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Subject HEW - Technical Progress Letter No. 24

To ... H. Evans

From ... W. Crane

Copy No. [redacted]

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December 23, 1944

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HANFORD ENGINEER WORKS
TECHNICAL PROGRESS LETTER NO. 24
December 15 through December 21

CANNING PROCESS

Production and Inspection Data

Slugs reported from inspection during the week were canned from December 9 to 15th inclusive and were comprised of 55.2% type X and 44.8% "Hold" material. The yields were:

Type	X % of Canned	Hold % of Canned
Class III	73.1	81.3
Reject Cause		
Non-seating	2.2	0.7
Not canned	0.1	0.0
Holes	0.1	0.1
Damaged caps	0.0	0.1
Bad welds	0.2	0.1
Thin caps	1.8	1.0
Surface Defects	4.3	3.2
Frost test 2, 3, 4	17.7	13.3
Penetration and Diameter defects	0.2	0.0
Miscellaneous	0.3	0.2
Total Rejects	26.9	18.7

Autoclave Testing

The results to date on class III submersion canned slugs are:

Type	Tested	Rejects		Rejects per 1000		
		Top	Can	Top	Can	Total
A	21,704	14	2	0.65	0.09	0.74
X	33,043	11	1	0.33	0.03	0.36
X	7,863	1	1	0.13	0.13	0.26
Hold	5,323	2	0	0.38	0.00	0.38

No material changes occurred during the week.

Development Line

All work on the development line for the past week centered around the lead - - Al-Si dipping process. Samples for corrosion testing were made as follows:

7 pieces lead dipped, dumped from can into quench.
6 pieces triple dipped, dumped from can into quench.
22 pieces when Al-Si dip increased and with Pb dip constant.

153 pieces were made with boss-type caps and 413 with 1.370-inch squashed caps using the following procedure:

Dip in Pb agitating vigorously.
Dip in Al-Si
Can in 2S Al can that has been preheated in the normal manner

The Frost test inspection of 347 of these pieces showed 4 in Class 2, and 3 in Class 3. (% Rejects = 2.01%)

In further study of the lead - - Al-Si process, slugs were given various lengths of dips in the Al-Si bath prior to canning to determine the effect of dip time upon lead content of the bonding layer and structure of the compound layer.

Plant Assistance

Study of etching methods for stained Al cans has been continued. An etch of $NaHSO_4$, followed by the standard etch in H_3PO_4, removed the stains completely and gave a surface of good appearance. Results of a production run on 398 cans treated in this manner indicated some improvement in wetting as judged by 12% frost test rejects on these cans in comparison to an average of 21% rejects for the same shift on six preceding runs.

Micro cross-sections of Al cans with badly galled surfaces from a lot of cans most recently received showed the defects to be not greater in depth than approximately 2 mils. However, despite the shallow nature of the defects, the cans have an undesirable appearance.

100 AREA

Physics

During the week 105-D has been run at various levels between 115 and 130 MW. On Dec. 17 the H.P. A. carried out tests which resulted in a complete power failure which was long enough to permit the Xe poisoning to grow to the point that the nearly 30 inhour excess reactivity available at the time of shut down was consumed, and the pile was down for about 8 hours. During the day the H.P.A. tests caused numerous surges resulting in scram shutdowns. On December 20 the pile was shut down to prepare for loading a total of 2004 tubes.

On Dec. 17 power operation of 105-D was begun with tubes 2671 and 2176 charged with poison slugs and tubes 2681 and 2166 charged with poison slugs and lead slugs alternating. After calibrating rod A with rod 9 withdrawn and adjusting the position of the chambers connected to the galvanometers, the power was taken to about 55 MW and held at this level until rod A was completely withdrawn. Frequent readings of critical positions and graphite temperatures were taken in order to establish a base point for the graphite temperature relaxation time and the power coefficient before the Wigner disease had altered the graphite thermal conductivity. When rod A was completely withdrawn the power was raised to 75 MW. On Dec. 19 it was raised to 125 MW and on Dec. 21 to 150 MW. At 8 A.M. on Dec. 22 all rods were withdrawn except No. 9 which was at 120 inches and rod A at 176.8. The unit had been shut down for 28 minutes as a result of a scram which occurred early on the morning of Dec. 22. Preliminary estimates indicate that with the present poisoned tubes it will not be possible to go to a 30°C temperature rise in the hottest tube, which is the level planned for the next month's operation. At 150 MW, the hottest tube in the inner orifice zone (0.240-inch) is running with about a 26° rise and, in the next zone (0.200-inch orifices), about 28°. It is estimated that, at 150 MW, the equilibrium excess reactivity will be about 25 inhours. Poisoned tubes will be discharged to permit again to the desired 30° temperature rise.

Engineering

The motion indicators on the 105-D unit were calibrated. The readings obtained during start up were of the expected magnitude, less than 10 mils. The optical device on the rear tight wire gave the most significant readings, indicating a 4-mil dishing in going from 55 to 75 MW.

Further work to evaluate water flow through one side of the retention basin at 100-B with the unit operating was done by continuous uniform addition of a concentrated solution of dichromate to the outlet process water. Total flow was about 29,000 gal./min. Samples of water were then taken over a period of time from several points in the basin and analyzed for dichromate. The results indicated considerably less channeling along the flume side of the basin than was first thought.

Consideration was given to a proposal to reduce the vacuum in the top section of the 105-B downcomer by installing a specially designed section in the end of the vent line to reduce the pressure drop due to entrance losses.

No action was taken, however, as operation of the downcomer at 105-D, where a 12-inch equalizing line was installed, indicated that reducing entrance losses would not appreciably lower the vacuum in the downcomer, but would increase the amount of air drawn into the system.

The tests on the downcomer and cushion chamber at 105-D have been completed. The stop log was replaced, but at a lower level than originally installed. This allows sufficient free space over the water t prevent the 12-inch pressure equaliser line from becoming sealed off. The air pumping is at a slightly higher rate than previously but the pressure in the cushion chamber has dropped to about 1/2 inch of Hg. The corridor is safe from the standpoint of leakage through the floor seals, and, therefore, no further action is scheduled.

A vertical safety rod stuffing box proposed by the Design Division will be tested at 100-F. The design is currently under study and plans are being formulated for the test.

Modifications in the CO_2 supply system for the vertical safety rod thimbles at 105-F are being incorporated which should improve operation. With these changes the CO_2 can be supplied at a volumetric rate equal to or greater than the rod withdrawal rate.

Satisfactory progress is being made on the slug discharge investigation. Several discharge devices for starting "free fall" have been designed and equipment for breaking the fall and directing the slugs into the pit is being assembled for experimental tests.

The No. 2 Purification Unit at 115-F has been operated. The gas in the main circulation system was brought to 99.7% purity in 20 hours. There is enough 99.4% purity gas in storage for about 200 days operation without further purification.

Water and Corrosion

An attempt was made to correlate pressure variation with iron analyses through recent periods of relatively wide variations in the latter. So far, no significant correlation has been developed, but it is planned to continue the study at least one more week.

The average pressure drop across the eight selected tubes of the 105-B unit has increased somewhat in the past week and most of the improvement caused by the 1-2/3 hr. oxalic acid purge has been lost. Another purge is scheduled for December 23, and is to be made at full flow rate with the unit shut down, and lasting 4 hrs. if necessary.

The iron content of 100-B filtered water has varied somewhat but a marked improvement has been noted over the preceding week, particularly for the past 3 days. The improvement is attributed to reducing filtering cycles from 75-90 hrs. to 48 hrs. The silica addition to process water was raised from 10 ppm. to 20 ppm. on December 13 to protect against coagulation of the larger amounts of iron. This will be reduced when it is felt that the iron content can be expected to stay consistently at about the 0.03 ppm. level.

[redacted]

The difficulty in controlling the iron content of filtered water experienced at 100-B was repeated at 100-D. Reduction of the filtering cycles to 48 hrs. seems to have corrected the condition and it has not yet been necessary to increase the 10 ppm. silica addition.

An oxalic acid purge was given the unit on December 15 to insure clean surfaces of the tubes and slugs preparatory to start-up. The purge operation was carried out without difficulty.

The flow laboratory tubes were started up on December 17 at the same time as the unit. The flow in one tube is controlled by a 0.24-inch orifice and in the other by a 0.20-inch orifice. A 375 lb./sq. in. supply pressure is being held in the D Area as compared with the 350 lb./sq. in. in the B Area. The outlet temperatures are being held the same as those of the hottest tubes in the respective zones of the unit.

Instrument Development

Significant information on the performance of the differential thermocouples in 105-D was obtained during D area start up. This equipment, which indicates pile power by measuring the difference between inlet and outlet water, allows readings of thermal voltages equivalent to less than 1/40°C. The arrangement is more sensitive than that in 105-B because lower resistance thermocouple leads allow the galvanometer to be operated nearer to optimum conditions. In addition to reading the difference in water temperature across the pile, the temperature of the water in any inlet riser or of the exit water can be read with the instrument. A differential thermohm system has been assembled to investigate the possibility of application to the measurement of pile power. The equipment shows high sensitivity and is now undergoing tests to determine its characteristics.

The neutron thermopile has been indicating the neutron intensity in hole F in 105-D since start-up. With the device placed only 6 feet in the hole, the sensitivity is 57 megawatts/millivolt. As soon as experience has been gained with it in this position, it will be moved in further to increase its ability to detect low pile power.

The helium thermometer is operating satisfactorily in 105-D although the graphite temperature, as indicated on 140 thermocouple, has not changed sufficiently to be readable on this thermometer. Data taken on the DSCF thermocouples in the pile show them to be operating correctly.

The spherical monitoring units to measure the gamma activity at the exit of the 107 retention basins are installed in 107-D and are nearing completion in 107-B. The beta monitor is also being put in 107-B.

An optical viewer for use during the discharge operations in the 105 buildings was completed. This device can be used in the labyrinths on either side and at any of the four levels.

The pile discharge face deflection indicator was installed at Building 105-D. This device consists of an optical lever arranged so that deflections of a spot of light on a scale can be read through the ceiling periscope.

200 AREA

General

Processing of active material through the extraction and decontamination equipment of the 221 Building started on December 18 with the first "X tracer" runs. Two runs, X-1 and X-2, have been processed through extraction and well into the first decontamination cycle. The product concentration in these runs, about 100 mg./ton or 1/30 Clinton concentrations, is too low to permit reliable analytical figures to be obtained by the micro-techniques developed for use at normal HEW concentrations. Hence, the preliminary analytical values, obtained by micro methods, are being checked by conventional macro methods and until results from the latter are available, too much significance should not be attached to the yield values so far obtained.

The extraction yield on X-1 was 73% and the waste loss 26.5%. For run X-2, the indicated yield was 82% and a complete figure on the waste loss is not yet available. In the first decontamination cycle, only the yield loss in the by-product precipitate for run X-1 is available; this is 1.5%.

Decontamination in the extraction step appears normal. Values for the decontamination factor of 6.5 and 9.0 for gamma's and 16 and 16 for beta's were obtained for X-1 and X-2, respectively.

During this period, a series of "T tracer" runs was underway in the Hood Kiwi equipment. The product concentration for the T tracer is about 500 mg./T and the micro analytical methods, is correspondingly more reliable. Two runs, T-7 and T-8, have given the following results:

Run No.	Extraction Yield %	Extraction Waste Loss %	Gamma Decont.	Beta Decont.	By Product Waste Loss, %	By Product Gamma* Decont.
T-7	100%	3.6	4.0		3.8	174
T-8	94.5	1.7	6.8	20.8	1.8	116

* Accumulative

The waste losses and yields are about as anticipated.

A further complication, in the case of the X tracer runs arise from the high MnO_4^- content of the HEW, as reported last week. This condition is known to result in higher extraction waste losses. The T tracer HEW on the other hand contains less MnO_4^-, which is probably also reflected in the lower losses encountered in T-7 and T-8.

[redacted]

221 Bldg.

In addition to the X tracer runs, the chemical runs started last week were processed through the decontamination cycles and the "B spike" (One ton of dead slugs plus 32 active slugs from 105-B) was dissolved and blended with the UNH from 3 tons of dead metal preparatory to the B tracer runs. The chemical runs proceeded without incident. Further data was obtained on the procedure for removing the $BiPO_4$ product cake from the centrifuge bowl. Confirming the result s reported last week, it appears that the product cake should be slurried out with five rather than three portions of HNO_3.

The following results were obtained on dissolving the B spike:

Weight of metal charged	-	2200 lbs.
Dissolving time	-	16½ hours
HNO_3 in 40% UNH	-	5.4%

The composite metal solution for the B tracer runs has been analyzed as follows:

Sp gr	- 1.39
% UNH	- Analytical result s not yet available (estimated 45%)
Acidity	- 3.8% HNO_3 (H_2SO_4 not yet added)
Fe	- 0.0047 M
Product	- 0.1 μ g. /ml., 0% oxidized.

Routine production and performance records have been instituted, starting with the first tracer runs.

224 Building:

Maintenance work has continued in preparation for chemical runs. Overflow baffles and agitator shrouds have been installed on the precipitators to prevent splashing of the distributor spray. A partition is being installed to separate the control gallery in Cells F and G from the operating equipment.

The product solutions from chemical runs C-1 and C-2 were transferred from Bldg. 221 to the receiving tank in Bldg. 224 and then returned to Bldg. 221 for disposal. Chemical run C-3 then was transferred from Bldg. 221 and started through the crossover in Bldg. 224. This run will be processed through a complete trial of the flowsheet process. The operating procedures for the chemical and tracer runs have been drawn up.

231 Bldg.

Installation of tight covers and the modified samplers for the process vessels in Cell 1 has been started and installation of a spare ventilating fan (steam driven) for the exhaust system of the process heads is under way. Preliminary design work has been completed on an improve closure and methods of filling and emptying the 224-231 transfer can.

The present status of the program, set up several weeks ago, to evaluate improved arrangements of the sample can is as follows:

1) A design for a simple valve assembly to convert the sample can for pressure shipment has been received from Wilmington. No further work is planned along this line unless trouble is encountered in service with method (2) below.

2) The most satisfactory solution appears to be to replace the present capillary vent with a 1/4 inch i.d. tube, baffled at the open end to reduce the possibility of a direct splash getting into the tube. Tests conducted both here and in Wilmington indicate that this modification will practically eliminate liquid getting into the dust filter chamber. Procurement has been started.

3) Evaporation of the solution in the can to a solid is practical and relatively rapid. Tests using UNH as a stand-in for product have shown that only two hours are required if both the can and the air used for evaporation are heated to 90° - 110°C. However, this method would require an extensive installation in the process hoods and is not being considered further at the present time.

4) The product solution will solidify at dry ice temperatures and could, in theory, be shipped in this condition if necessary. The freezing point-composition relation for various concentrations has been determined. This method is the least attractive alternate.

Semi Works

221 Head End

The performance of the equipment during the B tracer runs, discussed above, has been satisfactory. Occasional plugging of the centrifuge and precipitator sprays has been overcome by alternate vacuum and pressure treatment.

The cause of the precipitate formed in the dissolver during the metal solution runs, repeated last week, has been traced to the presence of appreciable H_3PO_4 in the HNO_3 feed. Investigation by Operations revealed a leaking valve in a cross connection between the HNO_3 and H_3PO_4 lines in 211 Bldg. This interconnection has been removed to prevent a recurrence of the difficulty. The rate of leak together with the small volume of HNO_3 used in the S.W. dissolver was such that sufficient H_3PO_4 was introduced to combine with about 10% of the UNH, precipitating uranyl phosphate.

221 Bldg.

Calibration and checking of the equipment continues on a one shift basis. Centrifuge jet dilution average about 7% at the specified feed rate of 5.5 lbs/min. The chemical header system including the feed pumps have been checked with water and found to be satisfactory.

Process Chemistry

Precipitate Washing Studies

Ammonium fluosilicate, alone and with dilute HNO_3, did not produce any substantial removal of Zr and Cb from an extraction precipitate. The results of the NH_4HF_2 washing technique, reported last week, were substantiated by a second run with identical results, a decontamination factor for Zr and Cb of 120 through extraction (including the NH_4HF_2 and KOH cake washes) and an overall factor of 10^5 at the end of the first decontamination cycle of the present plant process. It has also been found that an appreciable amount of Zr and some Cb can be removed from the extraction cake by washing with strong H_3PO_4.

Iodine Evolution during Dissolver Operation

This work has been continued using radio-iodine to trace the course of the evolved iodine. Three runs have been completed. The liberation and evolution of the iodine (from KI) under conditions existing during metal solution step is rapid (90% in 20 min.), the rate falling off as the concentration decreases. These results indicate that the rate at which the iodine is released from the metal and its subsequent adsorption on equipment surfaces are probably more important factors in controlling the overall evolution rate than is the liberation of iodine from the solution once it is released from the metal. An analytical method for iodine in the HNW solution has been developed.

Purity of Product Stock Solution

Further determinations indicate quite definitely that the product obtained from Clinton contains about 30% impurity. It has also been found that the impurity does not follow the product (and can be effectively removed from it) in a peroxide precipitation. Tests for thorium gave negative results, La is suspected.

Tracer precipitation

The Ru tracer received from Clinton (42d Ru^{103}) has been made up as a stock solution and standardized. Another precipitation of 39 tracer has been completed and turned over to the Analytical Division.

Equipment and Instrument Problems.

Work continues on setting up the quartz micro balance which is expected to be in working condition by December 26.

Several product standards were prepared for the calibration of Plutes as well as for the counters used in counting collected air samples.

The addition of a bakelite backing to the sample shelves of the standard beta counters was found to have no effect on the counting rate. The bakelite backing is used on all H.E.W. counters.

[redacted]

A supply of absorbers is being prepared and calibrated covering the range of 2 - 1720 mg. Al and 60 mg - 14.5 gr. Pb.

Instrument Development

The stack monitor equipment in Building 292-T was used during the dissolving of a 1 ton charge comprising 1/3 hot metal and 7/8 dead metal. The Xenon concentration peak, occurring soon after the appearance of the first dense fumes, indicated about twice tolerance before dilution at the top of the stack. Iodine was detected, but in very small quantity. It is apparent that at this time that contamination of the equipment will present somewhat of a problem because of the background. Methods of overcoming this are being considered.

An experimental centrifuge tachometer with a strongly suppressed zero was built. The purpose is to demonstrate whether or not a tachometer having an expanded scale over a limited range would be useful in detecting, by speed changes, the end of jetting. The model has not yet been demonstrated.

Light transmission measurements of the Building 221-T periscope were made. The results will be compared with subsequent measurements made after exposure to radiation, in order to correlate radiation exposure to rate of darkening of the optical system.

The PP-5A hot sample chamber and accessory equipment were completed, tested, and delivered to the Building 3706 counting room during the week.

A Portable G.M. Tube Survey Meter has been modified by placing the G.M. tube on a probe and by adding headphones so that an operator can hear pulses created by radiation falling on the tube. More work is being done on this type of instrument to make it more applicable to field needs.

In order to avoid discrepancies in bug calibrations which may result from brass enclosing cases, lucite cases are being made for the 25 milligram units. It is hoped to use a magnetic type of bug handling rod with the new cases.

ANALYTICAL

A tentative method has been developed for the determination of acidity in high product solutions. A hood for use in this determination was built and is operating satisfactorily. Numerous other protective and remote handling devices are being developed, particularly those required for use in analyzing high product solutions in Bldg. 231.

Efforts are continuing to apply the volumetric method for determination of heavy metal in process solutions to smaller amounts. The method depends on titration of the reduced sample with ceric sulphate.

[redacted]

Considerable emphasis is being placed on development of satisfactory methods for the determination of fluorine, potassium, and minute amounts of hydrazine in process solutions.

Studies are being continued on development of a chemical method for the determination of product in strong nitric acid solutions.

Various operations in the 300 area process are being studied to determine whether or not hazards exist with respect to toxicity. This work is being done at the suggestion of the Safety Department.

P. W. CRANE
TECHNICAL SUPERINTENDENT
By W. O. Harrington