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CHEMISTRY DEPARTMENT MONTHLY REPORT

FEBRUARY 1965

AUTHOR

M.T. Walling

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2nd REVIEW-DATE: 2-17-81
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CHEMISTRY DEPARTMENT

MONTHLY REPORT - FEBRUARY 1965

Compiled by Members of
Chemical Research, Chemical Development,
Analytical Laboratories, Chemical Effluents Technology

Submitted by M.T. Walling

March 15, 1965

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CHEMISTRY DEPARTMENT

RESEARCH AND ENGINEERING

FISSIONABLE MATERIALS - 02 PROGRAM

IRRADIATION PROCESSES

Overheated N-Reactor Fuel Study

Two additional irradiated co-product target elements were subjected to rapid thermal transients to simulate the effect of overheating followed by delayed emergency cooling. The two elements were heated to 935 C in individual experiments over a period of about 5 minutes then quenched immediately with a flowing stream of 66 C water. In both tests the elements failed and the core material flowed away from the element at a temperature near 800 C. About half of the metal displaced from the molten core is believed to have been washed or otherwise quickly ejected out of the furnace tube to the downstream catch tank by the action of steam and water during the quench. This was evidenced by a mat of metal particles conforming to the bottom and sides of the catch tank. Cooling was rapid enough to solidify a portion of the aluminum alloy and bond the Zircaloy jacket to the Vycor furnace liner. Virtually all of the core alloy had flowed from the jacket during rupture and quench. A major reaction between jacket and core metal to form the higher melting $ZrAl_3$ did not occur. This was in contrast to the observation of significant $ZrAl_3$ formation in earlier heating tests with radiative cooling alone. Swelling prior to rupture increased the diameter by about 0.04 inch.

A cooperative study was initiated to establish internal bursting pressures of target elements under temperatures expected in loss-of-coolant incidents.

Reactor Studies

Bromide ion was added to the process water used in one-half of H Reactor and the Br-82 formed was measured in the effluent water from both halves of the reactor to determine the extent of cross mixing. These data are important in assessing the validity of half-reactor tests. If no channels of cross mixing are available other than the reactor front and rear face piping, about 6% of the bromide ion is found in the control side. A correction factor must be applied to all half-reactor tests to take this cross mixing into account.

SEPARATIONS PROCESSES

Disposal to Ground

Only minor changes, most of them showing decreasing concentrations, were observed in radioisotope analytical results for the ground-water monitoring program during the current month.

The source of low-level radionuclides in several wells near the Yakima River is apparently the river itself. Isotopic analyses of water samples from these wells show the presence of natural uranium and potassium-40. The Yakima River is somewhat higher in uranium content than are project ground waters.

Plutonium Aerosol Studies

Two additional plutonium rods were ignited at about 600 C in air flowing at 3 cm/sec. The objective of these tests was to determine the particle size distribution of the oxide when plutonium ignition occurs at higher temperature. On a weight basis less than 2% of the oxide consisted of particles smaller than 44 microns in diameter. Particles smaller than 15 microns accounted for less than 0.24%. The size distributions of the large particles were essentially the same as obtained from earlier ignitions.

One observation of interest was that oxidation could be terminated after ignition by cooling with a current of air. The cooling of the air appeared to be sufficient to lower the temperature below the point required for oxidation.

Thorium Dissolution Studies

As part of the study to develop a flowsheet for co-dissolution of aluminum cladding and thoria core, the influence of high concentrations of aluminum on the dissolution rate of thoria was determined. In boiling, vigorously agitated 12M HNO_3-0.025M HF, the rate of penetration of Sol Gel thoria particles is reduced by a factor of about 10 as aluminum concentration is increased from 0.1M to 0.64M. When the hydrofluoric acid concentration is increased to 0.08M, a penetration rate in 12M HNO_3-0.65M $Al(NO_3)_3$ equivalent to that in 12M HNO_3-0.1M $Al(NO_3)_3$-0.025M HF is attained. Use of the high hydrofluoric acid concentration may not be practical from a corrosion standpoint.

Acquisition of data on the range of nitric acid, hydrofluoric acid, mercuric nitrate and nickel nitrate concentrations over which activation of aluminum can be attained was completed. Aluminum dissolution rate studies are in progress.

Polonium Chemistry

Two more potential polonium extractants, di-n-octylamine and di(sec-butyl) phenyl phosphonate, each representing a class of compounds, were examined. Maximum extraction of polonium by 10% di-n-octylamine is at 6M HNO_3 with a polonium-bismuth separation factor of six. Ten percent di(sec-butyl) phenyl phosphonate in xylene gave distribution ratios of less than 0.1 for both polonium and bismuth. Neither compound appears as good as dibutyl carbitol. A report covering flowsheets using the latter compound has been written by Richardson, Sheppard and Guay (BNWC-44).

Polonium Recovery Development

Pulse column development was continued on the bismuth-polonium solvent extraction process described last month. The work this month demonstrated considerably improved performance with organic-continuous column operation with respect to capacity and dispersion when compared with aqueous continuous operation. Special flowsheet modifications were made to permit estimation of nitric acid HTU's in the extraction and stripping contactors. The HTU's (overall raffinate basis) were about 1 to 1.3 ft. in both columns. In addition, the bismuth scrubbing efficiency was measured in the scrub column using 2M HNO_3 as scrub. The scrub decontamination factor was 280, giving a bismuth HTU of 1.6 ft., in line with that previously reported with 0.1M HNO_3 scrub. The overall bismuth DF was 1.2×10^4 in the simulated polonium product streams.

In laboratory studies batch contacts simulating the extraction, scrub and strip columns of a proposed flowsheet for separating polonium from bismuth (dibutyl carbitol as solvent) were performed using uranium (U-233) in place of polonium. Favorable distribution ratios indicate uranium may be used as a stand-in for polonium in Cold Semiworks pilot-plant studies. Additional nitric acid extraction data were obtained to aid in defining process conditions.

As part of the development of an aqueous process for recycling bismuth at Hanford, successful pilot plant runs with the 14-in. spray calciner showed good denitration of 1.52M $Bi(NO_3)_3 \cdot 5\ H_2O$, 3.2M HNO_3 feed at 3.0, 4.0 and 5.0 gph at 750 C wall temperature. The bismuth nitrate contained 1.2, 1.1 and 1.4% residual nitrate respectively. Calciner wall deposits were low with no sticking observed.

[redacted]

WASTE MANAGEMENT AND FISSION PRODUCT RECOVERY

Semiworks Support - Manganese Removal

Two alternate processes to the controlled pH, D2EHPA solvent extraction process for manganese removal from rare earth fission product concentrates have been scouted in laboratory scale experiments. One is based on rare earth oxalate precipitation and the other on sulfate precipitation. The more promising sulfate process was carried through hot-cell testing (with Semiworks feed stock) and has now been used in the plant, apparently with quite satisfactory results.

The development of a process was complicated by lack of a centrifuge in the Semiworks, which required use of decantation for phase separation. The final process, which is not pH sensitive, involved:

1. Rare earth precipitation by addition of 5M $NaHSO_4$ followed by 15-minute digestion at 80 C and one hour for precipitate settling.
2. Decantation (drainage of the tank through a dip leg, followed by a 1M Na_2SO_4 wash).
3. Metathesis with 3.5M NaOH (80 C).
4. Dissolution with HNO_3-HEDTA. The HEDTA is required because some residual sulfate and lead remain because of inability to completely drain the tank.

Dissolution is followed by batch extraction of the total rare earths into D2EHPA followed by cerium oxidation and trivalent rare earth stripping with an oxidizing solution of 2M HNO_3, 0.2M $Na_2S_2O_8$, 0.02M Ag^+. Preliminary results from the plant indicate 98% rare earth recovery through step 4 and a manganese decontamination factor of 50 (which is adequate).

Strontium Semiworks Solvent Treatment

Previous work showed that washing degraded D2EHPA-TBP-diluent solvents with HNO_3-$KMnO_4$ solutions restores cerium(IV) extraction capacity. It is desirable to solubilize solid manganese dioxide formed in this treatment to aid organic-aqueous phase separation. Addition of oxalic acid, citric acid or hydrogen peroxide to the aqueous phase prior to phase separation reduces manganese to soluble Mn(II). Oxalic acid appears to be the most satisfactory of the three reagents.

[redacted]

Persulfate Solubility

In both laboratory and plant cerium removal (via cerium oxidation and D2EHPA extraction), potassium persulfate ($K_2S_2O_8$) has been used as oxidant, and the relatively low solubility of the compound has required large solution volumes. The solubilities of both the potassium and sodium salts were measured (as a function of HNO_3 concentration) and $Na_2S_2O_8$ found to be much more soluble than $K_2S_2O_8$ in all cases. Solubility (at 25 C) of $K_2S_2O_8$ in 0, 1, 2 and 4M HNO_3 was 0.22, 0.264, 0.285 and 0.37M, whereas $Na_2S_2O_8$ was soluble to the extent of 2.4M in water and 1.7M in 2M HNO_3. Hot cell performance of the two chemicals was identical. Use of $Na_2S_2O_8$ is accordingly recommended. Besides much lower solution volumes and improved operating flexibility, a saving in reagent cost may also be realized.

Cobalt D2EHPA Extraction Behavior

The production of Co-60 as a by-product of uranium irradiation (via use of a thin cobalt-containing "sweater") was recently suggested. The D2EHPA solvent extraction behavior of cobalt was briefly investigated to ascertain the feasibility of its recovery from Purex waste. The cobalt extraction coefficients (E_a^o) increased from very low values at pH $\leq$ 1 to over 100 at pH 5 to 6. Acetic, tartaric and hydroxyacetic acid (added as possible complexants) had very little effect on its extractability while EDTA and HEDTA effectively suppressed the extraction at all pH's and citric acid was intermediate in its effect. It appears that cobalt can be readily extracted and stripped and that separation from other metals should be straightforward.

In other studies, a wash with 2M HNO_3, 0.2M $K_2S_2O_8$, 0.02M Ag^+ was shown to regenerate Cold Semiworks D2EHPA solvent which was used in cerium pulse column studies.

Clean U-233 Studies

Continuing tracer-level experiments were aimed at determining: (1) the effect of nitrate on the uranyl sulfate anion exchange process (for recovering ultra-pure U-233 protactinium decay product); and (2) the role of "unadsorbable" Pa species in Vycor adsorption.

Target elements of cold-pressed spray-calcined thoria (67% theoretical density) were prepared for reactor irradiation as a "proof of principle" and, more importantly, to see whether irradiation will have any adverse affect on the excellent dissolution kinetics of spray-calcined thoria. A series of additional calcination runs is being made in the 8-in. spray calciner to: (1) explore further the effects of calciner variables on

product quality (particularly cold-press density and ability to form a sol); and (2) test the calcination behavior of the thorium nitrate product of the recent 6-ton Purex processing campaign. The latter effort (calcination of Purex thorium nitrate) will also provide material needed for certain radiological measurements.

In-Tank Solidification

Tests were completed in the 10-ft. diameter model tank to extend the data on circulation patterns to include the effects of circulator submergence. The general circulation pattern was similar to that observed in earlier studies. The test results show an approximate 40% decrease in circulation velocity as the water level above the circulator is decreased from 8 inches to zero. When the water level was reduced below the top of the circulator, the velocity pattern reversed its expected trend and showed a slight increase above that measured at the point of equal level.

The purpose of these recent tests is to provide information for the prototype in-tank solidification unit which may be operated at various levels of circulator submergence.

Recovery of Neptunium and Plutonium from Purex Plant Stored Waste Sludge

Recovery of neptunium and plutonium from acidified Purex process stored waste sludge (PAS solution) by extraction with D2EHPA-diluent is under study. Plutonium(IV) is extracted into an equal volume of 0.4M D2EHPA-0.2M TBP-Soltrol extractant. Without the organic phase being removed, a reducing agent is added to the aqueous phase to reduce Np(V) to Np(IV). On recontact of the two phases neptunium is extracted without appreciable stripping of plutonium. Hydrazine alone will not reduce Np(V) to Np(IV) in PAS solution at 25 C. Either ferrous sulfamate (0.01 to 0.05 M) or ferrous sulfamate (0.01 to 0.05M)-hydrazine (0.01-0.05M) will reduce Np(V). Greater than 99% extraction of both neptunium and plutonium from simulated PAS into an equal volume of solvent was attained with either of these reducing conditions.

Cerium-Rare Earth Partitioning

The Strontium Semiworks currently uses a batch persulfate oxidation-D2EHPA extraction process to separate cerium [as Ce(IV)] from the other rare earths. A continuous process has been proposed for this operation using the 1A column to extract the rare earths into the solvent (0.4M D2EHPA-0.2M TBP in a n-paraffin hydrocarbon diluent) and a persulfate strip in the 1C column to selectively remove the trivalent rare earths from the solvent. Cerium would be left in the solvent as Ce(IV), to be

removed later by batch washing with a nitric-nitrous acid solution. "Cold" pilot plant runs, using a 3-in. diameter pulse column with a 16-ft. tall cartridge (having plate geometry similar to that of the Semiworks 1C column)- have been made to test the feasibility of the process. Most of the runs have been made with the 1CF heated to 60 C; with the aqueous phase continuous; with a strip solution containing 2M HNO_3, 0.2M $K_2S_2O_8$, and 0.02M $AgNO_3$; and with a 1CF/1CX flow ratio of 5/1. An organic scrub stream (1CS) was introduced at the bottom in runs in which the feed was introduced above the bottom. The flow ratio was then modified to give 1CF/1CS/1CX = 4/1/1. The primary variable studied so far has been feed introduction point. The following cerium DF's and rare earth losses were obtained, with Ce-144 tracer used to follow the rare earth behavior:

Feed Point, Inches Above Bottom	Feed Temperature, C	Cerium DF	% Rare Earth Loss(a)
0	25	3	-
0	60	6	-
22	60	≥ 12	-
30	60	≥ 27	0.5
102	60	135,190(b)	1.1

(a) Measured in separate runs with no $K_2S_2O_8$ and $AgNO_3$ present.
(b) Duplicate runs.

EQUIPMENT AND MATERIALS

Rejected 304-L Stainless Steel Pipe

Several lots of 2-in., schedule 80, 304-L stainless steel pipe intended for use in the Purex annular dissolvers were rejected because results of Huey tests (made off-site) were borderline. Corrosion test specimens from the various lots were sensitized (1250 F for 1 hour and water quenched) and exposed to boiling 65 w/o nitric acid in the multisample tester. Corrosion rates for these samples, measured after 665 hours of exposure, varied from 6.6 to 20.7 mils/mo. Most of the attack occurred on the inside surface of the pipe samples. The corrosion rate for a sensitized "standard 304-L" coupon exposed along with the pipe sections was 0.6 mil/mo.

[redacted]

Non-Metallic Materials

Two polypropylene filter materials were exposed to boiling 60% nitric acid. A 100-micron woven cloth (B-3403 National Filter Media) disintegrated in less than 1 day. A mechanically felted material (Troy Blanket Mills) disintegrated in about 10 days.

Two graphite bearing materials (Graphitar 145C and AP-80) were not damaged during exposure successively to 2 M HNO_3-0.1 M HF and 20% TBP-CCl_4 solutions. Exposure to each solution was about 200 hours at 50 C and 120 hours at ambient temperature.

Pump Development Studies

Development studies are being performed on a variety of liquid and slurry pumps for pilot and production plant service. Chempumps with heat exchangers installed in the bearing recirculation line have been tested for the pumping of hot, radioactive, corrosive slurries. Modifications to the originally installed seals have been completed and the life of the seal faces has been extended well over three times their original life, with no evidence yet of degradation. New bearings of boron carbide have been ordered to further improve life.

Tests with water as the test fluid have been conducted on the Integral canned motor pump. Operation has continued for approximately 1700 hours at a flow rate of 5 gpm against a head of 40 psig with no appreciable wear resulting on the pivoted shoe, modified Kingsbury thrust bearing. A specialized jet type pump manufactured by the Deming Pump Company for handling of corrosive hot light slurries has been under investigation, following nodifications to improve performance with slurries. Although operation is continuing, insufficient data have been compiled for final evaluation.

Preliminary design evaluations have been completed for a pump required for moving concentrated wastes from one underground storage tank to another in the in-tank solidification program. A model pump has been specified for test. This pump is a composite unit combining the best features of several vertical slurry pumps.

Computer Process Control Studies

The feasibility of using the GE-412 computer for process control studies of remotely located systems, utilizing telemetering techniques, was investigated. The reference system for the study utilizes a data logger tc scan and digitize 20 instrument signals at a rate of 2 signals per

second in a fixed sequence. The digitized signals can be transmitted via a telephone data transmission sub-set using a voice quality telephone circuit on a dial-up basis. A device to simulate the telephonic transmitting equipment was built and installed on the data logger currently used to scan the experimental C-column control instrumentation. Using the simulated transmitter, the programs that have been written to re-assemble and process telemetered data are being evaluated.

REACTOR DEVELOPMENT - 04 PROGRAM

PLUTONIUM UTILIZATION PROGRAM

Salt Cycle Process Studies

Several laboratory attempts have been made to prepare 250-gram batches of 20 w/o PuO_2 in UO_2 by electro-codeposition from chloride melts. Thus far, the attempts have been unsuccessful although material of this composition had been made previously on a much smaller scale. The work has demonstrated the need for very careful control of the electro-codeposition process.

Salt Cycle Process Control Studies

Two approaches to the problem of instrumenting a salt bath for Salt Cycle process control are being studied. In one approach, measurement of the "back emf" of the electrolytic cell gives promise of serving as a guide to the PuO_2/UO_2 ratio in the cathode deposit during co-deposition. Preliminary investigations give reproducible results in the range of $\gtrsim$ 0.5 w/o PuO_2. In measurements to date, the "back emf" has proven to be independent of variations in total current, current density, and sparge gas composition. Anode-to-reference potentials suggest that the variations in melt conditions which control the composition of the deposit also control the cell potential. Further investigations will determine the effects of plutonium and uranium concentrations in the melt on the cell potential.

In the other approach, a new light pipe photometer sensing unit for operation in a salt melt has been designed. The present system uses a GE No. 1962 quartz light bulb, encased with its leads in a hermetically sealed tube containing helium and immersed in the melt, as a light source. A prototype source has been maintained at 600 C for over 2 weeks with no sign of deterioration.

Close-Coupled Fuel Processing Economic Study

A report entitled "Economic Evaluation of Close-Coupled Fuel Processing with Plutonium Recycle" has been completed and will be issued as BNWL-28.

This report investigates the relative fuel cycle costs (and elements thereof) associated with two alternative reactor fuel cycles employing light water moderated reactors and either central plant reprocessing-refabrication or close-coupled reprocessing-refabrication. Three reactor types, a typical water reactor with Zr clad fuel, a typical

water reactor with SS clad fuel, and an advanced pressurized water reactor with SS clad fuel were examined. Two fuel management concepts were considered: one with several reactors at a single site with staggered fuel discharges averaging 1 to 3 months between discharges; and the other, a single reactor discharging fuel at 12-month intervals.

During the terminal stages of this study a scope design and preliminary cost estimate was made of the construction cost of a close-coupled plant serving 1000 Mw(e) reactor capacity and providing the process functions of reprocessing, remote fabrication of mixed oxide fuels and UO_2 to UF_6 conversion. Approximately $6,200,000 was indicated.

One of the principal findings of this study was that the fuel exposure for a close-coupled plant cycle optimizes (depending on input data) at 15,000 to 20,000 Mwd/ton resulting in an annual throughput of 40,000 to 50,000 Kg/year per 1000 Mw(e) installed capacity. This optimum is largely independent of close-coupled plant investment. Since the close-coupled plant shares, with most chemical operations, a great economic sensitivity to throughput, increases in throughput rapidly escalate profits and incentives, but lower throughputs result in the opposite effect. The study results indicate that a $6.2 million close-coupled plant serving a 1000 Mw(e) reactor complex can be competitive with central plant processing. Less reactor capacity (or lower throughput) can eliminate the incentives but more reactor capacity (or higher throughputs) can increase incentives markedly.

The annual fuel cycle savings (as compared to a central plant cycle) with the $6.2 million close-coupled plant, and exposures of up to 20,000 Mwd/ton in a 1000 Mw(e) reactor ranged from $0.6 million to $1.6 million 0.09 to 0.22 mills/kwh, depending on reactor type and using the high range costs and high decontamination factor input data. Based on low range cost inputs and a close-coupled plant decontamination factor of 6, the maximum annual savings were $0.3 million, 0.037 mills/kwh, and in the worst case required the close-coupled plant investment to be reduced to $4.9 million to break even. It should be noted that the savings quoted are over- and above-normal profits, amortization, taxes, insurance, etc.

The incentive for close-coupled processing is substantial if fuel exposure is limited to 15,000 Mwd/ton in the central-plant cycle (possibly as high as 0.4 mills/kwh) but drops off sharply at higher fuel exposures.

[redacted]

RADIOACTIVE RESIDUE PROCESS DEVELOPMENT

Hot-Cell Glass Experiment and A-Cell Renovation

Decontamination of A-Cell (following completion of the full-level Hot Cell Glass Experiment and the earlier pilot-plant and pot calcination studies) is proceeding in a very satisfactory manner. An invaluable innovation in equipment removal has been the remote use (with manipulators) of a portable rotary saw, with alundum abrasive cut-off wheels, to cut almost all of the equipment (including the spray calciner) into pieces of suitable size for disposal as dry waste. Result is virtual elimination of personnel radiation exposure and scheduling problems which would otherwise have been involved. Following removal of most of the equipment the cell was further decontaminated to a low level (prior to personnel entry) by vacuum cleaning and high-pressure spraying with a series of suitable decontaminating solutions. This operation was made possible (and practical) by the smooth integral stainless steel liners and "hot" floor drains which were an important design feature of the High Level Radiochemistry Facility. Although full-level Purex waste from several tons of irradiated uranium had been calcined in the cell (and there had been numerous leaks and general gross contamination), only 2 months of day-shift effort were required from termination of the glass experiment until entry into the cell. The importance of stainless steel cell liners (vice painted concrete or mild steel) can hardly be overemphasized.

In other work, leaching studies are continuing on the glass samples from the Hot Cell Glass Experiment, and the final report is being written.

Systematic Glass Studies

Work is proceeding on the determination of the glass-forming characteristics of the sulfate-free Purex waste system. One series of melts has been prepared for this purpose. The drip temperatures of these were found to be lower than the corresponding sulfate-bearing compositions. Solubilities, however, were only slightly lower, both the sulfate-containing and sulfate-free glasses showing a minimum at $M^+/P = 2.3$. The sulfate-free system shows an additional minimum at $M^+/P = 2.1$.

Preparation of an interim formal progress report on the phosphate work to date is about half completed and will soon be issued.

Preparation of Glasses

A series of melts containing simulated Nuclear Fuels Services (NFS) waste and phosphate was prepared As the metal ion equivalent to phosphate

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ratio in the melts increased from 1.7 to 3.1, the fusion temperatures increased from about 960 to 1040 C. Appearance of the solid products changed from glassy at the 1.7 ratio to rock-like and somewhat non-homogenous at the 3.1 ratio. All melts appeared sufficiently fluid at 1050 C to pour satisfactorily.

Coupled Spray Calciner-Melter Solidification

The range of phosphoric acid concentration which produces an operable solidification flowsheet was tested in two runs with simulated 80 gal/ton U 1WW containing 0.4M and 0.63M H_3PO_4 (M^+/P = 2.9 and 1.8). The lower phosphoric acid concentration ran smoothly during a 12-hour run at calciner wall temperatures of 700 C and a melter susceptor temperature of 1125 C. The feed rate was maintained at 5 gallons per hour. Filter pressure drop reached equilibrium at 15 inches of water during the first 3 hours. Melt product was a gray crystalline solid with a density of 3.2 g/cc. When the phosphoric acid concentration in the feed was increased to 0.63M (M^+/P = 1.8) in the second run, the calciner filters showed excessive pressure drop within an hour. Adjusting the filter blowback time cycle from 5 minutes to 2 minutes and switching to repeated manual blowback did not reduce the filter pressure drop. The melt was an olive-green crystalline solid with a density of 2.9 g/cc.

The operability of phosphoric acid-bearing 1WW has now been studied in spray calciner-melter runs with concentrations ranging from 0.4 to 0.96M PO_4^{-3} (M^+/P = 2.9 - 1.1). Phosphate ion concentrations from 0.4 to 0.52M (M^+/P = 2.9 - 2.3) result in workable feeds causing minimal calciner wall deposition and filter pressure drop (~15 inches water). All produce free-flowing melts at 950 - 1050 C which cool to dense crystalline solids.

Solidification of simulated 1WW waste was tested in the coupled spray calciner-melter by the separate addition of 0.5 inch borate glass balls to the melter where they fused and dissolved the calcined 1WW. A two-phase glass with an additive-to-waste calcine weight ratio of 1.6:1 was formed. The glass consisted of a dark brown glassy phase and a lighter yellow crystalline phase tentatively identified as sodium chromate.

The extremely high viscosity of the melt at 1050 - 1100 C (~ 200-250 poises) caused very slow drainage of the melter and prevented continuous calciner-melter operation at 3 gallons per hour of 80 gal/ton U 1WW. High viscosity was caused by glass balls of unknown boron concentration rather than the 100% borax glass ($Na_2B_4O_7$) as previously supposed.

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Waste Solidification Engineering Prototype

Design verification tests were continued with the completion of a 12-inch pot calcination test with a TBP-25 waste as feed. The evaporator and fractionator were coupled to the pot calciner and overall equipment performance was good. Minor flow difficulties are being corrected. Power and water outage was experienced during the run without incident other than delay. The pot contained a 5-inch draft tube for promoting circulation and an experimental pulse reflectometer for ascertaining the liquid or foam level. Twelve hundred and thirty-five liters of feed were calcined in 86 hours of furnace operation. Pot feeding was continued until "off-on" feeding began after about 46 hours of operation (average rate of 27 liters/hour). Performance of the pulse reflectometer was excellent. A continuous steam purged weight factor dip tube was used as a check on the reflectometer and was also successfully used for automatic pot level control.

Data Logging of Waste Solidification Test Runs

Three experimental runs with the Waste Solidification prototype were successfully data logged with the GE-412 computer. The runs included a simulated waste process run, the acid fractionator and evaporator test, and the pot cooling test runs. Twenty-five analog input signals were monitored and converted to engineering units for log out and for use in the special calculation routines. Included in the calculations were periodic averages of furnace power, material and flow balances, conductivity measurement, off-gas accumulation, liquid levels, steam flow, and overall processing rates. Evaluation of the furnace power calculations pointed up the need for recalibration of the instrumentation measuring power input to each zone. A statistically-designed calibration procedure was provided for this purpose. The computer operated continuously during the several tests without failure, although building power transients caused temporary failure of the peripheral typewriters.

Intermediate Level Waste Treatment

A pilot scale run was completed to demonstrate the use of a horizontal plate filter (Sparkler Model HPC-10) with a steam stripper and two bed anion-cation ion exchange system for decontaminating alkaline condensate. Feed rates to the stripper, filter plate and ion exchange beds were 1 gal/min, 1 gal/min/ft^2 and 4 gal/min/ft^2, respectively. The filter operated continuously for 64 hours. The filter effectively removed the particulate matter in the waste.

All radioisotopes were reduced below their respective MPC_w values except Ru-106, which was 1.8 MPC_w. The Ru-106 exceeded its MPC_w value after

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about 100 bed volumes through the anion exchange column. The Ru-106 breakthrough coincides with chloride ion breakthrough. Since nearly all of the chloride is introduced by the addition of hypochlorite, anion bed performance is expected to be improved under plant conditions where hypochlorite or chlorine treatment would not be necessary (added in pilot scale tests to kill bacteria in feed storage system).

CONTAINMENT SYSTEMS EXPERIMENT

Major Facilities and Equipment

Chicago Bridge and Iron Company, contractor for the three-part containment vessel, requested and was granted an extension of the contract to March 30, 1965. The extension request was based on further delays in receipt of the material from the steel supplier. Also, the installation of the bulkheads and reinforcement steel of the wet-wells took longer than was estimated, even thought the construction crew was increased to speed production.

Struthers-Wells Corporation reported that they are on schedule with the design of the reactor simulator vessel. The stress analysis performed on the first design revealed several high-stress areas. The design of the vessel is being revised at present to eliminate the trouble spots.

Installation of the laboratory equipment was started with the goal of having beneficial use of the laboratory by April 1, 1965. Purchase of the material and equipment for the auxiliary systems to be located on the canyon-deck level and crane-cab level was started.

Mathematical Models

The blowdown portion of the digital program was altered to include the effect of vessel sensible heat and to indicate temperature gradients in the vessel walls. Results indicate that severe gradients may develop in that portion of the vessel in which water is retained after the blowdown (gradients perhaps as great as 260 F/in). These calculations will be refined to take into account the various regimes of boiling that may occur at the surface.

Program Planning

An analysis of the interrelation of instrumentation sensitivity and testing time was made to aid in developing tests whereby the leakage rate of the containment vessel is determined by the rate of change of the differential pressure manometer used in the reference vessel method. This method, first used in last year's test of the PRTR leakage rate,

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shows great potential advantages in decreased testing time; and it may also permit less attention to changes of air temperature and humidity during tests of only a few minutes duration.

Fission Product Simulation

Two runs were made during February in the Aerosol Development Facility. In both runs stainless steel clad UO_2 specimens irradiated to 2×10^{17} nvt were melted as a fission product aerosol source. In one case the furnace atmosphere was pure steam; in the other it was a mixture of steam and air. The furnace effluent aerosol was blown into a 1.5 cubic meter tank containing a 1:1 mol ratio of steam and air at 1 atmosphere pressure.

Samples were taken from the tank as a function of time in order to obtain measurements of "air-borne" fission product concentrations, concentrations in the steam condensate, iodine forms, deposition rate on various materials, condensation nuclei concentration, and organic composition and concentration in the gas atmosphere. In addition, grids were exposed for electron microscope pictures. The samples are in the process of being analyzed. No firm conclusions can be made until the results are available, but a very tentative observation is that the iodine from "real" fission product release sources did not behave noticeably different from that from earlier synthetic source releases. No visible fumes were deposited in the furnace tube with the pure steam atmosphere; this is in contrast to abundant fume deposition in air atmosphere.

Two "cold" Zircaloy-clad UO_2 specimens were melted to see whether the water-cooled quartz furnace method of heating was suitable. No problems were encountered due to this different cladding material, but it was noticed that the cladding oxidized in place and retained its cylindrical shape. Four unirradiated specimens were melted in steam or steam-air atmosphere for as long as 17 minutes. The quartz furnace tube was damaged in each case due to insufficnent cooling but did not fail during the heating period.

OFF-GAS DISPOSAL TO GROUND

An analysis of errors in flow paths and times due to: (1) ignoring compressibility of the gas in obtaining the potential pattern, (2) using finite difference approximations, and (3) excluding the well casing effects was outlined and is being made. The effect of excluding the well casing appears to cause an error of < 1% in potentials. It is expected that the error due to ignoring compressibility will be somewhat larger but tolerable. The error due to using finite difference approximations is expected to be quite small.

Analysis of a gas flow problem is under way for homogeneous soil in which the horizontal permeability is greater than the vertical permeability.

SHIPPING HAZARDS

Experiment designs were developed for heating tests of specimens of calcined and vitrified wastes. In this study the release of specific isotopes from various waste calcine and glass formulations will be measured. Equipment required for the study was being located, fabricated and assembled.

COLUMBIA RIVER STUDIES

A submergible television camera was proven to be of value in Columbia River sediment studies. A very clear picture of the river bottom can be seen on the screen in the boat with the camera submerged to depths greater than 70 feet. This closed circuit system will be useful in locating silt deposits and in characterizing the types of sediments to be found on the river bottom.

BIOLOGY AND MEDICINE - 06 PROGRAM

TERRESTRIAL ECOLOGY - EARTH SCIENCES

Hydrology and Geology

The program "Steady State Flow Through Soils" was evaluated to determine the inherent error due to the approximation of continuous derivatives with discrete segments used in the solution of finite difference equations. Also, the error magnitude related to averaging permeabilities about the central node in solution of flow through heterogeneous soils was determined. Statistical analysis, using exact closed-form solutions for comparisons, showed a standard error of difference of 2.56×10^{-5} due to the derivative approximations and a standard error of difference of 8.88×10^{-4} due to permeability averaging. These relatively small errors lend confidence to the nearness of approach to exact solutions for this particular program.

A tracer test was run in three piezometers which are sand-packed in well 699-10-E12. The purpose of the test was to determine qualitatively the interflow of water to a pumped middle tube from tubes above and below. Fluorescein and nitrate traces were added to the upper and lower tubes, respectively, while the middle tube was being pumped at a rate of about 10 gpm. Fluorescein was detected after pumping about 1100 gallons. Nitrate analyses are not yet completed. Comparisons of the concentrations of the two tracers in samples should provide evidence of the relative amount of water entering from above and below the pumped section.

Geologic mapping west of the Hanford Project up to the Yakima Firing Center disclosed that the four Priest Rapids basalt flows pinch out within a short distance. The stratigraphic section thus thickens rapidly toward the Pasco Basin center, and the Pasco Basin on the west is structurally more sharply downwarped than earlier believed.

Activation Analysis

Four samples of basalt taken from different locations in two characteristic basalt flows were analyzed by neutron activation, simple chemical separation, and measurement of multidimensional gamma ray spectrometry. Preliminary values of Sc-46, La-140 and Eu-152m indicate significant differences between the basalt flows may exist large enough to provide a characteristic identification technique. More samples are being analyzed for a greater variety of radionuclides to verify the value of this method.

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Soil Chemistry

The mineralogical composition of the clay fraction from surface horizons of two Hanford soils was determined by X-ray diffraction analysis. One soil represents surface soil material in the valley south of Gable Mountain; the other was collected near the Columbia River in the vicinity of 100-F Reactor. Clay fractions were separated by a centrifuge procedure. The fractionation followed treatments which removed exchangeable divalent cations, free carbonates, iron oxides and organic matter.

Semiquantitative analysis of minerals showed that montmorillonite is the dominant clay mineral. Vermiculite, mica, chlorite and traces of kaolinite also were detected.

RADIOLOGICAL AND HEALTH CHEMISTRY

Radiation Chemistry

Additional measurements of the ESR spectra of the product from the dithionite reduction of p-nitroaniline confirmed that both it and the radical obtained by γ-radiolysis of p-nitroaniline in air-free solutions are the same. A similar comparison of the spectra from dithionite-reduced p-nitrobenzoate and the γ-radiolysis of p-nitrobenzoate showed that the radicals obtained by these two procedures are not the same. As yet no structure has been found with reasonable splitting constants to give a synthetic spectrum which matches that obtained experimentally by γ-radiolysis. Interest in this radical is considerable since it has a half-life of 8 minutes in oxygen-free water and its structure could give important clues as to what features stabilize a radical.

Germanium (Li) Detector Studies

Gamma ray spectra of gross fission products of U-235 and Pu-239 have been accumulated as a function of time after bombardment for the period of 4 to 32 days. The spectra are very similar but with some notable differences. The principal observed differences are the much higher yields of Ru-103 and Cs-136 which are observed in the fission of Pu-239.

ATMOSPHERIC RADIOACTIVITY AND FALLOUT

Aerosol Sampling Studies

The study of subisokinetic sampling errors was terminated. A first draft of a report of this work was completed.

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Turbulent Deposition

Markedly improved precisions in deposition measurements were achieved through using very narrow size range of particles prepared by the spinning disc generator. Some important observations were made from experiments to accurately determine the deposition velocity, K, for particles moving in a 0.5 inch diameter tube. The length of tube from the entry point required to develop fully turbulent flow conditions was shown to be of the order of 75 tube diameters for the 1/2-inch tube used. This was much longer than anticipated and suggests that initial measurements of deposition velocity for larger than 1/2-inch diameter tubes may have been determined for the entry conditions rather than for fully developed turbulence. Beyond the entry section, the derived exponential equation for decreasing concentration with distance fits the data nearly perfectly. Of even greater significance is the higher dependence of deposition velocity on average flow velocity. For a correlating parameter, ϕ_{S^+}, of less than about 100 the deposition velocity, K, is dependent on a higher power of the average air velocity than the 5th power initially determined from a composite of all earlier data. Beyond S^+ of about 100 the velocity dependence is considerably less. The improvement in accuracy achieved may require revision of the models previously held for turbulent deposition. Studies on larger tubes and particles are planned.

Fallout Studies

A major increase in the Ba-140 concentration of air was observed on two air filter samples collected from January 25 to February 1, and from February 1 to February 5. An increase in Co-60 was also noted for these two samples. These increases may be from the USSR underground test reported to have been exploded on January 15, with considerable venting to the atmosphere.

ISOTOPES DEVELOPMENT - 08 PROGRAM

PROMETHIUM ISOTOPIC FUEL DEVELOPMENT

Promethium Ion Exchange

Two column runs were completed to determine the effect on degree of rare earth separation of substitution of sodium hydroxide for ammonium hydroxide for adjustment of eluant (EDTA) pH. The effect, if any, was very small. These runs complete the comparative study of effect of elutriant choice (EDTA, DTPA or HEDTA) and operating conditions on promethium purification. A final formal progress report is being prepared.

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Compatibility Studies

Following the preliminary screening studies reported earlier, a large number of samples have been prepared for more intensive testing of the compatibility of proposed promethium core compounds with candidate cladding materials. Neodymium oxide (Nd_2O_3), which has been through the same chemical preparation steps that will be used for Pm_2O_3 (to insure a proper distribution of trace impurities), is being used as stand-in core material in these long-duration heating tests. Metals and alloys under test at 1100 C include Hastelloy-C and -F, 304L and 310 SS, Haynes-25, Inconel, Incaloy-804, Ta, Ta-10W, W, W-25 Re, Mo, Pt, Carpenter-20, and TZM. The coupons are pressed in Nd_2O_3 compacts in mullite crucibles and the latter are then sealed in Vycor tubes, under a 150 mm He atmosphere. (Purpose of this pressure of helium is to provide mechanical support to the Vycor at 1100 C.)

Equipment is being assembled for higher temperature (up to ~ 2000 C) testing of Ta, Ta-10W, W, W-25 Ru, Mo, and Pt with Nd_2O_3 and Sm_2O_3. The samples will be packed in graphite crucibles which will serve as the susceptors for induction heating in a vacuum induction furnace. A somewhat larger number of metals and alloys will be tested at intermediate temperatures (ca. 1500 C).

Additional metals and alloys will be added to the test program as samples become available. Samples will be removed from the furnaces after various periods of time and subjected not only to metallographic examination and measurement but also to mechanical testing and micro chemical analysis (to determine the identity of diffusing species, new phases, etc.).

Besides the above experimental program, thermodynamic calculations have been made in an effort to determine the probability that various conceivable clad-core reactions will occur. The free energies of reactions of the type

$$Pm_2O_3 + \text{Metal} \rightarrow 2PM + \text{Metal Oxide}$$

where M was Cu, Ni, Mo, Fe, W, Cr, Ta, Ti, Zr, etc., were in all cases positive, and generally strongly so, implying that the reactions should not occur to a significant extent (i e., equilibrium should be far to the left).

Compound Properties

A first attempt at direct experimental measurement of the surface tension of neodymium fluoride (by the sessile drop technique) has given a value

far in excess of the expected value. More refined equipment is being readied to study this in more detail, as well as to measure the thermal emittance of rare earth metals and compounds.

Re-entry Burn-Up and Cool-Down

A part-time effort has continued to be devoted to computer studies of re-entry hazards to guide our clad and core development effort. Calculations appear to be in reasonable agreement with those of General Electric MSVD, Hittman Associated, Martin Nuclear, and Sandia. Whether or not burn-up will be achieved is found to be highly dependent on the initial conditions assumed. These include not only the dimensions of the capsule and the particular clad and core materials but also the assumed velocity, angle and altitude of release

The computer program calculations bear out the belief that promethium metal will burn up more easily (or more completely) than strontium fluoride (SrF_2), because of its higher density and lower melting point. Whether burn-up will occur in either case will depend greatly on the clad. Thus, a 2-inch x 6-inch promethium capsule clad in Rene-41 will be only partially burned up for an expected exposure altitude of 75,000 meters, descent angle of -1°, and velocity of ≤ 6700 meters/sec. With lower melting clads, the situation is much more favorable (however, the lower melting clads may be ruled out because of launch pad abort safety considerations). Even with lower melting cladding materials, burn-up cannot be relied on for lower-altitude, lower-velocity release conditions. Intact re-entry accordingly appears to merit serious consideration.

In other calculations, the impact velocity and capsule temperature were calculated, as a function of capsule size, for assumed release at a speed just below Mach 1 at 12,000 meters altitude. The promethium (metal) was assumed to be at the melting temperature at time of release and both isotopic heating and aerodynamic cooling were included in the computations. It was found that impact velocities and temperatures will be fairly low and the core solid in all cases considered (right circular cylinders up to 3 inches in length and diameter). Impact velocities range from 40 to 70 meters/sec and surface temperatures from 300 to about 600 C.

Prototype Heat-Source Fabrication Equipment

Completion of design of the equipment items required for promethium prototype heat source fabrication, writing of specifications, placement of orders, and liaison with the shops has continued to receive top priority and to occupy considerable time during the month. Most

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designs are now in the shop, and delivery and assembly is confidently expected by the time decontamination of A-Cell is completed.

As a result of conversations with the Chemical Processing Department of the General Electric Company and the Division of Isotopes Development of the AEC, the promethium purification capability of the rework columns, which are a part of the A-Cell package of equipment, was evaluated. (The rework columns were initially provided to remove daughter samarium and other impurities from reclaimed prototype heat sources.) If a feed no higher in total ionic impurities than that produced by the Semiworks is available, purification capacity should equal the requirements of the experimental prototype program (estimated as about 3 megacuries per year beginning FY 1966). With certain relatively minor equipment changes, capacity could be increased several-fold. An important feature (besides capacity and reliability implicit in a manipulator operation) of A-Cell for promethium purification is the fact that its heavy shielding precludes the need for prior cerium removal.

Pneumatic Impaction Process Development

All bolts on the 1220-F Dynapak machine were torqued to the specified level. Preliminary tests of frame mounted strain gages indicate that this system has promise as a method for measuring punch pressure during impact.

In other activities, design of the can heating and loading mechanism and scope of the mechanism for remotely changing the punch and die were completed. Cerium oxide and cerium fluoride have been produced with the multipurpose precipitation-filtration equipment from a nitrate feed solution. The suitability of the product for feed to the pneumatic impaction process is currently being evaluated.

Characterization of the Dynapak impaction machine performance is under study. Initial emphasis is on measurement of stresses using cemented strain gages and a memory oscilloscope for readout. Eight successive impactions of a steel cylinder resulted in 100 microstrains initially, increasing to 300 microstrains, in the lower platen. The initial strain pulse occurred over a time interval of about 2 milliseconds, corresponding to a frequency of 250 cps; additional higher frequency vibrations were also observed. Preparations are being made for additional measurements using strain gages and accelerometers with readout by means of a light beam oscillograph.

M. T. Walling, Jr.
Manager
Chemistry Department

MT Walling:cf

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