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MONTHLY PROGRESS REPORT

For January 1953

Technical Division

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February 10, 1953

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E. I. du Pont de Nemours & Co. (Inc.)
Explosives Department - Atomic Energy Division
Wilmington, Delaware

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I. HEAVY WATER

The first heavy water brought to final concentration by the Savannah E Plant was drummed about January 20.

GS Process

Corrosion Inhibitors

The Engineering Research Laboratory program on corrosion inhibitors for carbon steel exposed to H_2S-H_2O has been completed. Data recently obtained covers various combinations of tetraethylene pentamine, ammonia, and NaOH. Object of the tests was to determine whether such materials might be more effective as inhibitors when used in combination rather than separately.

As an aid to interpretation of results we now have a general curve showing corrosion rate vs. time of exposure for carbon steel in H_2S-H_2O without inhibitor. This curve is based on several hundred individual coupon tests, a sufficient number, we believe, to represent a reliable average. Using this curve as a reference, we find that only a few inhibitors show improvement in cumulative corrosion rate over the uninhibited system after 2000 hours exposure. For instance, tetraethylene pentamine at 500 ppm concentration shows about 0.5 mil per year corrosion compared to 1.0 mil for the uninhibited system. No inhibitor or combination of inhibitors in concentrations of 100 parts per million or less shows a corrosion rate as low as 1 mil per year. This conclusion is based on a sufficient number of repetitive tests to permit averaging of results.

Certain combinations (for instance, tetraethylene pentamine and ammonia) show lower corrosion rates at a total concentration of 100 ppm than either used separately at concentrations of 100 ppm. As previously noted, however, no such concentration of inhibitor reduces corrosion sufficiently to constitute an improvement over the uninhibited system. In fact, low concentration of inhibitors, such as 100 ppm, would, it now appears, permit a continuing corrosion rate of 2 to 4 mils per year.

It should be noted that most process equipment will last indefinitely when subjected to a corrosion rate of 1 mil per year, 3 mils being considered about the upper limit of acceptability for commercial design. The most encouraging finding of our corrosion research program is this resistance of carbon steel to H_2S-H_2O once a protective coating of iron sulfide has been formed on the surface.

At those points in the process, however, where high velocities and spray impingement occur, the protective sulfide film does not form and carbon steel is rapidly corroded. In-

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hibitors might be of value under these conditions, except for another problem of major importance, namely, that inhibited systems carry iron sulfide corrosion products in suspension, and this iron sulfide rapidly plugs bubble cap trays. For this reason, the practical solution to the problem of erosion-corrosion appears to be the use of more corrosion-resistant materials (stainless steels) for items subject to erosion-corrosion. The GS plants are now equipped or are being equipped with stainless steel tower trays, heat exchanger tubes, pumps, and control valves. (At Savannah these items, with the exception of control valves, were specified as stainless steel in the initial design.)

For the time being at least, the testing of inhibitors has been stopped at the Engineering Research Laboratory.

Sulfide Deposition

Although the solubility of iron is relatively low in the H_2S-H_2O system and iron sulfide forms as a coating on exposed surfaces, some iron is carried in solution. There is a gradual increase in iron content of the water as it passes through the cold part of the system, and deposition of iron sulfide occurs wherever the solution is heated. Removal of such deposits by mechanical cleaning from the liquid heaters and from the waste water strippers at Dana has caused some outage. This problem is being intensively studied at the Engineering Research Laboratory and at the plants with the object of preventing the deposition, if possible. So far, such deposition has not been encountered at Savannah where more resistant tower trays (304 stainless steel rather than 410 stainless steel) might be expected to reduce the amount of iron picked up by the process water. The amount of iron picked up by the water, while sufficient to cause plugging at certain points in the system, is not enough to constitute a serious loss of metal from the equipment.

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II. REACTOR MATERIALS

Uranium Fuel Elements

Metal Supply - Fernald Liaison

The Feed Materials Production Center expects to resume regular production of SRP slugs, discontinued December 10, on about February 19. However, early in January some slugs were fabricated from residual rod stock. This rod stock was, for the greater part, 1-1/16" diameter material that had been in storage since last August when the size of rods for SRP slug production was increased to 1-1/8" diameter. The old, smaller rods were machined for the purpose of reducing inactive metals stock.

On January 20, 21, and 22, an experimental batch of 150 ingots was rolled through the FMPC mills to 1-1/8" diameter rod. This material is being machined into SRP slugs. The experimental batch was processed for the purpose of making a yield comparison based on ingot surface characteristics. Three groups of 50 ingots each were included in the batch. The ingots in one group had been scalped (lathe turned), the second group had been pickled in phosphoric acid, and the third group was in the "as-cast" state. It is intended that a comparison of yields from these three groups will lead to a decision as to whether the conditioning of ingots must be continued.

Fernald liaison activity has been transferred to the Manufacturing Division beginning February 1 and will be covered in their reports in the future.

Aluminum Sheaths

A shipment of 100 each of 2S and 99.6 deep drawn aluminum cans from Olin Industries has been received at Savannah, and a program of evaluation of these cans is planned. The remainder of the 10,000 experimental deep drawn cans (5000 of each composition) is scheduled for completion next month. Olin has received partial shipment of AlSi clad 2S sheet from Alcoa. This material is supplied for the program of making 10,000 deep drawn aluminum cans lined with 12% AlSi.

Non-Destructive Testing

Development Program

A large part of the work this month in non-destructive testing has been in the planning and procurement of the test devices. The preliminary plans for the production-line helium leak test have been turned over to Engineering for final design.

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Plans of a device to hold and rotate the slugs during radiography of their welds have been turned over to production, and a device to test slugs for transformation has been recommended to production. Test devices purchased this month are a Wide Band Converter ultrasonic instrument and a High Precision Radiographic Unit.

Plans have been made and construction started at ANL on an alternating current bridge to be used in conjunction with the Multitest Probe device. This addition will enable the Multitest Probe to be operated at frequencies of 25, 30, 35, and 40 KC. At these high frequencies any effects due to slug warpage or eccentricity can be eliminated from the output of the device so that only point penetrations are detected. With the Cyclograph Probe, a new Croloy probe core has produced better sensitivity than has been possible previously and has eliminated the end-effect. Both probe instruments can resolve 1/32 in. diameter defects 3/32 in. apart, and both have a scanning time of roughly five minutes per slug.

A new method of recording using electrosensitive paper instead of film is being employed with the ultrasonic test set-up at ANL. This method reduces scanning time to four minutes per slug. A method of changing the pulse rate of the Reflectoscope has been proposed for reducing scanning time to possibly one minute per slug.

Savannah Activity

The majority of the production slugs are being cyclographed, and approximately 300 have been machine stripped to check for penetration. The cyclograph definitely determines penetration with significant differences in reading for various degrees of penetration. However, it does not seem likely that better correlation between cyclograph readings and minimum can wall thickness can be obtained with the present concentric coil set-up, because this measures only the average penetration and can be used only to predict the probability that a slug is bad. It may develop that the point probe referred to above will give almost 100% correlation.

Approximately 50% of the month's canned slug production has been radiographed, classified, and tabulated. The autoclave program has been resumed and slugs have now been exposed at 125 psi steam pressure for a total of 407 hours. The caps of three groups of slugs have been ground down to expose voids and these have been exposed in the autoclave 44, 277, and 363 hours without a failure, except for one in which the uranium was exposed.

Radiography of the cast aluminum billets in 320-M Building has been improved and now the radiographs show the contours of the pipe and detect small internal voids, as well as surface irregularities. Further work is being done to reduce the background due to scattered radiation.

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New Fuel Element Development

Work has continued on setting up programs to cover various phases of new fuel element development including work on core, bonding layer, sheath, and testing. Attention is being given to the fabrication of the core by wrought metal and by powder metallurgy techniques. There have been several discussions with the Sylvania Electric Products Co. in regard to powder metallurgy work on the core and related work to make the complete fuel element.

A tentative program, involving major work at SRP, Battelle Memorial Institute, and Sylvania, and with minor work at several other sites, has been developed and is ready for discussion with the AEC.

SRP Metallurgical Development Facilities

During February it is expected that the first equipment originally provided for the metallurgical development work in 773-A Building will be ready for use. Equipment has been provided for melting, casting, rolling, straightening and machining uranium to provide up to 10-foot plates. The rolling mill has been operated and is undergoing run-in tests. A temporary straightener is 85% complete. Jigs and fixtures for machining 10-foot plates are 60% complete. All equipment has been installed, except the following: cleaning tanks, degreaser, tilting vacuum melt furnace, and punch press.

All critical material and supplies needed for the production of flat plates are on hand.

Installation of new development facilities authorized for 313-M Building has been temporarily deferred pending a study of the expansion of 773-A Building to house in a single location all equipment required for flat plate work. This will include facilities needed for bonding work and for testing the completed assemblies. These had not been specifically provided for in the earlier program. It now appears the extension of the Metallurgical Development Laboratory in 773-A will provide the best answer for a flexible set-up suitable for the present flat plate fuel element development and for future developments, such as tubular fuel elements.

Bonding

At SRP two small resistance-heated pots have been temporarily installed in the Special Process Room of 313-M Building for dip coating work. Crucibles, uranium strips, and various master alloys are on hand for the preliminary dipping work. Thin uranium plates have been dipped in the production AlSi bath to produce relatively even coats of AlSi approximately 2 to 3 mils thick.

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BMI has all the facilities ready for the hot dip bonding program. No results have been received as yet.

Contacts have been made with a number of organizations regarding the possibilities of an electroplated bonding layer applied either directly on uranium or plated on the inside of the aluminum sheaths. Pieces of flat plate uranium have been supplied both to KAPL and to Sylvania for application by these organizations of the nickel coatings that they have developed. Information received from other sources indicates that it should not be difficult to plate satisfactorily on the inside of sheath tubes and that a satisfactorily adherent nickel on aluminum plating will not require more than very thin undercoats of zincate and copper layers.

Sheathing and Welding

ALCOA has been successful in flattening ribbed aluminum tubing to required shape by the use of a properly designed die. The flat tubes were used with steel cores in preliminary studies of sheathing prototype fuel elements. Some minor difficulties experienced are believed to be easily corrected, and experiments will then be carried out using uranium. Later work is programmed to produce sheath tubes with an AlSi lining.

ALCOA has accepted a Purchase Order for the fabrication of sheaths and process tubes of the now preferred design. Dies for the extrusion of the process tubes are currently being made. Primary effort is being placed on this item in order to provide tubes for the flow tests required to prove out the sheath design.

Investigation has been made of the possibility of using unribbed sheaths and welding the necessary ribs on after the assembly of sheath and core has been completed. Experiments with resistance and induction welding were unsuccessful, but preliminary results at Battelle with percussion welding appear promising.

Alloy Development

An extension of the present BMI contract has been approved for continuation of the alloy program and other miscellaneous work for a period of nine months starting February 1.

A series of alloy heats has been induction melted in graphite varying the time molten before pouring. This variation seems to cause no significant change in homogeneity, carbon content, or corrosion resistance of the alloys. In addition, it was observed that top pouring caused no apparent increase in carbon content, versus about 700 ppm pick-up during bottom pouring.

Corrosion testing continued during the month and has affirmed the characteristics of the alloy previously described.

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Special Materials

Thorium

The final work necessary for the irradiation tests programmed at Hanford with thorium slugs is now being performed at SRP. Techniques have been developed for metallographic examination and for making several desired measurements on the pieces. A satisfactory solution for etching thorium prior to canning has been found in 40% nitric acid used with the addition of 0.2% fluosilicic acid at a temperature of 50°C. A small number of slugs are being canned to check the suitability of the canning procedures (the same as for lithium-aluminum alloy) for the slugs to be irradiated at Hanford. Particular attention is being given to the possibility of dimensional changes during the die-sizing and swaging operation.

A formal letter has been received from the Oak Ridge National Laboratory advising of their willingness to make dimensional and hardness measurements on the thorium slugs after irradiation, and a visit is scheduled to Oak Ridge to go over the work in detail.

Bismuth

The procurement of bismuth was discussed with the supplier. Approximately 1900 additional slugs are being fabricated at the present time for shipment to Savannah River in the near future. The supplier plans to provide Savannah River with a reserve stockpile of approximately 10,000 - 13,000 pounds of acceptable slugs later in the year. Inventory and status reports regarding Savannah River are to begin with the first shipment of irradiated bismuth to the processor. The processor can perform calorimetric tests for us if we desire them as a means of determining flux densities and patterns.

Crucibles and Blend 80

The requirements for crucibles and Blend 80 for 1953 were discussed with the supplier. The supplier will evaluate different methods of achieving improved cylindrical contours. No supply of low-silica MgO is immediately available.

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III. REACTOR TECHNOLOGY

Control

A decision was made to install position-demand equipment in all five piles. Details are being reported by the Process Section.

Tritium Production

Forecasts of tritium and plutonium production for various loadings of the Savannah River piles with enriched and natural uranium have been prepared on the assumption that the piles will start up and rise to power according to the Manufacturing Division's latest schedules. Among the cases considered are the following:

1. All five piles loaded for plutonium production.

2. R, P, K, and C Piles loaded for plutonium production; L Pile loaded with enriched alloy slugs.

3. All five piles loaded for tritium production with 3/4-inch slugs or with a loading of one-inch slugs having equivalent lattice properties (see below).

4. All five piles loaded for plutonium production, but with a producer blanket of lithium alloy.

5. R and P Piles loaded initially for tritium production, L Pile enriched, and K and C Piles loaded for plutonium production. R and P Piles to be converted to plutonium production when the tritium production from L Pile becomes available.

These studies showed that although Case 2 can meet the long-range requirements for tritium, it cannot economically meet the early needs. Cases 3, 4, and 5 show some hope of meeting the early requirements. In particular, calculations on Case 3 indicated that it may be possible to duplicate the lattice properties of the 3/4-inch loading either (a) by loading only three columns of slugs in each Q-tube or (b) by loading four columns of slugs, but separating the slugs in each column an appropriate distance apart by means of tubular aluminum spacers. In this way it may be possible to improve the tritium production without requiring development of major new components. An extension of the ZPR-II program to determine the optimum loading scheme, and later to explore enriched loadings, is under discussion. It is hoped that the feasibility of attaining the production goals by modifying the loading of natural uranium can be determined within the next two months, and that exploration of enriched systems can begin shortly thereafter.

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CMX

Two experiments completed in January give further confirmation that film formation in heat exchangers cooled with raw chlorinated Savannah River water will be almost negligible if either (1) the exchangers are cleaned once or twice a month, or (2) the raw water is chlorinated intermittently. In an experiment with Foster-Wheeler exchangers (100 Area prototype) the resistance to heat transfer increased only two percent in three months of operation with continuously chlorinated raw water. The exchangers were cleaned with 100 ppm of oxalic acid every three weeks. As judged by thermal measurements, no film formation was detected in the Griscom-Russell exchangers in a four-month run with intermittently chlorinated raw water. Although the heat transfer resistance increased 4 percent during the run, the application of conventional heat transfer equations to the data showed that the increase merely reflected a declining river temperature. The Griscom-Russell exchangers also were cleaned with oxalic acid every three weeks, but the results indicated that frequent cleaning may be unnecessary when intermittent chlorination is employed. A run is in progress which should establish this point more firmly.

During the past month, all heat exchanger tube bundles were removed from their shells and the tubes examined. With the following exceptions, all tubes were in excellent condition.

1. Isolated small tubercles were found. These appeared to have been produced by small specks of iron occluded in the stainless steel surface during the tube drawing operation.

2. The cold end of the G-R exchanger which had recently operated on raw unchlorinated water was partially coated with a fine light colored film. A second cleaning with oxalic acid removed this film.

Several experiments were started during the month with the objective of determining whether silt will deposit in 100 Area exchangers at reduced flow rates. Results are inconclusive thus far.

Moderator Purification

A distillation system has been selected for installation in "C" Area for the purification of the moderator. This choice was based on both technical advantages and estimated reduced investment over the resin bed system selected for R, P, L and K. CMX has recently shifted to this method of purification of process water in order to assess such operating difficulties

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as may be inherent in this system. The purity of water produced by distillation as measured by electrical resistance was about 0.5 megohms compared with 2 megohms previously produced by resin beds. It is believed that much of this difference is due to the CO_2 absorbed from the air. This will be eliminated both at CMX and in the 100 Area by (1) a helium blanket which should exclude air and (2) by the addition of a small resin bed to the distillation effluent line. The rate of aluminum corrosion will be measured in the water purified by distillation only.

Reports from Chalk River indicate that an improved method of detecting slug rupture, based on delayed neutron emission, has been developed. This detection system signalled that a fuel element had failed when operating at high power six hours before their standard gamma monitor gave a positive signal. The early warning given by sufficiently sensitive instrumentation can therefore assist in limiting the size of ruptures. A mechanism of increasing the sensitivity of the SRP gamma monitors may be to increase the time allowed for the decay of interfering background materials. Tests of this type are underway both at SRP and Hanford.

Heating in the 105-R Concrete Shield

The height of many of the duriron blocks in the thermal shield of 105-R was near the lower tolerance and shims were required to compensate for the cumulative effect of this deviation. To prevent blocking of water channels, the shims were placed only at the ends of the grids, leaving gaps about 1/8" high in the center. The radiation passing through the gaps has been estimated to be about 80 BTU/ft^2-hr. at 700 MW, or about eight times as great as that passing through the solid duriron. When there is no water flow through the cooling coils of the concrete shield, the temperature rise in the concrete due to the presence of the higher intensity beams was calculated to be about 25°F, or about 1.6 times the value predicted for a thermal shield with no gaps. Although the higher intensity beam is absorbed in only one-tenth of the total concrete volume, the heat generated in this portion of the shield (on a direct line with the impinging radiation) is rapidly conducted to the adjacent sections where the heat generation is lower. Thus, the effect of the increased heating is to cause a general temperature rise in the concrete rather than to create localized hot spots. The cooling coils in the concrete which were added originally as a safety factor should reduce the concrete temperature to that expected with perfect thermal shield blocks.

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Columbia University

Heat transfer measurements using an electrically heated mock-up of a reactor fuel channel were extended to include coolant velocities up to 46 ft/sec and heat transfer coefficients up to 10,000 pcu/hr $ft^2{}^oC$ with a heat dissipation of 520 KW (equivalent to a reactor output of 970 MW). The heat generation in the tube was distributed sinusoidally to simulate that expected in the reactor.

In a new series of tests designed to explore boiling disease problems, the electrically heated tube burned out at a point 10 feet down the tube when the coolant flow was reduced to 10 gpm (30% of normal) with a total heat release of 270 KW (equivalent to a reactor output of 500 MW). At this reduced water flow, both steam and water issued from the tube exit. Additional tests are required before it can be established whether this defines the limit of an operable zone or whether the burnout was related to causes applicable only to the Columbia test setup.

A satisfactory design has been developed for making corrosion tests on 1-inch 2S aluminum tubes at heat fluxes up to 100,000 pcu/hr.ft^2. A test run has been in progress for 400 hours.

Preparation for Startup

A test was made at NYX to determine whether it will be possible to separate the metal and moderator power coefficients of reactivity by manipulation of water flows in such a way as to produce different temperature transients in metal and moderator. The test showed that large and unpredictable temperature gradients are produced by mixing of the water discharged from the Q-tubes with the water in the tank, and also by thermal convection within the tank. It therefore appears unlikely that the reactivity effects produced by the metal will be quantitatively separable from those produced by the moderator.

ZPR-II

Measurements of Savannah-type safety rods in ZPR-II have confirmed earlier estimates that the Savannah safety system will control about 3% $\Delta k/k$, which is adequate.

The program in support of the plutonium producer has been concluded, and a report presenting the theoretical interpretation of all data taken with the ZPR-II lattice is in preparation. As noted previously, a new program dealing with special loadings is under discussion.

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IV. SEPARATIONS TECHNOLOGY

Purex Process Studies

The recent incident with the Savannah River Laboratory uranium nitrate evaporator has re-emphasized the potential hazards involved in the acidic evaporation and acid recovery steps of the Purex process. With assistance from KAPL and ORNL, a process study has been undertaken with the aim of eliminating potentially hazardous operations in the 221 Building. Among the items being considered are (1) barricading the hydrate evaporators in the 221 Building "A Line" (this operation corresponds quite closely to the process which was being carried in the SRL evaporator), (2) relocation and modification of the present ICU and IEU aqueous uranium evaporators and possible substitution of continuous evaporators with small liquid holdup, (3) neutralizing second uranium and second plutonium cycle acidic wastes before evaporation, and (4) flowsheet modifications which would eliminate acidic uranium and plutonium evaporations.

221 Building Equipment

There has been concern that the unusual rise (about 25 feet) of the steam condensate lines above heated vessels in the canyons would cause condensate flashing, and result in poor steam trap operation and steam or water hammer in the coils. An investigation of the performance of the prototype evaporator at ORNL revealed no trap difficulties or noisy operation, but calculations showed that sufficient cooling probably occurred in the long condensate line to prevent flashing. A comparable heat loss to air is not possible in the 221 Building, but recent estimates indicate that there will be sufficient sub-cooling in the submerged 1-in. lines from the coils to prevent flashing. Consequently, bucket-type traps are considered adequate for the evaporators.

The first production model of the 25 gpm transfer steam jet has been received and tested at the SRL semiworks on water and organic. It is considered to be a good machining job and has performance characteristics acceptably close to those of the prototype. The testing of the plant transfer steam jets (1 out of every 10) will be extended on the first one or two of each type to fill in gaps in present jet performance data, and to investigate such things as reliability of starting (10 consecutive successful starts) and the ability to start against a head of 10 feet of fluid. A standard plant gang valve will be used in the tests.

A study of the placement and effectiveness of the canyon vessel ionization chambers has been completed. The results indicate that (1) in many cases the activity of each

vessel may be measured with sufficient accuracy for process requirements with the present design of ion chamber located in the concrete walls, (2) in some cases, permanently installed or portable lead shields around the chambers are required to obtain the desired directional reading, and (3) substitution of a scintillation crystal and photomultiplier tube unit for the ion chamber may be desirable in some cases to permit selective measurement of certain gamma energies. Considering the required ruggedness and durability, the suitability of the scintillation unit for canyon service may be questionable.

Alternate Diluents for Purex Solvent

On the basis of an 18°F higher flash point and otherwise equivalent properties, "Ultrasene", a special kerosene marketed by the Atlantic Refining Company, has been recommended as a replacement for the Amsco 123-15 diluent. The program for procurement of samples of substitute diluents having higher flash points and less tendency toward fission product pickup and emulsion formation is being continued, and several samples were ordered for evaluation at KAPL.

Final Purification of Uranium Product Stream

The silica gel process for removal of residual zirconium and niobium activities and particulate material from the aqueous uranium product from solvent extraction employs oxalic acid as a column regenerating solution. Brief experiments are being conducted at the Engineering Research Laboratory to determine the corrosion rate of stainless steel subjected to boiling alkaline solutions of sodium oxalate. These experiments will indicate the feasibility of neutralizing and evaporating the oxalic acid waste from the proposed silica gel process. Neutralization and evaporation of this waste appears preferable to destruction of the oxalic acid with $KMnO_4$ or other oxidizing agent.

Long Fuel Element Handling

On the basis of holding to a minimum the amount of contamination in the operating areas, it has been proposed that the future long fuel elements will be handled in the 200 Area as bundles of uncut, unbent, jacketed elements. The Engineering Department has been asked to make a design layout study of a new type of dissolver that will handle fuel elements of the maximum length commensurate with the present reactor design.

Savannah Separations Pilot Plant

On January 12 an evaporator explosion occurred during the final stage of deacidification of a batch of nitric acid - uranyl nitrate solution. A separate report covering this incident has been issued.

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Jet Studies

Additional prototypes were tested and found satisfactory for the organic rate jet and the high-head 25 gpm aqueous jet. As a result of these prototype tests, a jet has now been recommended for each specified 200 Area service.

The first plant production jet (25 gpm) was tested and its performance duplicated that of the prototype jet previously accepted for this service.

Unstable rate control resulted during mock-up tests of the original rate feed-jet system. A modification in the mock-up piping has been made which permits the rack piping to run full without venting. The changes resulted in satisfactory feed control to the bank during a series of tests. On the basis of these tests the Design Division will redesign the plant piping for the rate feed-jet system.

Head-End Studies

The redesigned centrifuge bowl was tested and the results were equivalent to those of previous tests.

Cake removal nozzles designed by ERL and those originally designed by the TNX Semiworks have been compared for thrust and spray pattern by using a bench test apparatus. Although the ERL nozzles gave some additional thrust at equal water pressure, the difference was not considered sufficient to affect the ease of cake removal or to permit a significant reduction in water pressure.

Oak Ridge National Laboratory

Laboratory Section

Experiments by the Laboratory Section support the explanation that small amounts of fluoride ion in the system were responsible for the low absorption of Zr and Nb on the resin at the time plutonium was being absorbed from the first of the Pilot Plant's runs in which 60-day-cooled feed was used. The 60-day-cooled runs followed the processing of metallurgical recovery feed which contained 0.16 M fluoride ion. The laboratory showed that 0.005 M fluoride in the uranium elutriant was effective in removing the bulk of Zr and Nb absorbed on an experimental bed. The amounts of fluoride involved have not significantly increased plutonium losses. Although no further work is now scheduled, it would appear desirable at some time to investigate more completely the feasibility of deliberately adding fluoride to the ion exchange feed, or to one of the elutriant streams with a view to reducing activity buildup on the resin and improving decontamination.

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The Laboratory Section has developed a combination ion-exchange and electrolytic process to convert uranyl nitrate to the tetrafluoride, and has demonstrated the major steps using plastic equipment. Their rough cost analysis indicates that the conversion would cost 25 cents per pound of uranium. The process involves the absorption on cation resin of uranium from a 50 g./l. dilute nitric acid solution, quantitative elution with 4 M HF to give UO_2F_2 at a concentration of 150 g./l., and electrolysis in 3-4 M HF to give a 99% conversion to UF_4, which is then centrifuged and dried. The major development difficulty anticipated is to find equipment which meets corrosion requirements and is otherwise practical and available.

In an effort to throw some light on the recent rapid oxidation which occurred in the 678-G evaporator at Savannah River, a few laboratory experiments have been carried out in which the uranium solvent extraction product was gradually heated with 30% TBP and 100% TBP. Upon heating and evaporation to the zone of denitration, a reaction appeared to take place as evidenced by a temperature rise from between 150° - 170°C to 220°C within one minute. In one experiment black flecks of partially destroyed organic matter were found in the residue.

Pilot Plants Section

The fifth and final run with 60-day-cooled feed has been completed. A reduction in the first-cycle uranium feed rate to give a low (50 vs. 75 normal) percent saturation of the organic phase reduced the gamma decontamination by ten fold over the I-A column which bears out other experience that high saturation of the organic phase by itself contributes to good decontamination over the I-A column. A third uranium cycle of decontamination was employed in this run and provided a gross gamma D.F. of about 40; because of the poor first- and second-cycle decontamination, the value is much higher than might be expected in normal operation. First-cycle solvent recovery employing a carbonate-caustic system gave gross gamma decontamination of 18 as compared with 6 for a water-carbonate system. Decontamination of Purex Pilot Plant equipment is under way and will be a joint and well-documented effort by the Pilot Plant and Unit Operations' Sections. Following decontamination, Purex Pilot Plant equipment will be left in place for possible future use and Thorex equipment will go into presently unused cells.

Knolls Atomic Power Laboratory

Purex Program

Semiworks activities during January have provided additional demonstration of improved plutonium UX decontamination effected by recent KAPL modifications in the second cycle solvent extraction flowsheet. These flowsheet changes have been substantially incorporated in the tentative plant

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startup process. The modifications include two-fold increase in concentration of the Purex effluent, facilitating subsequent operations.

Initial semiworks trial of an ion exchange process for coupling the first cycle plutonium effluent directly to the button line was completed smoothly from an operational viewpoint. Gamma decontamination by ion exchange, however, was considerably below the performance which has been consistently demonstrated in the laboratory; overall decontamination accruing from head-end, solvent extraction, ion exchange, and two peroxide precipitations was 10^6, rather than 10^7 as anticipated. Elimination of the second Pu extraction cycle is of particular interest in connection with reduction of separations waste volumes. The study of ion exchange coupling of the first cycle product will, therefore, receive continued emphasis.

Despite relatively poor head-end performance, excellent solvent extraction decontamination has been obtained in the four recently completed semiworks runs. A thorough check of head-end equipment has been completed, and procedures streamlined to provide maximum significance to the plant processing aspects of future runs.

Laboratory studies, including several successful process demonstration runs in mixer settlers, indicate that it is chemically feasible to replace ferrous sulfamate with a combination of hydroquinone and sulfamic acid as plutonium partitioning agent. Use of an organic reductant has certain inherent advantages, as well as possible disadvantages. Since elimination of the use of an iron salt is desirable from the viewpoint of ionic purity of the uranium, as well as smoothness of plutonium coupling operations, this investigation will be actively pursued. The other items of major interest on the current laboratory program are:

1. Determination of the specific chemical and physical properties of TBP and diluent essential for minimum fission product contamination of Purex solvent.

2. Search for a practical method of eliminating the acidic evaporation step between the two uranium extraction cycles.

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V. INSTRUMENT DEVELOPMENT

Savannah River Laboratory

Counting Room Instruments

A catalog of equipment for use in the SRP counting rooms is being prepared. Each item is being tested and samples are being prepared for exhibition to the prospective users. The first SRP production scaler has been received and put into service. Several defects were located and corrected.

Process Water Monitor

The four channel count rate circuit and recorder for use in the Hanford injection tests was built, checked out, and shipped to Hanford.

Scintillation Probes

Samples of the new DuMont photomultiplier tubes were received and tested. Initial tests showed very superior resolution properties compared to the RCA 5819's. It appears probable that all pulse height analyzers should use the new tube.

Dimple Water Monitor

With minor modifications, the A. M. & F. dimple water monitor will be satisfactory. The required sensitivity to both alpha and beta-gamma has been achieved. The alpha measurement currently requires the use of poor statistical accuracy which would result in unnecessary alarms in use. A new alpha probe has been tested which increases the geometry five-fold and should correct the statistical error deficiency.

Blackness Tester

The blackness tester sensitivity was increased with the large Building 305 neutron source to the point where the alloy content could be determined to $\pm 0.05\%$ in less than two minutes. Density and chemical analysis correlate with the "blackness". The blackness tester will be applied in the Building 320 process as a Go - No Go test of the finished producer slugs. A plant type prototype is being designed.

[redacted]

[redacted]

Pin Monitor

Preliminary experiments have demonstrated a device for automatically locating a "hot" spot. Commercially available TV accessories may be directly adaptable to this problem.

Sweeping PHA

Two somewhat different sweeping pulse height analyzers are being assembled by minor adaptations of existing equipment. One goal is an analyzer for routine use by the chemists, while another is to provide basic data for further work on the process water gamma monitor.

[redacted]

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