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TECHNOLOGY - HANFORD PROCESSES
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TECHNICAL PROGRESS LETTER
FOR
SEPTEMBER 1950

COMPILED BY MEMBERS ON THE TECHNICAL DIVISION:

OCTOBER 5, 1950

HANFORD WORKS
RICHLAND, WASHINGTON

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Technology Hanford Process
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This document consists of 12 pages [redacted]

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HANFORD WORKS
RICHLAND, WASHINGTON

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PILE TECHNOLOGY - W. K. Woods

PHYSICS - P. F. Gast

In preparation for operation of the DR pile, charging of uranium metal was begun during September. The critical size of the pile with no cooling water in the tubes was found to be the same as that of H Pile under the same conditions.

The critical size of a pile containing cooling water was determined for the first time since the initial loading of B Pile. The observed reactivity effect of the water at DR was found to be in good agreement with the original B Pile measurements and to amount to 2% k.

A reactivity coefficient test was performed in the small wet pile. This will be compared with a coefficient test in the fully loaded pile to determine the effect of neutron leakage on the coefficients.

The H-10 loading was effectively completed with the special loading of 796 tubes in a pattern which encloses 846 tubes. The rate of reactivity loss has been moderate, and it is indicated that no enriched uranium will be required to compensate reactivity losses as exposure continues.

A loss of 70 ih has been observed in the reactivity of D Pile. This loss has been associated with a high rate of moisture removal from the circulating gas of the pile. The source of the water leak has not been discovered.

Critical masses of plutonium solutions have been determined in 12 and 13 inch diameter spheres with full water tampers and interpretation of the data is in progress.

The precision of positioning of the control rod in the Test Pile was greatly improved during the month by changes in the mechanical system.

ENGINEERING - G. E. McCullough

In order to reduce expansion of the H Pile, modifications were made in the graphite structure so that tube bearing graphite blocks could expand without distorting the pile. With this modified structure the expansion of the pile as a whole is determined by the expansion of the non-tube blocks until the point is reached at which the expansion of the tube bearing blocks has closed the original gaps which were provided. Measurements of the pile structure during the past year indicate that the H Pile expansion will be very low in comparison to that experienced at the B, D and F Piles as long as the rate of expansion is controlled by the non-tube bearing blocks. During the past month samples of graphite were mined from the bore of process tube bearing blocks. X-ray measurements of these samples indicate that these blocks have expanded at a rate which, if continued, would require about 13 years to close the gaps around the blocks.

The stainless steel nozzles at the D Pile were replaced with aluminum nozzles during the latter part of August and the first week of September. This was done to reduce the serious corrosion of the ends of the aluminum process tubes which was caused by the galvanic couple between the aluminum and stainless steel. Similar replacements will be made at the B and F Piles as fabrication of the new nozzles is completed.

A serious leak of water into the graphite of the D Pile became evident shortly after the long shutdown for nozzle replacement. Removal of water from the pile atmosphere by the dryers has been between 100 and 200 pints per day. Operation of the pile has been continued although temporary reductions of power level have been required. Various methods of locating the leak have been unsuccessful.

The Naval reactor high pressure water recirculating test equipment (ANL-140) was given a final 48 hour test run at 1500 psi and 550°F, following which the pressure tube was installed in "A" Hole of the H Pile during the pile shutdown of September 4-8. The equipment with a dummy charge has been operated satisfactorily during pile operation using pile process water as coolant and also with normal recirculation of distilled water at 1500 psi and 540-550°F, for 210 hours.

METALLURGY - R. Ward

The average reactivity of canned process material has shown no improvement during the past month. Tests designed to determine the causes for the low reactivity have been delayed due to the overhauling of the 305 pile.

Observations made on the gas analysis line indicate that the inconsistencies in the amount of hydrogen found in various P-10 target slugs may be caused by hydrogen diffusion through the hot furnace tube walls. The apparent hydrogen content of a slug can be increased by increasing the humidity adjacent to the outside of the furnace tube wall.

X-ray orientation studies on uranium, rolled at temperatures (1150°F and 950°F) which may represent the extremes in the present rolling process, show that the degree of preferred orientation changes markedly in this range.

P-10 OPERATIONS - J. C. Chatten

During September, 1950, 144 slugs were extracted in 33 batches and one batch separation of accumulated outgas fraction was made. Two of the batches were inadvertently air-contaminated and eight other batches had product purities below 90%.

Two glass blowers received internal contamination in excess of the current working limit. The source of personnel contamination is still unknown.

Extraction operations were transferred to the evening and night shifts during the month, to permit erection of a 300 foot stack during daylight hours by construction personnel without protective equipment.

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New facilities permitted welding of canned slugs to be performed in Bldg. 108-B, rather than in 300 Area, during September. This avoids the possibility of contamination by atmospheric humidity during shipment of unwelded slugs to 300 Area. A total of 3,174 slugs were fabricated (machined, canned, and welded) during the month. Production rates were reduced to 1250 slugs per month at about mid-month and the operating force was reduced to five men. An inventory of 7749 slugs existed on September 25, of which 1130 had been tested and approved for pile use.

P-10 DEVELOPMENT - W. M. Harty

During September the basic health standards for P-10 were revised by the Health Instrument Divisions in a direction to permit major increases in the permissible body limit, permissible atmospheric concentration, and permissible daily stack emission. The new health standards have a profound effect on the P-10 production program, the Hanford P-10 development program, and the supporting activities of the Knolls Atomic Power Laboratory and the General Engineering and Consulting Laboratory. The stripping of P-10 from by-product streams of the production operations is no longer required for health hazard control; stripping for economical reasons remains to be evaluated but this can be developed in a logical manner rather than as an emergency.

As analytical tools are developed further, additional information will be obtained.

The Knolls Atomic Power Laboratory has indicated the feasibility of stripping P-10 from by-product gases using palladium black and uranium. Using palladium black, the product will contain P-10 oxide if the feed gas contains P-10 oxide or air. Using uranium, gradual poisoning is encountered if air is present. Using a combination of both palladium black and uranium, a satisfactory product should be obtained meeting existing specifications in all respects except isotopic purity. During the remainder of 1950, the Knolls Atomic Power Laboratory will continue with the research and development of such a stripper line and work with the General Engineering and Consulting Laboratory in evaluating engineering feasibility. An economic study of the desirability of providing a stripping line then can be made.

Equipment development work on the glass lines is in progress; the first revised line should be completed October 9, 1950.

The design and construction of the metal line by the General Engineering and Consulting Laboratory is proceeding on schedule. Life tests on various pieces of equipment, including the helium operated metal Toepler pump and check valves, are in progress.

WK Woods:jr

W. K. W.

TP-3

PROCESS - J. B. Work

Separations Plant Assistance

The metal solution storage tanks (6-1) used for batch make-up have been recalibrated for use with manometers for weight determination. It is expected that this will provide better control in making up batches.

The production test designed to shorten the lanthanum fluoride by-product precipitation cycle has been temporarily suspended to allow investigation of factors leading to poor and erratic decontamination.

The Metathesis cell (F Cell) has been repiped at T Plant to incorporate the use of two centrifuges. A time cycle of seven hours with a decrease in waste loss has been demonstrated.

The assay of the first product solution in the Isolation Building is now determined by counting methods. This formerly was a chemical analysis.

Purification and Fabrication Plant Assistance

Production Test 234-1, titled "The Destruction of Oxalic Acid and Hydriodic Acid in Oxalate Supernates," was started during the month of September. Solidification of the concentrate in the still on the first run required that the program be delayed till the solids were identified.

Production Test 231-10, Supplement A, as described in HW-18659, was written and completed during the month of September. It was determined by this test work that a load factor of 3.03 ± 0.15 grams per gram of product could be used as the end point for the evaporation carried out in the 231 Building.

Routine sampling of the P-4 solutions in the 234 Building was changed during the month of September from one sample from each batch to one sample from every tenth batch.

A production test, HW-19005, for the evaluation of a skull recovery process, is being circulated for approval. This process involves the dissolution of skulls in a HNO_3 - HF acid mixture and the recycling of this solution through the 231 and 234 Buildings.

Two castings with a four-hour outgassing period, as prescribed in a part of Production Test 235-1, were being processed at month's end.

Coating rejects increased to approximately 35% of throughput during the month of September. Since several pieces were rejected due to contamination at points where the tripod tips touched the pieces in Hood 25, a triple tripod support similar to the one in Hood 26 was also installed in Hood 25. In addition, minor changes in process are being tried in an attempt to obtain a more uniform heat distribution over the piece during coating.

Separations

234-5 Process Development

Although it has been shown in the laboratory that the skulls produced in the casting operation in the 235 Building can be dissolved by boiling in a mixture of 16 M HNO_3 - 0.4 M HF, preliminary corrosion studies indicate that Pyrex glass and 25-12 Cb stainless are attacked markedly by this hot acid mixture.

The use in the laboratory of hydroxylamine hydrochloride instead of HI to obtain Pu(III) from the higher oxidation state led to a bulky and slow settling precipitate when plutonium oxalate was struck.

Cut film and the necessary fixtures to autoradiograph the final product from the 235 Building for coating thickness were received from Los Alamos.

Stack Gas Disposal

The silver reactor and Fiberglas filter to be installed in the B Plant dissolver cell have been placed in mock-up test. At an air flow rate of 100 cfm, approximately 8 hours are required to bring the reactor assembly to the operating temperature (200°C). It has been determined that the equipment will enable the reactor to operate with a high degree of thermal stability under conditions of widely and rapidly varying heat loads, although minor changes in the temperature regulation point are required.

CHEMICAL DEVELOPMENT - R. B. Richards

During the period, all necessary "cold" solvent extraction development for both the Redox and Waste Metal Recovery processes was completed. Further studies for training purposes will be resumed during February, 1951. Several Redox studies were conducted in the Scale-Up unit to test the effect of the IBF "cone" distributor on flooding. While the distributor lowered the flooding point somewhat, the contactor will operate satisfactorily as presently designed. Flooding and losses in the Redox IB and IID contactors were also checked for the effect of ferrous sulfamate and ferrous ammonium sulfate plus sulfamic acid. The results indicate no appreciable effect on either losses or flooding. The latter reducing agent can be purchased directly, thus avoiding the manufacture of ferrous sulfamate in the Redox plant.

The TBP studies were discontinued following a series of runs in the Demonstration Unit (3-in. dia. pulse column) which established the following conclusions: (1) In the RC system, "hybrid" plates (one side coated with polyethylene) having 0.079-in. holes displayed waste losses not significantly lower than those for the present "standard" cartridge containing 0.125-in. holes. (2) The use of "nested" plates (9 plates spaced 1/4-in. apart) with a 5-in. spacing between nests did not improve losses or flooding. (3) A 2-7/8-in. diameter cartridge in the 3-in. column indicated that the diametrical clearance for the plant cartridge may be 1/8-in. without affecting column efficiency. (4) RA and RC feeds heated to 110° ± 10° F. did not produce an appreciable change in uranium losses. (5) Addition of coating removal solution to simulate the silica content of the metal waste resulted in standard behavior in the pulse column. (6) A study with simulated

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supernate produced losses of 4.4% and pointed to a change in pulsing conditions or flow ratios if the plant is called upon to process this material alone. (7) Processing of a simulated "soft" sludge resulted in standard waste losses. (8) The substitution of 0.01 M H_2SO_4 for 0.01 M HNO_3 in the RCX stream produced similar extraction efficiency.

The studies described above (101 TBP runs, 9 Redox runs) were conducted during the first two weeks of the period. The remaining time has been directed toward placing the solvent extraction equipment in a stand-by condition. Several processing steps are being designed for addition to the Demonstration Unit to increase its value as a training unit. Work on the preparation of technical manuals and operating procedures for Redox and TBP is actively under way.

CHEMICAL RESEARCH - O. F. Hill

Diluents for Tributyl Phosphate Extraction Process

The achievement of desired physical properties of the tributyl phosphate system for metal extraction depends on dilution of the TBP with an inert solvent. Criteria in the choice of diluent include viscosity, density, flash point (safety), cost, effect on distribution of metal ions and chemical stability. Chlorinated solvents would probably meet the requirements if an "inverted" system (organic phase heavier than aqueous) were used. Since this is not contemplated for a Hanford process, the commercially available petroleum products appear to offer the greatest promise.

Properties of ten diluents recently investigated included ASTM boiling ranges with initial boiling points from 160°C. to 200°C. and final boiling points from 190°C. to 270°C. and indicated much more selective refining in some cases. Densities varied from 0.754 to 0.804 and viscosities from 9 to 19 millipoises at 25°C. Viscosity of a 15% solution of TBP in such a diluent would vary only slightly above that of the diluent alone. The materials examined showed aromatic and olefins below 1% in most cases and the highest content found was 10% aromatic and 2% olefin.

Extraction and scrub studies were carried out on a feed solution 0.33 M UNH - 3 M HNO_3 prepared from Hanford dissolver solution to determine the effect of the diluent on decontamination in tributyl phosphate extraction processes. One extraction and three scrubs were employed simulating the RA Column in a Metal Recovery or Purex-type process. Shell Spray Base, Gulf BT (Varsol), Stoddard Solvent, and Standard Oil Company Special Base Oil gave decontamination factor values of the order of 10^5 while AMSCO-149 was slightly lower, though still acceptable, with a value of 4.5×10^4. Carbon tetrachloride was somewhat superior to hydrocarbon diluents with a decontamination factor of 3.2×10^5.

Spike studies with various organic impurities which might be expected in kerosene-type diluent revealed no deleterious effects on decontamination for aliphatic olefins, cyclic unsaturates, or for branched chain aromatics. Aromatics and olefins may be objectionable from a chemical inertness standpoint; however, the relative importance of this has not been completely established, especially as to the effect of continued contact.

Separations

No significant difference is found in uranium extraction as the diluent composition is changed.

On the basis of these studies, there is little to choose from among these solvents. However, from an operational standpoint, a low viscosity and low density are desirable while maintaining a flash point in excess of 130°F. Accordingly, for a given extraction column design, specifications for physical properties should be limited to considerably smaller ranges than observed among these diluents.

Bismuth Phosphate Concentration Process Modification

Laboratory studies are being made to determine the feasibility of recycling the solution obtained by rework of the lanthanum fluoride by-product cake to the bismuth phosphate by-product precipitation step rather than to the lanthanum fluoride product precipitation step as is now done. This proposed change would result in materially reducing the total waste volume from the concentration process and in reducing centrifugation time in the lanthanum fluoride product precipitation step. Although all plutonium losses found on a laboratory scale were somewhat higher than those obtained in the plant, the losses obtained when recycling the rework solution to the bismuth phosphate by-product precipitation were not appreciably greater than those obtained when following the current procedure.

Scavenging of Dissolver Solution by MnO_2

The addition of $Cr(NO_3)_3$ (5% excess) to dissolver solution containing permanganate precipitates MnO_2 which is an excellent scavenger for zirconium and niobium. Although the scavenging efficiency of MnO_2 is not a function of the rate of precipitation, it does increase as the acidity decreases. Using 6.5 g/l MnO_2 (same cake volume as 20 g/l Filtrol), the amounts of zirconium and niobium removed are 90.7% and 87.6%, respectively, at +0.58 M HNO_3 and 99.3% and 98.6%, respectively, at -0.1 M HNO_3. Intermediate scavenging efficiencies are obtained at acidities within the above limits. Multiple scavengings of dissolver solution with MnO_2 increases the removal of zirconium and niobium activities such that removal of $>99.9\%$ is possible.

If more than a small excess of reducing agent is added to dissolver solution containing $KMnO_4$, some of the MnO_2 (or MnO_4^-) is reduced to Mn^{++}, thus lowering the quantity of MnO_2 formed. Since some permanganate is reduced to MnO_2 by water or dissolver solution impurities at elevated temperatures, it is desirable to know the actual amount of permanganate present before the addition of reducing agent so that a large excess of the reducing agent is avoided.

Neither plutonium (IV) nor plutonium (VI) are adsorbed by MnO_2 and mother liquor containing plutonium can be removed from the MnO_2 cake by 0.01 M HNO_3 washes. The MnO_2 may be conveniently dissolved in nitric acid containing a slight excess of H_2O_2.

RH Beaton/jw

RH Beaton

ANALYTICAL LABORATORIES - F. W. Albaugh

Mass spectrometric analysis of P-10 by-product has shown somewhat higher tritium contents than those indicated by KAPL and Los Alamos, and has indicated the presence of appreciable quantities of water and organic fractions. It is suspected that the impurities result from improper preparation of sample bulbs and a test has been initiated to verify this. Analyses of by-product and product, together with the determination of ionization factors, accounted for the forty-odd mass spectrometric analyses completed on the two-shift basis during September.

The determination of hydrogen and total gas in canned lithium-aluminum slugs proceeded routinely on a two-shift basis. Approximately one-third of the samples, representative of canned material awaiting test in the Bldg. 305 pile, have been analyzed. Although it is agreed by all parties that the gas density method for assay of product is inaccurate, and should be replaced as soon as possible by mass spectrometric or other reliable procedures, two chemists have been trained in the conduct of this analysis and will be available on a two-shift basis when production operations are resumed. In all, twelve members of the Analytical Section are participating in the P-10 program on a full-time basis.

Continued effort in the development of improved fission product methods has led to modifications of the standard methods for determination of radio zirconium and radioniobium. In both cases the time of analysis has been reduced about four-fold.

Preliminary tests made with the newly received hollow cathode spectrographic excitation source have shown that the unit emits oxygen and fluorine lines. This tends to confirm literature references that the unit has the unique advantage of permitting spectrographic determinations of the nonmetals.

ENGINEERING SERVICES - C. A. Rohrmann

The review of the Rosener preliminary plans and specifications for the Radiochemistry Building was completed, and arrangements were made through D & C for certain essential design changes. These will raise the architect-engineer fee about $3,400, but should result in a construction cost reduction of about $50,000. Most of this saving will result from the adoption of constant-volume type laboratory hoods. No extension in design time will be involved.

A.E.C. approval was obtained for Project C-394, Part II, which covers preliminary construction work on the Hanford Works Laboratory site, and this field work was initiated by D & C.

Technical liaison work continued on the design scoping of the Pile Technology Building, and considerable time was spent with D & C in connection with their estimation of this building's cost as presently planned.

An increasingly large proportion of the total capacity of the Technical Shops (both machining and glass work) had to be assigned to the needs of the P-10

program. Most of the experienced glass blowers were required to work a 6-day week and by month-end plans were completed to place the entire Building 101 Shops on a 6-day week, beginning October 2.

STATISTICAL SERVICES - B.F. Butler

A unique sampling plan developed by the Statistics Group has been used successfully in connection with the reactivity testing of P-10 fuel slugs. This technique permits the rejection of lots containing slugs exceeding specifications, with any desired risk, at the same time requiring no assumptions concerning the variability within a specific lot. Only the average and range of a particular sample are required. Hence, the test is easily applied to production conditions. The principle involved is equally applicable to many other sampling problems. This technique will be submitted for publication in a technical periodical.

LIBRARY AND FILES - C. G. Stevenson

Operations in the Library and the Classified Files proceeded routinely during September.

TW Hauff:mcs