFIELD HANFORD COMPANYProgram II - Plutonium Scrap ProcessingSolvent Extraction Studies

(E. C. Martin, Applied Chemistry Section)

Treatment of degraded tributyl phosphate (TBP)-diluent extractants with diazomethane has been shown to reduce the concentration of DBP. Diazomethane is normally generated in an ethereal solution. Safety aspects of applying such treatment in plant operations would be much improved if the ether solvent could be exchanged for a nonflammable solvent such as carbon tetrachloride. The difficulty is to separate a very low boiling material, diazomethane (bp -23°C) from a low boiling material, ether (bp 35°C). The isolation of pure diazomethane involves considerable risk since this material is unstable. Attempts to generate diazomethane from its precursory N-methyl-N-nitroso-p-toluenesulfonamide in a CCl_4 solvent have not been successful. Diazomethane reacts rapidly with the codistilled water and the yield of the desired product is negligible.

As a means of circumventing these problems, laboratory apparatus has been designed in which a condenser capable of using ice water is so situated as to cause ether to reflux but permit diazomethane to remain in the vapor phase and distill over to a receiver. On the opposite side of the apparatus, an air-cooled condensing column permits the distillation of CCl_4 . These vapors will condense on a cold finger where they will pick up the diazomethane vapor thus reducing the likelihood of spontaneous decomposition. The apparatus has been constructed and testing is scheduled for December.

Program III - Environmental and Regulatory TechnologyRadionuclide Transport Model

(D. B. Cearlock, Water Resources Systems Section)

A second rerun of the Variable Thickness Transient (VTT) simulation for the test period 1968 through 1971 using a modified storage coefficient distribution was completed. There was minor improvement in

the match between observed and predicted groundwater elevations. In the areas of poorest model performance, the storage coefficient values are at their reasonable limits. Thus no further improvement is possible through iterative adjustment of this parameter. While the match between predicted and observed groundwater elevations is good over much of the project, the results indicate that the transmissivity is the most sensitive input parameter and that in the areas of poor model performance, the calculated transmissivity distribution should be improved.

Neutron logging of the lysimeter tubes was done at mid-month. Data collection from Wells 2D and 2C is continuing on a routine basis.

The major effort was directed toward documenting the results of the "Radionuclides in Soils" research program. First drafts of most of the twenty-two documents were completed.

Soil-Waste Reactions in the Transport Model

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

Documentation of the transport model was continued. The documentation is presently divided into four anticipated documents, which will be entitled: (1) One-Dimensional Model of the Movement of Solutes through Soil Columns: The PERCOL Model; (2) PERCOL Model User's Manual; (3) Experimental Support Studies for the PERCOL and Transport Models; and (4) Laboratory and Computer Methods for the PERCOL Model Verification. Effort during the month was directed to writing the rough drafts of all documents. Documents (2) and (4) have been completed and are in internal review. Documents (1) and (3) are 65% and 80% complete, respectively.

Program IV - Waste Management

Soil Decontamination

(J. L. Ryan, Applied Chemistry Section)

A study of soil decontamination is being initiated. Preliminary efforts will concentrate on characterization of plutonium species by a study of the literature and correlating observed behavior of plutonium on soils with known chemical behavior in macro concentrations.

Corrosion Studies

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

The corrosiveness of "salt cake" prepared from simulated alkaline Purex high-level waste solution to mild steel is being determined as a function of composition, temperature and water content. Four compositions are being evaluated; two have caustic contents of 400 and 200 g/l; in the other two the caustic has been neutralized with phosphoric acid. Temperatures evaluated are 60, 80 and 95°C; water levels are about 6 and 15 percent. Preliminary results (2 months exposure at 80°C) indicate essentially no corrosion of specimens exposed to salt cake neutralized with phosphoric acid, while erratic results (0.2 and 2.0 mils/mo) were obtained in unneutralized waste. A complete series of specimens will be evaluated after six exposure months. As evidenced by the generation of ammonia, the primary reaction in the case of unneutralized waste appears to be

The containers are being vented periodically and water added as required.

Barium Decontamination During Strontium Fluoride Precipitation

(J. A. Partridge, Chemical Development Section)

The waste management program at Hanford calls for encapsulation and storage of strontium as strontium fluoride. Prior to conversion to the fluoride, the strontium is purified by a sulfate precipitation. Data show that the barium concentration in the strontium product after the sulfate precipitation can be as much as 10 percent of that of the strontium. It therefore appears desirable to reduce this barium concentration prior to encapsulation and thus reduce the volume of fluoride product to be stored. One possible point in the process where barium decontamination could be achieved is during the fluoride precipitation.

Solubility product data indicate that if there are no carrier precipitation effects, one should be able to achieve separation of barium from strontium, calcium and magnesium during a fluoride precipitation by proper adjustment of feed concentration and excess fluoride addition.

The objective of this study will be to determine if the barium decontamination factor is controlled simply by the solubility products or if carrier precipitation effects are also present, and to what extent process parameters such as temperature, pH, digestion time, etc., affect the barium decontamination.

Process Technology

Instrumentation Assistance to Separations and Waste Processes (O. H. Koski, Chemical Development Section)

Effort during the month included development work on in-line gamma analysis, in-line alpha analysis, pH control of cesium carbonate neutralization, flowmetering applications to waste processes and suspended solids analysis in the waste processing streams.

Tests on stream samples of Purex HAP, 2AW, 2BP and 1BP with a Ge(Li) detector multichannel analyzer system demonstrated that only the 2BP stream can be analyzed for gamma activity easily directly without the application of additional signal processing techniques.

Silicon surface barrier detectors were tested for possible use as in-line alpha detectors and with the addition of lithium and boron foil as neutron detectors. Although successful for both alpha and neutron detection in the dry state, manufacturing changes to allow exposure to nitric acid are necessary for in-line applications. Beta discrimination remained comparable to the diffused junction type reported previously.

Gamma radiation exposures of three test types of pH electrodes have reached 1.5×10^9 , 1×10^9 and 3×10^8 respectively with two failures by bursting noted on the least exposed type. Cable failures in the other two have terminated the testing.

A flowmeter for a stream carrying 30% solids at a flow rate of 14000 gal/min is being considered. Installation must be made without disturbing the piping previously installed. Under consideration are clamp-on ultrasonic, thermal and strain gage-target types. Although the target and thermal types will give only point-wise velocities requiring

profile velocity information to accurately measure flow, the ultrasonic type is not expected to operate well due to the solids content of the stream.

In-line measurement of percent solids has been demonstrated to be possible through the measurement of electrolytic conductivity using an electrodeless conductivity cell in-line. The solids being of low conductivity alter the effective cell constant. A change in conductivity from 145 to 32 millimhos resulted as percent crystalline NaNO_3 increased from 0 to 58 percent on a volume percent basis.

Instrumentation Studies in Support of the Plutonium Reclamation Facility
(O. H. Koski, Chemical Development Section)

Studies included ElW stream analyzer development and problems associated with in-canyon monitoring and in-tank inventory assay.

Moulding of scrap Kynar into cell parts suitable for monitoring the ElW stream has been successfully performed with sufficient parts manufactured for three cells. Inner cell wall finish is superior to machined parts so that low background buildup and ease of decontamination is expected. Fabrication of the lead shielding for the cell is about 70 percent complete.

Studies relating to the change in the gamma spectra of various sources when viewed through an aperture through lead shielding as projected for use in the in-canyon monitoring system were performed. Typically the effect of the aperture is to increase the low energy portion of the spectra. For canyon monitoring and in-tank assay only higher energy gamma is expected to be useful for assay. However, low-level discrimination will be required to prevent overloading of the telemetry system.

Strontium Purification - Sulfate Precipitation Flowsheet Development
(L. A. Bray, Applied Chemistry Section)

Tests were completed last month to review the sulfate precipitation flowsheets for the purification of strontium. The product strontium solution obtained after solvent extraction treatment of Purex Acidified Sludge (PAS) is expected to contain large amounts of calcium (Ca/Sr

mole ratio >6). Sulfate precipitation is used to separate the calcium from the strontium. Tests were completed to compare Sr recovery and Ca removal results using 1, 0.5 and 0.2M Na_2SO_4 . Use of 0.5M Na_2SO_4 was selected for further tests. This will lower the sulfate concentration in the flowsheet, increase the calcium solubility and will effectively cut in half the volume of sulfate required to separate essentially all of the calcium as soluble sulfate waste (a 10/1 Sr to Ca mole ratio is the flowsheet objective). Rare earth element carrier added to the sulfate waste recovered as a precipitate 80-90 percent of the strontium lost in the initial precipitation, while providing a Ca decontamination factor of ~ 5 . Reuse of the rare earth carrier for more than one cycle (before it is sent to solvent extraction for rework) is possible by dissolving the rare earth sulfate precipitate with 0.1M HEDTA and reusing it for a number of cycles. After three cycles, 70 to 80 percent of the strontium was recovered and the Ca DF was about 12. No Ba decontamination was obtained. Reasons for the failure to obtain Ba decontamination and process modifications to attain Ba decontamination will be studied.

Seismic Studies

(D. D. Tillson and V. L. McGhan, Water Resources Systems Section)

Preparation of systems operation/maintenance manuals for the continuous recording accelerometer units of 221-U plant and the short period tripartite seismograph stations were suspended as of November 1, 1972, due to a reduction in funding. Operation of all local seismic systems and maintenance of the U.S. Geological Survey microearthquake network continued.

Service was provided in maintaining the U.S. Geological Survey microearthquake stations and tiltmeter network. No quarry explosions were monitored.

The National Oceanic and Atmospheric Administration, Environmental Research Laboratory (NOAA/ERL) three-component, short period seismograph station at Gable Mountain (HAN) was maintained in continuous recording mode. Arrival time data on significant regional and teleseismic

data were reported to the National Earthquake Information Center, Boulder, Colorado.

The three station triaxial accelerometer system at 221-U plant was maintained in optimum recording mode. A power supply loss to the 221-U Building on October 28th required a brief shutdown and recalibration of the system. During this period, alternate recording systems were utilized to assure no loss of potential data.

Wind Trajectory Studies

(R. K. Woodruff and M. M. Orgill, Atmospheric Physics Section)

Work was directed toward the eventual augmentation of wind measuring systems on the Hanford Reservation. Most wind measuring sites were located. Temporary towers and recorder shelters will be installed during the next two months. The initial programming of the objective wind and trajectory analysis scheme is planned for next month.

Particulate Resuspension Studies

(R. K. Woodruff and G. A. Sehmel, Atmospheric Physics Section)

The installation of the particulate air sampling system at the U-Pond area is nearing completion. The electrical power distribution panel has been delayed in the shops. Electrical generators have been placed at both the east side (11 sampling towers) and west side (1 sampling tower) of the U-Pond area. Equipment is being assembled for erecting 2 cfm air samplers along the east side B-C Zone 1. These air samplers will be at 0.3 and 1.5 meter heights. Results from these low-volume air samples will be used to determine the radiochemistry sensitivity required for filter analysis and to determine more adequately filter placement for the subsequent more definitive high-volume air sampler resuspension experiment.

Atmospheric Release of Radioactive Particles

(L. C. Schwendiman and J. Mishima, Atmospheric Analysis Section)

A rough draft has been prepared for the document covering the findings from the study on the fractional release and size distribution

of particles airborne during the burning of flammable materials containing uranium compounds. Work is in progress on the document, "Some Experimental Measurements of Airborne Uranium (Representing Plutonium) in Transportation Accidents."

Characterization of Radioactive Particles in the 234-5Z Bldg. Gaseous Effluents

(L. C. Schwendiman and J. Mishima, Atmospheric Analysis Section)

Filter and cascade impactor samples of the particles in the 234-5Z Bldg. stack are under way and are scheduled for removal at the end of this month.

Corrosion Studies

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

Corrosion of Corronel 230, Inc 601, Inc 690, Tophet M and 304L SS was determined in 30% TBP-CCl₄+0.1M HNO₃-HCl-HF systems at 25°C. Tests were made using both static and agitated solutions. Specimens were exposed to both the organic and aqueous phases in the static tests, whereas one coupon was exposed to the combined phases in the tests made under agitation conditions. Corronel 230 and Tophet M exhibited excellent corrosion resistance (0.01 ml/mo) under all test conditions (max. 0.01M HCl, max. 0.5M HF). Significant corrosion of the Inconels occurred only in solutions containing 0.5M HF, while appreciable corrosion of 304L occurred in all fluoride-bearing solutions. Preferential attack of weld metal occurred on 304L weldments whereas no preferential attack occurred on Corronel 230 or Tophet M weldments.

TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Summary

Environmental Evaluations

No unusual results were obtained from the routine environmental surveillance program during November.

Radiation Standards and Engineering

AEC Headquarters personnel audited the AEC-RL Emergency Preparedness Program. The new portable Alpha Monitor procedure for the Portable Radiation Survey Instrument Manual is being distributed.

Radiation Protection

The CP and TP procedures and the Radeco Bench Monitor and Portable Alpha Monitor procedure are being distributed.

TECHNICAL ASSISTANCE TO THE HANFORD PLANT

Environmental Evaluations

(R. P. Corley, Environmental Evaluations Section)

No unusual results were obtained from the routine environmental surveillance program during the month. No contamination was detected on road or control plot surveys and airborne radioactivity remained within the expected seasonal range. The latest samples from the 200-East Area locations have the lowest reported particulate beta radioactivity for many months (less than 1 pCi/cm³ gross beta).

Radiation Protection

(K. R. Heid, Radiation Protection Section)

Calibration studies consisting of in-vivo measurements of volunteer human subjects who had inhaled palladium-103 and chromium-51 were completed. The palladium gives off 20 keV X-rays which is quite similar to the 17 keV X-ray from plutonium-239, while the chromium-51 gives off 323 keV X-rays. Since the material inhaled is combined in highly insoluble microspheres, the combination should provide an excellent opportunity to calibrate the plutonium lung counters.

Modifications were made to the light seal on the automatic reader for processing multipurpose thermoluminescent dosimeters. The new seal appears to be holding up well during the routine monthly processing.

Components for 3000 additional multipurpose dosimeters have been ordered. Vendor promises delivery during January, 1973. This should provide an adequate supply of dosimeters so that needed special studies can be initiated.

Scheduling of in-vivo measurements for Hanford works continues to become more complicated because of the rapidly increasing demand by the various contractor organizations. It appears that calibration and special studies may have to be done on swing shift to make more counting time available for measurements of plant workers.

Radiation Standards and Engineering

(H. V. Larson, Radiation Standards and Engineering Section)

A member of the Section attended the AEC Emergency Preparedness Conference, November 15-17, in the Germantown auditorium. A trip report is being prepared.

A meeting was attended with AEC Health and Safety personnel from Washington, D.C., who were auditing the RL Health and Safety Emergency Preparedness Program. RL-AEC and Hanford Westinghouse personnel were also in attendance. Of specific interest was the intercontractor operational relationship and functional capabilities in the 300 Area.

The CP and TP procedures and the Radeco Bench Monitor and Portable Alpha Monitor procedure have been printed and are now being distributed. The first draft of the low-level monitor procedure is being circulated for comments.

Meteorological Services

(E. H. Phinney, Synoptic Meteorology Section)

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

NOVEMBER, 1972

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