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HW-17679

Technology - Hanford Processes

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# TECHNICAL PROGRESS LETTER FOR APRIL 1950

Compiled by Members of the Technical Divisions

May 4, 1950

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Technology - Hanford Processes

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## PILE TECHNOLOGY - W. K. WOODS

### PHYSICS - P. F. Gast

Studies indicate that a multiple H-10 loading has marked advantages over a single R-10 loading for attaining increased P-10 production. A central loading in two piles of enriched uranium and lithium slugs of the type proposed for H Pile (H-10 loading) will give P-10 production equivalent to the R-10 type loading with only 5% greater investment of U-235. Loss of plutonium production will be only 70 - 80% of one pile's production as compared to a full pile lost in the R-10 loading. Difficulties attendant on the change from an H-10 to R-10 loading will require temporary use of extra U-235 in amounts comparable to the 5% excess needed for the double H-10 load. The double H-10 scheme also permits gradual expansion of production as required.

Just before the end of April the first experimental loading at DR reached critical size. The number of tubes required was about twice that predicted by calculations. This increase however corresponds to only a small difference in k, the measured k being 1.15 whereas the calculated one was 1.20. Further analysis of the results was in progress as the month ended.

The first experiment on critical mass of plutonium solutions was run in an 8 inch cylinder which had a complete water reflector. The assembly was not critical with 1000 gms. of plutonium and extrapolation of the data indicated that at least 1300 gms. would be required for criticality and that possibly a critical condition could not be achieved in an 8 inch cylinder. It is not possible to draw firm conclusions from this single experiment. However indications are that the critical mass for plutonium solutions is significantly larger than currently accepted values. Following the completion of the first experiment it was necessary to shut down for an extended period to correct leaks and mechanical and instrumental troubles discovered during the experiment.

### ENGINEERING - A. A. Johnson

The effect of the change from helium to carbon dioxide atmosphere in the B Pile is shown by the marked difference in graphite expansion between B Pile at present and the F Pile at the same exposure which was before any carbon dioxide had been added. The expansion at the center line was 3.2 inches at F as compared to 1.9 inches at B; at a point 3-1/2 feet into the graphite from the front face the expansion at F was 2.2 inches as compared to 1.5 inches at B.

The previously outlined plans to increase the carbon dioxide at all piles to 100% are in progress. The B and H Piles have been operating at 100 % carbon dioxide. No further additions of helium will be made to the D Pile after May 1. An increase from 60% to 80% was in progress at the F Pile at the end of the month and a further increase to 100% approved for two months later. It is expected that the power level of both the D and F Piles may have to be reduced from 305 MW to 290 MW or less in order to prevent vertical thimble temperatures above 400° C. An increase in power level of

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the B Pile to 290 or 305 MW appears feasible and is in process of approval. The H Pile power level was increased from 330 MW to 370 MW during the past month. A level of 400 MW appears to be about the maximum permitted by the present water temperature rise limits from boiling considerations.

X-ray measurements on 19 samples mined from the bore of tube channel 2406 B on 4-19-50 has shown that the expansion at the center of the pile is less than that near the front and back. At the front of the pile the expansion increases very rapidly with distance into the graphite, for example the expansion adjacent to the third slug is greater than the expansion at the center of the pile. The flux at this point is only 25% that at the center of the pile. At the back of the pile the increase in expansion with distance is not as rapid.

In P-10 extraction operations a total of 205 slugs was extracted. Five irradiated slugs covered with a black or bronzed sooty type material were encountered during this month. Analysis has indicated that this material contains sulfur 35. The can opening room has been contaminated with this radioactive sulfur and at month's end considerable difficulty was being experienced in decontamination attempts. The source of this black deposit is not known.

P-10-A slug manufacture and billet extrusion operations were suspended during the month pending results of the DR tests to determine the proper slug length. A total of 105 billets was cast.

METALLURGY - R. Ward

The initial phase of the problem of viewing operations in a shielded cubicle reveals that leaded glasses of 6.3 g/cc density will attenuate the gamma radiation from a 4-year cooled, irradiated wafer of uranium similar to the attenuation obtained with 7.8 g/cc density steel. Further comparisons with larger diameter pieces of leaded glass having densities up to 6.65 g/cc have shown this denser glass to be superior to cast iron, steel and the previously tested leaded glasses. The current results of this investigation are given in a report, "Attenuation of Gamma Radiation by High Density Lead Glass Compared with Steel," issued as Document HW-17288.

As an adjunct to the attenuation studies, several types of glass and plastics have been exposed to freshly discharged uranium slugs that produce dosage rates of about $0.2 \times 10^6$ R/hr. Preliminary examinations show that the leaded glasses are much less affected by this gamma radiation than are the optical and plate glasses. Subsequent annealing operations not only clear the leaded glass but also appear to cause them to become even more transparent than they were originally.

Two aluminum-uranium 235 slugs, one having high reactivity (J-29) and one having low reactivity (J-69), were sectioned for chemical analysis and metallographic examination to check the cause of the difference in reactivity. Slug J-29 had a higher uranium content than J-69 with an average value of 9.04% as compared with 7.1% for J-69. The metallographic structure of both slugs appeared normal.

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Five uranium rods, rolled previously from billets at 600° C, were rolled at the Simonds Saw and Steel Company from 2" diameter to standard size rod at 300° C. There was much less oxidation at this temperature and a smooth rod surface resulted. Rolling uranium at 300° C appears to be feasible, provided entrance and exit guides are used to prevent whipping of the stiff rolled rod.

Experimental trials of a newly developed bronze-dip agitator mechanism, designed for easy adaptability to time cycle control, indicated satisfactory workability.

Battelle reports that stresses of approximately 225 p.s.i. would be required to produce 0.5 per cent total deformation in 2S aluminum at 450° C within 10,000 hrs. Stresses on vertical safety rod thimbles are believed to be around 50 p.s.i.

WK Woods:bb

W. K. W.

## SEPARATIONS - R. H. BEATON

### PROCESS - J. B. Work

#### 221-224-231 Plant Assistance

Abnormally high product accumulation in the Extraction sections at B Plant was traced to insufficient agitation at low solution levels. This situation has been aggravated by the increase in enrichment level, which resulted in smaller process volumes. Replacement of the agitators appears to have reduced the amount of accumulation.

Polystyrene and Textolite adapter plugs were tested for use in the Isolation Building drying cycle. The Polystyrene plugs failed in all cases. The Textolite appeared to be satisfactory.

#### 234-5 Plant Assistance

The glass-lined valves between the transfer-head-tanks and the reactors in Wet Chemistry Hoods 5, 6, and 7 were replaced during April. The lining had failed in the valves removed and the valve bodies and flanges were severely corroded.

Data studies show that plutonium castings have less fluorine, boron, and magnesium but more iron and nickel than the buttons from which they are made.

Excessive amounts of flash and correspondingly low piece dimensions were obtained during pressing early in April. This was caused by high piece temperatures during pressing and corrected by changing the temperature control procedures.

Coating procedure changes were being made at month's end in an attempt to eliminate alpha counts found at the tripod support points by a newly available monitoring device.

#### 234-5 Process Development

A scintillation counter has shown promise as an instrument to determine the integrated alpha emission from 090 components made in the 235 Building.

The neutron count of the 090 assembly has risen with the use of higher MWD/ST material. This specification requirement is being modified by Los Alamos so that it will be consistent with the scheduled increase of MWD/T level.

The sintered platinum filter boat has been found useful in handling plutonium peroxide slurries for both the filtration of the precipitate and the conversion of the peroxide to fluoride. Hydrofluorination of the sulfate-free peroxide at low temperature (150°C.) gave a fluoride which produced very poor yield when reduced to metal.

A mixture of 16 M $HNO_3$-0.1 M HF appears to be suitable for the dissolution of the skulls produced in the melting operation in the 235 Building.

Stack Gas Disposal

A study of the ventilation air humidity data at T Plant has revealed that the water in the sand filter was removed by evaporation over the period of March 8 to April 15. Approximately 15,000 pounds of water were removed during this period. Coincident with the return of dry operation the pressure drop and indicated filtration efficiency returned to their normal range; i.e., to a pressure drop of 4.6 to 4.7 inches of water and a contamination removal efficiency of 99.3 to 99.5%.

Equipment has been placed in operation to permit the evaluation of relative life expectancies of Fiberglas and sand filters. An air stream, laden with a predetermined amount of methylene blue smoke, is passed through parallel filter units containing various grades of Fiberglas media and aggregate duplicating a portion of the present plant sand filters. The useful life of the filters will be determined by the pressure drop increase in relation to the quantity of dust deposited.

CHEMICAL DEVELOPMENT - R. B. Richards

Packed column specifications for the TBP Metal Recovery Plant were finalized and submitted formally to the Design Division during the month. The extraction (RA) and stripping (RC) columns will be 19-in. and 22-in. in diameter and the respective heights will be 50 and 38 feet. Stainless steel Raschig ring packing (1.5-in. dia.) is specified for both columns. Packed column studies indicated no scale-up factor in going from an 8-in. column to a 16-in. column. Specifications for the alternate pulse columns which will permit the use of unexcavated cells and may possess greater extraction safety factor and rangeability are being prepared for issuance about May 10.

Solvent extraction studies in the 8 in. pulse column established the absence of a scale-up factor, based on a comparison with the 3-in. column studies. The assumption that there will be a negligible scale-up factor for a 20-in. plant pulse column is believed reasonable, based on the lack of scale-up factor for the 16-in. packed column.

During the month, the Redox submerged "hot" pump units were removed temporarily from life-test to obtain wear and performance data to aid in the preparation of preliminary specifications for production units. Generally, the performance of the long-shaft units was considered very good. While a detailed examination of all component parts revealed design weaknesses in both the Roth and Peerless prototypes, it is believed that these are easily corrected and previous life-tests exceeding four months of continuous operation would predict a production plant life of six months to one year. Development of improved bearing and journal materials will probably increase this life expectancy.

Preliminary specifications have also been issued for materials of construction and protective coatings for the TBP Waste Metal Recovery Plant. Liquid and vapor phase corrosion studies of stainless types 304, 347, 309 SCb, and Carpenter 20 in waste concentrator solutions (RAW) containing 2.3 g./l. of chloride ion produced corrosion rates of 5 to 15 mils per year. Since corrosion

appears to be uniform, these rates are acceptable. Further definition of erratic corrosion on cold condenser surfaces must be carried out before specifications can be completed. Ucilon (United Chromium Co.) has proven to be the most satisfactory all-purpose protective coating to date. Tests thus far have been conducted with coated steels, but it is expected that Ucilon will prove satisfactory when coated on concrete. Such tests are now in progress.

CHEMICAL RESEARCH - F. W. Albaugh

Comparison of Diluents for Tributyl Phosphate in the Uranium Recovery Process

Tributyl phosphate, to be used as the extractant in the recovery of uranium from stored wastes, is mixed with an inert diluent in order to obtain a solvent phase of satisfactory density and viscosity for column operation. Criteria in the selection of a diluent include column operability, decontamination, uranium extraction, safety, and chemical stability. Carbon tetrachloride and a number of hydrocarbon diluents have been compared in these respects.

Column operability may be expressed in terms of the density differences between phases (in column operation) divided by the viscosity of the continuous phase, a factor proportional to the Stokes velocity of rise or fall of a droplet, which is a measure of column throughput capacity. With the organic phase continuous, the order in the RA Column would be hexane > Varsol = Stoddard Solvent > AMSCO 125-90W > Deo Base > Shell Base > $CCl_4$. In the stripping column the situation is reversed and the velocity in the $CCl_4$ system is much higher. With a continuous aqueous phase the velocities are nearly constant, except for hexane, where it is higher in both RA and RC systems, and for $CCl_4$ where it is much lower in RA and much higher in RC. These observations have been verified in pulse column operation, carbon tetrachloride giving high RC and fair RA throughputs, while Shell Spray Base shows high RA and low RC throughputs.

Decontamination has been found to decrease in the order $CCl_4$ > AMSCO > Deo Base; this decrease being in the order of increasing aromatic content.

The various diluents have similar effects on the uranium extractability of TBP in both the extraction and stripping columns and are all equally satisfactory.

From the standpoint of safety, hexane must be considered the least satisfactory because of its low flash point and carbon tetrachloride the most satisfactory because of its non-flammability. The differences in flash points of Shell Base, Deo Base, AMSCO, Varsol, and Stoddard Solvent do not appear sufficient to be taken into account.

All of the diluents considered are stable to 6 M nitric acid solution at room temperature. Although comprehensive comparisons have not been carried out under more rigorous conditions, carbon tetrachloride, AMSCO, Shell Base, and Deo Base are believed to be superior to Varsol and Stoddard Solvent.



Z-133-2

Exploratory Investigation of "25" Process Simplifications

The design of a plant for processing 3500 grams of uranium per day on the basis of the ORNL "25" Process flowsheet is complicated by the large number of duplicate vessels required to insure a critically safe process. Two ways in which plant design may be simplified are (1) to employ a more rapid method for U-Al slug dissolution than the batch $HNO_3$-$Hg(NO_3)_2$ method used at ORNL (26-hour cycle time per 350 g. U) and (2) to effect a head-end separation of uranium from the bulk of the aluminum, the latter being responsible for the awkwardly large volumes handled in the ORNL process. Two alternate methods for slug dissolution and several methods of Al-U separation have been studied.

Quite promising results have been obtained employing NaOH-$NaNO_3$ dissolution of U-Al followed by filtration or centrifugation to separate the insoluble $UO_x$ residue from the supernate, which is ca. 4 M aluminate solution. The residue is then dissolved in nitric acid to give a solvent extraction feed containing 50 or more g. U/l, very little aluminum and, it is hoped, only a small fraction of the original silica. Extrapolation of dissolving rate data indicates that dissolution of plant U-Al slugs should be complete in four hours. Acceptably low uranium losses, $< 0.05\%$, have been obtained in the separation from aluminum. This method appears highly attractive, combining rapid alloy dissolution with separation from aluminum, low uranium loss and a small volume of waste solution.

Continuous column dissolution of U-Al with $HNO_3$-$Hg(NO_3)_2$ solution is also being studied. Preliminary results indicate that a 5-in. column 6 ft. in length operated at ca. 80°C. could deliver 1.4 M ANN +2 M $HNO_3$ solution at a rate corresponding to dissolving 3500 grams of uranium per day.

Other methods of uranium-aluminum separation studied were precipitation of uranium peroxide, use of an anionic resin exchanger, and electrodeposition of uranium. The peroxide method showed some promise, the other two methods very little. Work on these methods has been abandoned to concentrate efforts on the caustic nitrate method.

RH Beaton/jw

## TECHNICAL SERVICES - T. W. HAUFF

### ANALYTICAL LABORATORIES - D. W. Pearce

#### Rala Laboratory

Full effort was continued on this program, with seventeen designers and draftsmen in the Design Unit (all working a 6-day week) and the equivalent of four full-time chemists engaged in analytical methods development.

Shielding calculations confirmed that the laboratory Cubicle should have 12-inch and 8-inch thick steel walls and top, respectively, and prints were issued for final approval on this basis. The Instrument Division outlined the optical systems of the five periscopes required in this Cubicle to the point where their formal drafting could be commenced. A feasibility report also was received from the Instrument Division, regarding the controls proposed for the Remote Control Transport system of the analytical line. It was concluded that this system could be made to perform the desired functions.

#### Isotope Determinations

Experiments with the alpha pulse analyzer indicate that Am-241 and Pu-238 produce overlapping waves, but that these waves are distinct from those due to Pu-239 and Pu-240. Limited chemical studies with Am have indicated that it may be effectively separated from Pu by making a preliminary oxidation of the latter with Ce (IV) and then carrying the Am on a cerium fluoride precipitate. Fission products are also almost completely carried by this precipitate. It is anticipated that this chemical technique may provide a basis for analytical control of the Am recovery operation recently proposed by Seaborg.

#### 234-5 Process Control

Increased proficiency and versatility of laboratory personnel allowed the 234-5 Process Control Laboratory to revert on April 24 to a two-shift schedule in accord with that of the S Division.

### ENGINEERING SERVICES - C. A. Rohrmann

#### Hanford Works Laboratory Area

Early in 1947 it became apparent that existing Hanford facilities for development and plant assistance work in radiochemistry, radiometallurgy, health physics, and pile technology were inadequate, and that provision of the required new laboratories and supporting facilities would involve a major design and construction program. Originally the proposed group of new buildings and services was called the "Technical Center," and a new outer area location was given serious consideration. Subsequently, for economy and to expedite construction, a site located adjacent to and southeast of the present 300 Area was chosen. The designation of these new facilities also was changed, and they now are known as the "Hanford Works Laboratory." J. Gordon Turnbull, Inc., was the architect engineer for the

initial scoping of this entire program, and has been retained for the Health Instrument Control and Development Bldg.

The facilities now contemplated for the Hanford Works Laboratory, and cost approximations for each, are as follows:

| | |
|---|---|
| Radiochemistry Building | $5,000,000 |
| Radiometallurgy Building | 1,500,000 |
| Health Instrument Control and Development Building | 1,550,000 |
| Mechanical Development Laboratory | 600,000 |
| Pile Technology Building | 2,900,000 |
| Plot Plan and Utilities | 1,900,000 |
| Library and Files Building | 850,000 |
| Laboratory Supply Building | 175,000 |
| Additional Hot Cells - Radiometallurgy Bldg. | 175,000 |
| Outside Services for Second Construction Phase | 350,000 |
| Total | $15,000,000 |

The A.E.C. has indicated that funds totalling this amount may be assumed to be available, with $9,525,000 for fiscal year 1950 and $5,475,000 for fiscal year 1951. The Technical Divisions have the basic "using division" responsibility for all of these facilities except the one Health Instrument Divisions building. The Design & Construction Divisions have the usual responsibility for project proposal preparation, subcontractor selection and contractual relations, as well as for all other G. E. engineering phases of this major design and construction program.

The Project Proposal covering design of the Radiochemistry Bldg. has been submitted to the A.E.C. for approval, and the Leland S. Rosener Co. of San Francisco has been recommended to the A.E.C. as the architect engineer for this facility. The preliminary design of this building, and a list of design criteria, currently are being prepared for presentation to the architect engineer at the start of contract negotiations. Assuming early approval by the A.E.C., final design of the Radiochemistry Bldg. should be completed by December, and construction should be completed late in 1951.

The Radiometallurgy Bldg. is planned for design and construction during the same periods as noted for the Radiochemistry Bldg. The proposed building provides space and services for eight heavily shielded cells in which the physical testing and examination will be done. While the ultimate need for eight such cells is recognized, only the first four (three 30-curie and one 1000-curie) are included in the original proposal because of cost and the possibility for design revisions after some experience is gained on the proposed cell types. The Project Proposal for the design of this facility has been submitted to the A & B Committee.

The Project Proposal covering the design of the Plot Plan and Utilities for the first phase of the Hanford Works Laboratory Area is being prepared for submittal to the A & B Committee about May 15. This proposal is being made to include the Mechanical Development Laboratory, as considerable economy will result from the initial use of this building shell to house the offices and shops of the construction contractor(s). As the construction phase of the new area is completed, the interior of this building will be converted for

use as the Mechanical Development Laboratory.

The A.E.C. also has authorized preparation of the design project proposal for the Pile Technology Bldg., originally considered as second phase construction, and preliminary plans for this building are under study. Work on the other two buildings in the second phase of the present program (i. e., Library & Files and Laboratory Supply) has not yet progressed beyond the preliminary design stage.

All liaison between the Technical and the D & C Divisions on the design of these new laboratories and services (as well as the Redox Laboratory, Bldg. 222-S) is the responsibility of the Engineering Section of the Technical Services Division, and to date has been handled largely by two Contact Engineers. These men probably will have to start a 6-day work-week early in May.

Technical Shops

The transfer of machine tools and shop supplies from Bldg. 3706 to Bldg. 101 was begun by Maintenance to effect the consolidation of these Technical Shops into the latter location. Sufficient equipment is being retained in Bldg. 3706 to continue a small, one-man machine shop in that building. Arrangements for expanding the Bldg. 3706 Glass Shop into the larger room being vacated by this machine shop consolidation were made with the Maintenance Division.

STATISTICAL STUDIES - B. F. Butler

Considerable statistical assistance was given the Pile Technology Division in connection with P-10 fuel slugs. Charts of Test Pile reactivity checks on these slugs are being maintained daily. By analysis of variance, significant differences were found to exist between heats of this Al-U(235) alloy. The within-heat variation was significantly lower in the latest shipment. No consistent relationship was found between the order in which slugs were cast and their reactivity. Sampling plans for testing bare and canned slugs are under investigation.

A statistical study indicated that the dilatometric test on bare uranium slugs is satisfactory for determining the degree of transformation achieved in the bronze bath. Canned slug data are being gathered to determine whether a similar pattern will be followed.

LIBRARY AND FILES - C. G. Stevenson

All activities of the Information Group proceeded routinely during April.

TW Hauff:mcs