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August 10 - 13, 1959

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AUTHOR
K. J. Schneider
G. L. Richardson

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TRIP REPORT

BROOKHAVEN NATIONAL LABORATORY, ARGONNE NATIONAL LABORATORY, AND IDAHO CHEMICAL PROCESSING PLANT

August 10 - 13, 1959

By: KJ Schneider
GL Richardson

Chemical Engineering Development
Chemical Development
Chemical Research and Development
HANFORD LABORATORIES OPERATION

August 24, 1959

HANFORD ATOMIC PRODUCTS OPERATION

RICHLAND, WASHINGTON

GENERAL ELECTRIC

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TRIP REPORT - BROOKHAVEN NATIONAL LABORATORY, ARGONNE NATIONAL LABORATORY, AND IDAHO CHEMICAL PROCESSING PLANT August 10 - 13, 1959

INTRODUCTION

The authors visited Brookhaven National Laboratory on August 10, Argonne National Laboratory on August 11 and 12, and Idaho Chemical Processing Plant on August 13. The objectives of the trip were to obtain semi-detailed information on results of simulated waste calcination studies and to obtain engineering information on other high temperature techniques including fused salt, liquid metal, and volatility processes and systems. In addition, information was obtained on the ICPP Weiss-type countercurrent ion exchange unit.

BROOKHAVEN NATIONAL LABORATORY

Rotary Ball Mill Calciner - L. P. Hatch, R. F. Domish, E. J. Tuthill

The operation, characteristics, and performance of the six-inch diameter by thirty-six-inch long ball mill rotary calciner were discussed. The equipment, which consists basically of an electrically heated calciner, a vertical down-draft condenser, a relatively large condensate tank (which also serves as a condenser off-gas surge tank), and a small bubbler-type water scrubber, has been adequately described in BNL reports. Major emphasis on work with this calciner has been to determine over-all operability and to obtain general data on particulate entrainment in the off-gas with simulated aluminum-containing waste solutions. The effects of various operating variables on product quality, off-gas entrainment, etc., have not been completely defined to date. In general, product from this calciner has been of relatively small size, with sizes ranging downward from a maximum of about forty microns. Despite these small particle sizes, over-all solids decontamination factors are in the order of 4×10^{6} to 1×10^{10} with aluminum-containing solutions. Several short runs have been performed using a simulated Purex 1WW solution with low salt content. Over-all decontamination factors during calcination of this solution were 2×10^{6} to 6×10^{6}, somewhat lower than with other solutions.

Results of tests performed so far indicate that the ball mill calciner has a lesser off-gas entrainment problem them some other types of calciners. In addition, operating efficiency, versatility of operation, and long-lived integrity of the equipment are claimed to be at least as good as with other types of calciners. However, reproducibility of results and control of calcine quality has not yet been attempted. The only major difficulties encountered have been excessive corrosion and some caking on the feed nozzle.

The six-inch calciner unit is currently being dismantled and an eight-inch diameter unit is being installed. The new unit will have a total heated length of six feet with three individual temperature control zones and a total heat capacity of about 40 kilowatts (furnished by radiant electrical heaters). The off-gas system will include cyclones, a down-draft condenser, a water scrubber, and a filter. The calciner will incorporate the use of specially-designed steam-purged seals and a water-plus-air-cooled feed nozzle. Blueprints of the process flow diagram, the end seal assemblies, and the feed line assembly were given to the writers.

The unit is scheduled for start-up in about three months. "Cold" testing of the equipment will be made to determine detailed operating features, to control product particle size, and to determine capabilities with various feeds. One or more of these runs will probably be performed with a high-salt-content simulated Purex 1WW solution. After completion of the "cold" runs, the entire unit will be shipped to ORNL where it will be operated with spiked radioactivity.

One short run was performed with simulated high-salt-content Purex 1WW solution in an open-end four-inch diameter ball mill calciner used for crude testing. Considerable caking was encountered, thereby indicating that abnormal difficulties might be expected with this type of feed.

Ion Exchange of Fission Products on Clays - L. P. Hatch, E. J. Tuthill, R. F. Domish

Some of the work on ion exchange of long-lived fission products on clays (including clinoptilolite) was discussed briefly. Experiments using citric acid to complex iron at low concentrations (ca. 0.005 M) prior to ion exchange of nitrate solutions onto clays have been relatively successful. It was agreed that iron concentrations more closely approximating that in current Purex 1WW solution would be tried. In addition, sulfate ion will be added to the solution to determine if sulfate has a detrimental effect on the ion exchange.

Fluid Bed Hydrochlorination - L. P. Hatch, W. E. Regan

Work has been started on a proposed two-step fluid-bed process for hydrochlorination of zirconium cladding or zirconium-uranium alloys, followed by fluorination of the uranium to UF_6. The technique tested thus far involves inserting a ten-inch length of fuel element (with geometry similar to that of an MTR fuel element) into the reactor containing sand or steel shot as bed material. The bed is fluidized by gaseous HCl and is maintained at a temperature of about 400° to 500°C by control of forced convection cooling air flow rate around the external periphery of the reactor. The volatilized $ZrCl_4$ is then condensed. It is proposed that the uranium can then be removed as UF_6 using the same technique with fluorine gas. Early results are encouraging with uranium waste losses on the order of 0.5% or less indicated and complete removal of refractory zirconium oxide films in about 20 minutes.

The reactor consists of a stainless steel vessel six inches in diameter and twenty-six inches long with a three-inch external annulus containing vertical copper fins. Sand and 140 to 200 mesh steel shot have been used as bed material. Sand was found to be inferior to steel shot because of a slight tendency to plug the narrow fuel channels (about 0.1 inch). The estimated capacity of the reactor is 10 kg/hr of zirconium. Measured heat transfer coefficients were 40 to 60 BTU/(hr)(ft^2)(°F) from the bed to the wall and ca. 400 BTU/(hr)(ft^2)(°F) from the element to the wall. The off-gases are passed through a porous-metal filter with blow-back provisions to remove entrained UCl_3.

Fluoride Volatility Process - G. Strickland

The AEC has approved the construction of a pilot plant to demonstrate a new

process developed to eliminate the potentially dangerous reaction of BrF_3 with uranium metal. In this process, the entire fuel element is dissolved in a liquid mixture containing 15 to 25 mole percent NO_2 in HF at 100 to 120°C under a pressure of about 100 psi. This dissolvent readily attacks all present cladding and alloying materials at rates ranging from 10 to 1000 $mg/min/cm^2$. The products are complex salts, probably containing nitronium radicals. The zirconium salts are only slightly soluble, thus affording a means of separation by filtration. Subsequent process steps involve evaporation to recover the NO_2 HF solvent, oxidation of the uranium salts to UF_6 with BrF_3 and fractionation of the UF_6. Inconel and Monel appear to be adequate materials of construction for all phases of the process.

Advantages over other volatility processes are the elimination of BrF_3 contact with uranium metal, low operating temperatures, and existence of a suitable construction material as compared to the fused salt volatility process. The disadvantage, of course, is the high operating pressure.

The explosion of their earlier continuous BrF_3 dissolver has been tentatively attributed to dissolution of the gamma-extruded fuel element in a manner to produce hollow or perforated elements. Thus reaction could take place in the interior of the metal where heat dissipation was inadequate. The result was ignition and explosion, apparently as a chain reaction. A highly satisfactory detonation was achieved in the BNL ignition test facility in demonstration of this theory.

Fused Salt-Liquid Metal Extraction Columns - L. E. Kukacka

Loop N was constructed to demonstrate continuous countercurrent extraction columns for the removal of fission products and uranium from LMFR fuel (liquid bismuth containing about 1000 ppm uranium, 300 ppm magnesium, and 250 ppm zirconium). The extractant is the ternary eutectic NaCl-KCl-$MgCl_2$, which melts at 396°C. Unfortunately, a cutback of AEC funds has delayed operation of the loop, although they are hopeful of starting it up in the near future.

Some of the salient details of the equipment were obtained. Two extraction columns are included in the loop. One is two inches in diameter packed with 1/4-inch stainless steel balls; the other is four inches in diameter containing a disk-and-doughnut-type packing.

The salt phase will be the continuous phase; the bismuth is to be introduced as a spray in the gas phase. The bismuth flow rate will be 0.5 gpm, the salt rate about 0.1 gpm. This is believed to be near the capacity limit of the two-inch column and far from the flooding capacity of the four-inch column.

The designs of the columns were based on disperson studies with a mercury-water system. Mercury drops dispersed in a variety of ways into water appeared to maintain their initial size throughout the column, possibly because of little coalescence. Smaller drops were obtained by spraying the mercury into the gas phase. Backmixing studies with a dye tracer lead them to believe that each contactor may be equivalent to only one theoretical stage.

The vessels are constructed of a Croloy alloy (bismuth side of loop) and 347 stainless steel (extraction columns and salt side of loop). Heat is supplied primarily by Nichrome resistance wire with a sufficient number of circuits to provide isothermal heating and to permit melting of frozen salt from the top down to relieve pressure from expansion. A typical heat input is 250 watts per foot of one-half inch pipe. The system operates at 500°C and is insulated with about three inches of magnesia. The temperature is controlled by a second thermostatically-controlled one-off series of heating wires.

Flow measurement is by orifice meter or by liquid level measurement, using a conductivity probe which can be lowered to the liquid surface. The column interfaces are jackleg controlled. The columns are fed by constant-head tanks equipped with throttling valves. Magnetically-coupled pumps, described in BNL-506, are used to fill the feed tanks.

Sampling Device - G. Farber

A simple sampling device for obtaining samples of molten salt or metal from a vacuum or inert-gas-blanketed vessel was demonstrated. A small cup is suspended on the end of a long rod. The other end serves as a magnetic armature and fits into a long sleeve pipe which is flanged at the lower end and sealed at the other. A series of movable magnets which slip up and down the outside of this sleeve serve to raise or lower the armature and, hence, the cup. The flanged end of the sleeve is bolted to an air lock on top of the vessel to be sampled. Two Worcester ball valves are used in the air lock.

ARGONNE NATIONAL LABORATORY

Fluid-Bed Waste Calcination - A. A. Jonke, J. Loeding, L. J. Anastasia

Operation of the six-inch diameter fluid-bed calciner was discussed with emphasis on their tests with simulated Purex 1WW solutions. During the past few months, simulated Purex 1WW solutions, either "as-produced" or formaldehyde-killed plus three-fold-concentrated, have been calcined at temperatures of 350, 400, and 500°C. The biggest problem has been control of excessive agglomerate formation. This problem decreases with increasing temperature but is not yet satisfactorily solved. Improved feed nozzle design (i.e., with an extended liquid nozzle tip) has nearly eliminated caking on the nozzle tip but has not adequately reduced agglomerate formation. Future runs will be designed primarily to reduce agglomerate formation by revised feed nozzle designs and by the use of air-jet attrition methods. Other testing may include effects of higher temperatures, effects of varied feed composition, and operating performance with spiked radioactivity. The work with Purex-type waste solutions at ANL is tentatively planned to be discontinued when the fluid-bed calciner at HAPO is operating reasonably well.

Four extended runs with simulated Purex 1WW solutions as feed (at feed rates of about 6 liters/hour) have been completed. These runs were 8, 13.5, 21 and 30 hours in duration. In the last run, about 1.6 scfm of high-pressure attrition air was used; and although the particle size of the final bed was relatively large (60% +20 mesh), the particle size distribution remained constant after about 20 hours of operation. The product from this last run had

an untapped and tapped bulk density of 1.38 and 1.55 respectively. No pluggage within the feed nozzle has been experienced with Purex waste solutions. The operation of the ten sintered stainless steel bayonet filters in the calciner de-entrainment section has been excellent. The blow-back system, which provides an 80-psig air spurt for about 0.1 second every one to one and a half minutes for each of the filters, has kept the filters relatively clean (ca. 1/8-inch thick coating of fines) with no increase in pressure drop during all of the runs. Chemical stability of the formaldehyde-killed and three-fold concentrated Purex 1WW solution was found to be on the order of two days to two weeks.

Gasket materials used successfully at the nozzles are Durabla (an asbestos-base material) and platinum. For the large gaskets in high-temperature service, stainless steel Flexitallic gaskets with blue asbestos filler have given good service. High-temperature pipe joint compounds used successfully are Silver Goop and Felpro.

The following design prints of ANL equipment were obtained by the authors:

1. Several feed nozzle designs.
2. Top head assembly and details.
3. Fluidizing gas distributor plate assembly.
4. Calciner assembly and details.
5. Off-gas filter blow-back venturi details.

Numerous details were discussed, such as HAPO pilot-scale calciner design and plans, start-up, operating, and shutdown procedures and techniques, feed composition and make-up techniques, feed flow control systems, detailed design considerations, etc.

ADF Process - General Fluidization Studies - N. Levitz

The ADF Process uses successive fluidized beds to dry an aqueous HF solution of a low uranium, high zirconium fuel and to recover the uranium as UF by fluorination of the dried residue. The uranium recovery is not yet consistently as high as required (typically 95 to 98% recovery) unless the fluorination temperature is raised to about 700°C at which point ZrF_4 volatilizes. Drying parameters are being investigated to pinpoint the effects of temperature, residence time, etc. on the uranium recovery during subsequent fluorination.

A number of general fluidization studies are currently being undertaken and were discussed briefly. Among these are fluidization and mixing in tapered fluidized beds, studies of particle growth in fluidized bed calciners, attrition due to air jets in fluidized beds, measurement of bed particle size by differential pressure measurements, etc. Details of these studies are contained in recent ANL quarterly reports.

Fused Salt Volatility Process - W. J. Mecham

A graphite dissolver for dissolving Zr-U alloy fuels by hydrofluorination in a fused zirconium fluoride-sodium fluoride melt has been installed and tested. All parts of this dissolver in contact with molten salt during HF sparging are graphite. The transfer lines and the top flange of the dissolver are Monel.

Heat is supplied to the dissolver by graphite resistance heaters (ANL drawing No. CE-4200).

Transfers are made by pressurizing the vessels. Salt transfer lines are auto-resistively heated and use several special techniques. To prevent the salt freezing as the line passes through the water cooled dissolver flange, the line enters through a well (details shown in drawing No. CE-4150). Freeze valves are provided in the salt lines to prevent loss of HF during sparging. These are basically down-turning loops in the line. On the upstream side of the loop is an argon sparge to prevent siphoning; on the downstream side, the line enters an elevated trap which holds enough salt to fill the loop. When the transfer is complete, and the sparge is shut off, the salt in the trap drops into the loop. With the heat off, the salt freezes and produces an adequate seal. Additional heat must be supplied to flanges and the loop trap to prevent cold spots.

Work is continuing on the frozen wall concept for containing the molten salt-UF_4 mixture during fluorination. The present unit is heated by electrolytic heaters. Each 3/4-inch copper electrode is encased in a two-inch nickel pipe and brazed to the pipe at the bottom. This method of construction prevents short circuiting of the current at the top of the melt, thus providing more uniform heating. Resistance heaters are also enclosed in each pipe to melt the initial pool. The heat input is controlled by the electrical resistance measured between the vessel wall and the electrodes. This measurement can be calibrated to indicate the minimum frozen wall thickness. The vessel is externally cooled by water cooling coils. The power is supplied as 20 to 30 volt alternating current at a level of about 10 kilowatts. An inert gas sparge is used to improve mixing.

Prometallurgy - L. Burris, Jr., H. M. Feder

Melt-refining is the process proposed for EBR-II fuel decontamination. About 5 to 10% of the uranium charged to the melt crucible is left behind in the slag. It was proposed to recover the uranium from this by a Pyrozinc process - dissolution of the slag in zinc or a zinc-magnesium mixture. However, their emphasis now is on oxidation of the slag and dissolution of the oxide by a mixture of cadmium and magnesium. The chief advantage of cadmium is that it can be contained in mild steel, whereas molten zinc requires a non-metallic container. The use of a flux is being considered to dissolve MgO formed during dissolution. A commerical flux, such as one of the Dow fluxes, was highly recommended for a service such as this, since these fluxes are tailored especially to have certain desirable physical properties.

Some laboratory development work is being done using a low melting, high calcium alloy as a solvent for plutonium and rare earths. Details of this process were not obtained.

Inductive heating is used extensively. Tocco 15 kilowatt, 10,000 cycle motor generators mounted on wheels were used to service several experimental set-ups. Transfer of molten metal is primarily accomplished by pressurizing the vessels; however, they had just completed a loop test using a GE magnetic pump.

Prints of a typical tilt-pour furnace and magnesium distillation unit were obtained.

IDAHO CHEMICAL PROCESSING PLANT

Fluid-Bed Waste Calcination - E. W. Grimmett, D. M. Paige

Discussions were held on recent progress and future plans in the fluid-bed calcination field at ICPP. The majority of the development efforts during the past year have been with the two-foot by two-foot square calciner. The NaK heating system has been checked out and operated successfully over the past year. The system has been relatively trouble free and has provided good temperature control. However, a leak in the inlet header to the calciner tube bundle just outside the calciner developed recently, producing most of the expected fireworks. Inspection of the failure showed that corrosion (both inside and outside) of the tube bundle was negligible. The failure was attributed to excessive thermal stress and/or fatigue caused by the many start-ups and shut-downs. Failures occurred where two of the tubes were welded to the tube header. The failed sections will be replaced with new ones of revised design which will allow for thermal stresses.

A major point of interest brought out was that they have never successfully maintained constant conditions in their fluidized bed calciners during extended runs lasting for up to five weeks. Conditions have been maintained within certain reasonable and operable ranges but not as close as desired.

A theoretical equation was derived relating the particle size distribution in a fluidized bed calciner with particle density, particle growth rate, feed rate, bed weight, size and number of seed particles. The equation has been tested by making some runs (in the two-foot square calciner and in the six-inch diameter calciner) using radioactive tracers to determine particle growth rate and substituting the remaining values into the equation. The results indicate that the equation gives a fairly good correlation. The equation and most of the results of the tests are forthcoming in their quarterly progress report ending March of this year. Particle size distribution within the bed has been found definitely to be a function of the feed nozzle air-to-feed flow ratio, with increasing ratios resulting in smaller particles. Whether this is due to increased attrition or better atomization at the higher ratios, or both, has not yet been determined, but attempts to obtain this answer are planned. To maintain reasonable particle size distribution, air-to-feed flow ratios greater than 500 and 200 are required in the nozzles used in the six-inch calciner and in the square calciner respectively.

Work on fired-tube heating of a calciner has been discontinued since last fall when the NaK system was first put into operation. However, the results of the work last year were encouraging, so studies are to be resumed soon using small GE jet engines as burners. Results of last year's work with fired-tube heaters are presently being documented.

The Inconel-sheathed electrical, cartridge-type resistance heaters in the six-inch calciner have not visually indicated significant corrosion. No lifetime tests have been made with the elements, and sheath temperatures have not been determined.

Platinum has been used successfully for feed nozzle gasket material, and an

asbestos base material (US Rubber No. 17020) has been successfully used for large high-temperature gaskets. Silver Goop is used as a high-temperature pipe joint compound.

Some testing has been done with a small-scale wetted-wall electrostatic precipitator. Current operability and efficiency data at relatively low voltages (less than 10,000 volts) are encouraging to the point that considerable future work is planned along this line. No data have yet been obtained on the performance of an AEC-type of filter in the off-gas system of the six-inch calciner, but testing is underway.

A rebuilt calciner system is contemplated to replace the existing six-inch unit. The proposed unit will be about twelve inches in diameter with internal electrical resistance heating elements. The off-gas equipment will probably include a cyclone, a venturi scrubber, a condenser with distillation of the condensate, an electrostatic precipitator, a rotating blower (with recycle of some blower gas as fluidizing gas), and an absolute filter. The new system will permit evaluation of a different off-gas train from that currently being tested. Steam will also be tested as fluidizing gas in the proposed system.

A number of details were discussed, such as operating procedures, detailed design considerations, HAPO calciner design bases and plans, etc. A design sketch of a pneumatic operator for atomizing feed nozzle clean-out needles was obtained by the authors.

Fluid-Bed Waste Calcination Demonstration Facility - J. I. Stevens

Some of the design bases of the demonstration unit fluid-bed waste calciner facility were briefly discussed and the site was visited. The main calciner building and the calcined waste storage vault were nearly completed, but little piping and tankage have been installed.

A tentative appraisal of a fired-tube heating system compared to the NaK heating system to be used in the demonstration facility has been made. Preliminary calculations indicate that approximately twice the heat transfer area and several times the power are required to operate a fired tube heating system compared to a NaK system.

Design prints of the ruthenium adsorbers and the calciner assembly and details were given to the authors.

Dissolution Processes - J. A. Buckham, C. M Slansky, H. T. Hahn

A rather cursory inspection of laboratory and pilot plant dissolution processes and equipment was made. The pilot plant effort is devoted to long tube continuous dissolvers for enriched alloy fuels such as Al-U, Zr-U, and Cr-Ni-U alloys. The dissolvers were two to four inches in diameter, about ten feet tall. Processes being studied were mercury-catalized HNO_3 dissolution for aluminum, HF-HNO_3 (in Carpenter-20, heat-treated equipment) for zirconium, and Darex for the Cr-Ni alloy fuel. The Carpenter-20 dissolver was still under construction but should be operable soon. A new HF-HNO_3 resistant construction material, a platinum iridium alloy used as a 10-mil-thick lining, is being

evaluated. This material is supplied by J. Bishop and Co. Platinum Works.

A more exotic dissolution process (the Arco Process) is being developed in the laboratory. They propose to dissolve Zr-U alloy in molten $PbCl_2$ at 525°C. The lead pool formed extracts the ruthenium (probably the other noble metals also) while the UCl_3 and remaining fission products stay in the salt phase. The zirconium is volatilized as $ZrCl_4$ in a minimum of dilution gas. The salt mixture is then dissolved in water or dilute HCl where the $PbCl_2$ is precipitated. The solution is then acidified with HNO_3 and the excess HCl driven off. The resulting UNH solution is used as solvent extraction feed. The lead metal can either be discarded or dissolved in nitric acid, precipitated with HCl, and recycled together with the $PbCl_2$ from the UCl_3 dissolution step.

Countercurrent Liquid-Solids Contactor - Blake Brown, E. S. Grimmett

Two modified Weiss units have been designed and used for countercurrent alumina-leaching studies and ion exchange. These differ primarily only in diameter, one being two-inch and the other four-inch. The basic features of these units are the following:

(1) The vertical columns contain (typically) five to eight seive plates, spaced about five and a half inches apart, each with 0.079-inch diameter holes, 41% free area. The plate contains downcomers extending about four inches below the plate and two and a half inches above, giving an immersion of about one inch into the resin bed on each plate.

(2) A 3/8-inch deep bed of 3/32-inch ball bearings is placed on each plate to prevent resin from passing through the holes, while at the same time preventing excessive pressure drop for the pulsed liquid flow through the plate.

(3) An approximately sinusoidal pulse is applied to the column contents at an amplitude of 1/8-inch and a frequency of 500 to 700 cycles per minute. This pulse fluidizes the resin bed and expands it to about two and one half inches above the plate, or to the overflow of the downcomer.

Typical capacities of the units are ca. 250 to 300 lbs or Dowex 50W resin per hour per square foot at water flow rates up to 2,000 $lb/hr/ft^2$ for Al_2O_3 and 1100 $lb/hr/ft^2$ for the resin. Efficiency studies on copper ion exchange are now being conducted. The operation proceeds very smoothly.

Photographs of the column and blueprints of the pulse unit were obtained, as well as some literature on a SWECO separator which is used to separate the resin leaving the column from accompanying water.

GL Richardson
KJ Schneider /kb