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Subject ~~HEW - Technical Progress Letter No.~~ December 1 through December

[redacted]

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To ~~R. H. Evans~~

From ~~S. J. Bugbee~~

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HANFORD ENGINEER WORKS
TECHNICAL PROGRESS LETTER NO.22
December 1 through December 7

December 9, 1944

CANNING PROCESS

Production and Inspection Data

All slugs reported from inspection during the week were canned from November 25 to December 2, inclusive, and were type A material. The following yields were obtained:

Class	% of Canned
111	73.6

Reject Gauge

Non-seating	3.4
Not canned	0.1
Holes	0.3
Damaged caps	0.1
Bad welds	7.4
Thin caps	3.4
Surface defects	4.9
Frost test 2, 3, 4	5.9
Penetration and diameter defects	0.4
Miscellaneous	1.0
Total Rejects	26.4

The sharp drop in yield was caused principally by increases in welding and thin cap rejects. The welding rejects are considered to be attributable to the use of low-grade methanol (mentioned in connection with advance reports from the welding operation last week). The increase in thin cap rejects occurred at the time that fluoroscopic inspection was introduced.

Autoclave Testing

The results to date on submersion canned production are:

Class	Tested	Rejects		Rejects per 1000		
		Tot	Gas	Tot	Gas	Total
111A	12,175	8	1	0.66	0.08	0.74
111B	29,481	11	1	0.34	0.03	0.37
111X	4,486	0	1	0.00	0.23	0.23

[redacted]

The new failures on 111A production occurred on slugs made with the low-grade methanol. For this reason, all slugs made with this material are to be reclaimed.

Development Line

The development work on the canning method of preheating the slugs in lead (30 seconds at 600°C), dipping in Al-Si, and then canning in the standard manner, has continued during the week. 79 of the 80 slugs canned in this manner were class 1 in the frost test, and none showed any sign of penetration. The bond strength of the assembled piece, as measured by chiseling off the coat, is the highest observed to date. Extensive metallographic examinations and corrosion tests are now being made on these slugs.

Plant Assistance

A review of the specifications for essential materials for the 300 Area has been carried out. These, after approval, will be passed on to the laboratories for their use in setting up control methods for all incoming essential materials.

Twenty-two Chicago drawn cans have been used in the canning operation and all were Class 1 in the frost test. This demonstrates that it is possible to fabricate thick bottom drawn cans that can be used satisfactorily in the present canning operation.

The accuracy of the fluoroscope method was checked during the week by measuring the top and bottom thickness on 24 cans, and then removing the ends and measuring them with a micrometer. From the data obtained, it can be stated that the fluoroscope, when operated properly, gives the end thickness to an accuracy of ± 0.010 inches.

Metallographic examination of 6 slugs from a group of approximately 58 canned in MacCauley aluminum cans, wire-brushed in Al-Si during the canning operation, checks previous observations that the aluminum can is well wet with Al-Si. Minimum can wall thickness, as determined by chemical stripping of a total of 18 slugs, is as good or better than regular production slugs. Least minimum can wall thickness was found to be 0.015 inches, and the average minimum thickness was 0.025 inches.

Cross-sectioning of frost test rejects has shown that small melted areas are most commonly associated with voids in the Al-Si bonding area, while the larger melted areas are associated with thin can walls, un-wet cans, or shattered compound layers. Aluminum oxide stringe have been found between the aluminum can and the Al-Si preventing proper wetting of the aluminum can.

Testing

Slugs canned by dipping in lead at 600°C for 30 sec. and canning directly in Al-Si at 600°C are showing more resistance to water corrosion at 170°C in 100 lbs./ sq. in. pressure than regular production slugs. Both types of slugs had twenty 1/16 inch holes per slug drilled to the base metal.

Radiographs of 50 regular production slugs canned during the week showed an average of 0.10% voided area.

Chemical analysis of the bonding layer of slugs dipped 30 sec. in lead at 600°C and canned directly in Al-Si at 600°C has shown it to contain 3 to 4% lead. Other slugs have been given a quick dip in Al-Si just before canning and will be analyzed to see if the lead content has been reduced. Corrosion studies in plant water at 95° C will be made to determine the effect of lead on corrosion resistance of the Al-Si bonding layer.

Standard Pile Tests

The average value of dih for 111A slugs tested was +0.06 and the average stringer length was 7.985 feet. The slight increase in reactivity over last week's value is in agreement with the slight length reduction.

100 Area

Physics

The 105-B unit was started again late on November 30 after discharging metal for 200 Area and charging 95 additional tubes in the corners of the pile. These tubes are in positions chosen for cooling of the reflector as required for a 1500-tube loading and the increase in reactivity as a result of charging these tubes cannot be easily calculated. The pile was run at 125 megawatts and the critical position at the beginning was held with rods 2 and 4 completely out, 9 at 140 inches and regulating rod A at 135 inches. The other rods were all fully inserted. This condition with 1595 tubes loaded was the same control configuration used at the beginning of the run a month ago with 1500 tubes loaded except that then No. 9 was completely withdrawn and A at 140 inches.

The unit was run at 125 megawatts until December 3 when the excess reactivity had dropped to 20 inhours, as determined by using the calibration of rod A with 1500 tubes loaded. The power was dropped to 120 megawatts at this time. On December 5, there was a normal shutdown at a time when the estimated excess reactivity was about 17 inhours. The rods were immediately removed and the unit finally reached power again in about 40 minutes. Due to the combined effects of poison burnup, cooling of the graphite and heating of the metal as the power was increased, the reactivity, as calculated from the rate of power increase, was as low as about two inhours at one time while the unit was being brought up to power.

At 105-D the diffusion length was measured in the same way in which this determination was made at 105-B. The source was placed in the center of the central tube and the diffusion length measured in the central horizontal plane on the line of the midpoint of the tubes. The air had not yet been replaced with helium. The diffusion length found was 49.3 cm., which compares with a figure of 47.8 found at 105-B under the same conditions. The higher value is to be expected because of better graphite quality. Charging of 105-D began on December 5 and dry critical was reached on December 6. The unit was charged in horizontal rows 29 tubes wide. The procedure was essentially the same as that used in charging 105-B except that 35 slugs instead of 32 were loaded in each tube. Dry critical was calculated to be with 17.05 layers loaded. It was estimated that if 105-B had been loaded with 35 slugs, it would have been critical with 17.5 layers. The difference can be accounted for by better graphite quality and slightly larger diameter slugs together with thinner average end cap thicknesses. Analysis of the data is under way to reconcile the comparison, taking into account such details.

Graphite samples have been taken from the capsules attached to slugs which were discharged last week from 105-B after an exposure of 3.66 megawatt days of the central tube. The stored energy was found to be about 21 cal./gm. as compared with 18 found on samples taken from the 8 test hole at the same time. The electrical conductivity of the capsule samples had decreased by 81% of the initial. This compares with a 75% decrease for the test hole sample. A tentative advantage factor of at least two for the capsule samples in comparison with the test hole samples has been estimated based on the electrical conductivity--exposure curves determined to date.

Engineering

Operation of a purification unit in Building 115-3 was checked using gas from the main circulation system at a time when the 105 unit was shut down. The activity of the gas was too low to measure. Concentrations of impurities in the gas before and after purification, as determined by mass spectrometer, are given below.

Impurity	Vol. % in circulating gas	
	Before Purification	After Purification
Hydrogen	0.7	0.5
Nitrogen	1.6	0.0
Oxygen	<0.2	<0.2
Argon	<0.05	<0.05
Carbon dioxide	<0.1	<0.05

During this test the gas passing through the purification unit was sampled at a point between the activated alumina dryers and the activated carbon absorbers, and checked by chemical means for oxides of nitrogen concentration. These analyses indicate the possibility that a dangerous quantity of oxides of nitrogen may be charged into the activated carbon in as little as 100 operating hours. Check analyses are to be made on the starting of 115-3 purification units next week, but until more detailed data are obtained, a ceiling of 100 operating hours without changing the carbon will be observed.

The temperature near the center of a tie strap on the discharge face of the 105-3 pile, with the unit operating at 125 megawatts, was measured by means of a thermocouple and found to be only 2 - 3°C. lower than the outlet water temperature at that point.

A thermocouple, placed near the middle of a bucket of active slugs which had cooled about 10 days, showed the water temperature inside the bucket to be essentially the same as outside. Further measurements will be made with hotter slugs on the next push of metal. No serious problem of slug corrosion during storage has been indicated to date.

Mock-up equipment for experimenting on "free fall" discharge of slugs from pile units is being set up and an investigation is under way to develop a rapid and satisfactory plant procedure for routine pushing of active metal.

Water and Corrosion

A study of the header average pressures from November 7 through November 19 showed a mean rise in pressure of 0.13 lbs./sq.in. daily, with a corresponding standard error of estimate of header averages of 0.43 lbs./sq.in. These data have been used in the establishment of limits for gaging current data.

105-3 Area

Samples of the effluent from control tubes contained 4.7-5.2 x 10-7 hydrogen peroxide when the unit was operating at 130 MW. At 100 MW, 4.1-4.4 x 10-7 concentrations were found. Both results were somewhat higher than would have been predicted by extrapolation of data taken at lower levels.

Interruptions in the silica analysis have been traced to the deposition of a microfilm on the emitter bulb of the spectrophotometer. The condition is remedied by cleaning the bulb once a week.

An 8-point recording potentiometer has been installed in the Flow Laboratory and is being used to record water temperatures in and out of the 70-inch jackets. The first data obtained are much more consistent than readings from the indicating potentiometer and it is expected that the heat transfer data will be considerably improved.

The peroxide concentration in the flow laboratory tubes was increased on December 1, from 3.2 and 4.0 to 4.5 and 5.0 x 10^{-2} M in tubes 1 and 2 respectively. This change was based on the latest values obtained by analyzing pile effluent water.

100-B Area

Acceptance tests of the demineralizing equipment originally scheduled for this week have been temporarily postponed. A 24-hour acceptance test of the deaerators was completed. Oxygen content of the deaerated water was irregular; the minimum concentrations varied from 0.2 to more than 1.0 ppm for different deaerators.

Iron analyses have shown that the filter plant performance is about like that in the 100-B Area. Results are consistently 0.02 ppm Fe or less.

Instrument Development

Following the successful completion of a testing program on the new proportional counter sets, this equipment was installed in 105-B for use during startup. Check tests following installation indicated that the complete sets, including BC tubes, amplifiers and scalers, were performing satisfactorily.

The installation of six new BH units during the last shutdown of 105-B has rendered significant data on the cause of the change in counting rate with time. All indications are that the predominant cause of this decrease in counting rate is a result of a gradual decrease in counting efficiency of the boron coated tubes. The decay of contamination in the process tubes and slugs appears to be the lesser of the two causes.

The monitoring equipment for use with the differential thermocouples for water temperature measurements in 105-B has been reinstalled. It is felt that this equipment offers the best possibility of controlling the power level close to the operating limit.

The neutron thermopile has been installed in hole "F" in 105-B and is now ready for operation.

Two [redacted] bulbs have been exhaustively tested for installation in 105-B as part of the helium thermometer. It was necessary to turn down one of these bulbs to provide sufficient clearance for installation. The bulb showed no leakage, however, upon tests with a mercury manometer.

[redacted]

Technical Progress Letter No. 41

300 AREA

Plant Assistance

221 Building

Successful operation of the plant dissolvers has been demonstrated. Following the nitric acid boiling tests, reported last week, several charges of empty Al cans were dissolved to gain experience with the coating removal step. Six tons of inactive slugs were then decanned and dissolved in 3 row batches containing 1, 2, and 3 tons respectively. One batch was run in each of three dissolvers so that the performance of each unit could be determined. The results of these runs may be summarized as follows:

1) Control of both the coolant removal and metal dissolving reactions is excellent. The pressure difference (negative) between the dissolver kettle and the cell affords the most sensitive control point. The maximum reaction rate can be easily controlled with the kettle cooling coil to maintain this pressure difference between 10 and 12 inches of water.

2) The capacity of the column and condenser appears adequate; by keeping the kettle pressure within the limits given above, only a few (1-4) inches of water back pressure was developed across the column and the exit gas temperature could be maintained below 50°C.

3) The dissolver time cycles are more favorable than originally anticipated. For the 3 ton charge, the first ton was dissolved in 7 hours, the second ton in 9 hours and the third ton in 18 hours. These times include the time required to add the HNO_3 and heat the charge (approximately 2 hours). The coating removal step requires approximately 6 hours, including addition of chemicals and the water and acid washes. Consequently, when metal is processed at the rate of 1 ton per day, approximately 22 hours will be available for charging the dissolver, the actual charging time required will be considerably less (perhaps a third) of this.

4) The chemical reactions involved in the coating removal step appear to be more complicated than expected. At low temperatures, below about 60°C, the reaction goes mainly to nitrite,

$2\ Al + 2\ NaOH + 3NaNO_3 \longrightarrow 3NaAlO_2 + 3NaNO_2 + H_2O$

At higher temperatures the ammonia reaction predominates,

$8\ Al + 5NaOH + 3NaNO_3 + 2H_2O \longrightarrow 8NaAlO_2 + 3NH_3$.

Some hydrogen is also formed, chiefly, it is believed, from the Al in the bonding layer. The maximum concentration of hydrogen found in the vent gas was 2.4%. Approximately 1/3 of the Al takes part in the nitrite reaction. The use of a 5% HNO_3 wash following the caustic-carbonate treatment was found essential to remove a fluoride scale left by the caustic. The resultant decanned slugs were quite clean, and but little sludge was left in the dissolver.

 [redacted]

c) Chemically, the metal dissolving step behaved as expected from Clinton experience. It will be recalled that the reaction is judged completed when the specific gravity, as read on the gauge board, reaches 1.78. Analytical results so far available on the resulting UNH solution indicate that the "finishing off" gravity should be a little higher, perhaps 1.80 or 1.82 since the UNH concentration is slightly low and the residual HNO_3 slightly high. It was anticipated that minor adjustments of this sort would be required as a result of operating experience.

Following the three dissolver runs on dead metal, a charge consisting of one ton of inactive Hanford slugs and 96 active Clinton slugs was dissolved. The resulting UNH, containing approximately 0.36 grams of product, was blended with the UNH from three tons of dead metal and will be used for the first four tracer runs.

During the week, chemical runs have been in progress in the extraction and decontamination equipment. Analysis of the results of these runs will be reported next week.

224 Building

Operations has completed the heating and cooling tests on process vessels and the correlation of the data has been finished. Results are in line with those previously reported and will be summarized in report form.

An analysis has been made of the process volumes at various points through the 224 building process, based on the jet dilutions found on actual operation. The controlling volume is in the Cell F batch tank. The October 5 flowsheet, using a 3:1 jet dilution, shows this volume as 3470 gals., about 7 inches of free board. Allowing for the volume of the recycled solution from 221 Building and the actual jet dilution (average 5:1) gives a controlling volume of 3800 gal. equivalent to 13 inches of freeboard. Approximately 6 inches of freeboard can be gained by blanking off the overflow from this tank.

Tests have been made in the closures of the "transfer can", the vessel used to transport process solutions between Buildings 224 and 221. It was found that (1) the cover plate seat leaks slightly, (2) the bottom of the cover plate will allow drops of solution to drip on the top of the container when the vessel is opened, and (3) the capillary vent on the cover pumps drops of liquid into the outside of the container due to splashing in the container as was found with the 221 Building sample can. A modified design of the cover is under consideration.

221 Building

Several modified [redacted] vents for the "sample can" have been tested. The most satisfactory modification involves replacement of the internal capillary tube with a 0.25 inch pipe of the same length. This change eliminates liquid pumping due to splashing as long as the can is not turned upside down. Since an accidental upsetting of the can during shipment is unlikely, the modification is being considered.

[redacted]

Technical Progress Letter No. 52

A design of a tight closure and an adequate vent for the 221 Building process vessels is being developed. The covers will be machined and bolted in place, all fixed pipes welded into place and movable pipes and shafts for the decanter and agitator will be equipped with water seals. In this way, it is hoped to reduce or eliminate the contamination on the inside of the process hoods.

Semi-Works

221-T - Head End

Chemical and dummy runs are under way in this equipment. A triple batch of irradiated slugs have been dissolved and dead extraction runs have been made. The decontamination cycle is currently being run. It is expected that tracer runs, through extraction and one decontamination cycle will start as soon as active UNH is available.

221 Building

Construction work was completed during the week and the building was accepted by the Operations. A program, on a reduced basis, of equipment calibrations and water runs was begun December 6.

Plant Maintenance

Preparation of the 100 Area Operating Standards was completed in rough draft and circulated for comments. Completion of the final draft is scheduled for December 11 for discussion with Metallurgical Project personnel.

Laboratory work on improving the decontamination of Zr and Cb by washing the product cakes with HNO_3 and complexers to show very promising results. An overall decontamination through the extraction step of 100-200 for these elements has been obtained. This simple modification of the present process may significantly increase the decontamination attainable in the plant, particularly in the early steps.

Process Chemistry

The shipment of radiocobalt from Site V has now arrived and the previously discussed tests on variables affecting the evolution of iodine during dissolver operation will be made. Tests on the density of UNH solutions, and on the dissolving of waste metal from the 221 Building for use in the semi-works are being made. Preparation of bismuth phosphate product slurries for use in testing 221-T sampling devices is in progress. Corrosion tests are being started on 304-SS sheet -60 graphite combinations at the request of the Plant Maintenance group and on 304-SS in HNO_3 solutions.

The effect of hydrazine in interchanging with product carrying in the extraction step is being brought to a conclusion and will be summarized in a report.

The tests of decontamination using bismuth compounds as carrier in place of bismuth phosphate have not given promising results and are being terminated.

The Solid and Fission Product Chemistry group is engaged in examining and preparing for distributing the Cb_2, Zr_2, Sr_2, and Y isotopes which survived from Site I on December 6, in separating Ba_2, Fe_2, Ba_2, and Fe isotopes from uranium recently received from Site X, in separating plant fabrication dust which arrives from various sites

chemical origins, and in determining the effects of small variations in plant variables on specific decontamination.

The Basic and Micro-Chemistry group has completed preparation of a summary of product solubilities in the presence of process anions at each step in the process, and is preparing a similar summary on oxidation states relative to the potentials of process solutions. They are carrying out certain determinations of the solubility of product in process solutions and of the effect of flocculants on such solubilities. Preparation of the quarts micro-balance is continuing. A determination is being made of the purity of the product received from Site X since there has been reason to suspect that considerable inactive material is present.

The Isolation Chemistry group is spending full time advising on construction problems in 231 building, testing methods of overcoming the leakage difficulties found in the product shipping container, completing work on the effect of reducing peroxide concentration in isolation process solutions, and on the stability of peroxide solutions in the isolation process.

Instrument Development

When the first active material was being dissolved in Building 221-T, the stack was monitored. Monitoring equipment operated satisfactorily, but no activity was detected in the stack gas.

Two of the four proportional alpha counters received from Chicago have been tested and put into operation in the counting rooms located in Buildings 3706 and 222-T.

Humidity and temperature measurements made in Building 221-T operating gallery, and temperature measurements made near the 8 inch GE tubes located in the 6 inch pipes in the cell walls, indicated that air drawn from the operating gallery to the GE tubes via the conduit does not form condensed moisture.

Calibrations of all process GE tubes in Building 221-T was completed during the week.

[redacted]

1-981

Technical Progress Letter No. 21

ANALYTICAL SERVICES

Emphasis is being placed on development of methods and necessary equipment for analyses required in Building 231.

The method for the determination of chromium in process solutions from Buildings 221 and 224 has been improved. In order to eliminate fading of the color, sulfuric acid was substituted for acetic in the diphenyl carbazide method.

A satisfactory titration method for the determination of OH^- in process solutions from Bldgs. 221 and 224 has been developed. A mixed indicator (Methyl red and bromcresol green)is used. The titration is made with alcoholic HCl in butanol-ethanol medium.

A fluorimetric method has been developed for the determination of fluorine in process solutions where the fluorine is present in quantities of 3-10 micrograms. However, the method is difficult to control and work on other methods of determination is proceeding.

A colorimetric method has been prepared for the simultaneous determination of iron and aluminum in process solutions from Bldg. 221. The method depends on the color produced by these ions with hematoxylin and subsequent spectrophotometric measurements.

Extraction of product from solutions with hexone and preparation of counting samples from the hexone solution was compared with the lanthanum fluoride procedure. The hexone procedure gave slightly higher results. Further work is being done on this problem.

S. J. [redacted]
TECHNICAL SUPERINTENDENT

[redacted]