- Australia V		75721		DU#-1
DECLASSIFIED	ECRE	, ,	16-19. 11	
F	PROJECT 9536	K.		۱ . الرابية سور
		File _		30
			a Pero	
Route List	Date _	7/7/44		
· Watty of	Sub jec	t	n TWX Technical	Section W
	Will Committee	Progre	ss Report - Jun	e 28 to Ju
	To	File	·	
Bo	From _	W.E.K	irst	
19.5	WLY.	Cop. No.		
REFERENCE USE I U	.	•	HOOD WORTH	46704
EFORE READING THIS DOCUMENT, SIGN	AND DATE BEL	W:	· · · · · · · · · · · · · · · · · · ·	
			11.	
Hors Mr. reach - 27 1/4			15.	
Luciality 840				
Corporate 5/10				ĺ
11 12 11 11 11	- 27			
			PROVED FOR	,
Marie 1/22.			I IC DELEACE	02
Marie 1/22.			PROVED FOR LIC RELEASE Snown 3-22-0)J
May 10 1/22.			I IC DELEACE)J
May 10 1/22.			I IC DELEACE	2
Mary 10 1/22.			I IC DELEACE	2
May 10 1/22.			I IC DELEACE	2
May 10 1/22.			I IC DELEACE	2
May 10 1/22.	¥		LIC RELEASE	10
May 10 1/22.	4	DJ7	LIC RELEASE	
May 10 1/22.	¥	DJ7	LIC RELEASE	10

This document consists of 12

page and

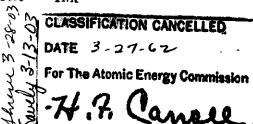
henres.

H. I. DU POIT DE NEMOURS & COMPANY

WILMINGTON, DELAWARE

Explosives Department - TNX

BEST AVAILAULE REPRODUCED COPY



Chief, Declassification Branch mt

R. E. DeRight

0

2. R. E. DeRight H. Worthington ₩.

4 2 R. L. Doan

5. M. D. Whitaker

M. D. Whitaker 6,

7. S. W. Pratt

8. W. O. Simon

9 " TNX Clinton Subject

10... TNX Clinton Chronologi

July 7, 1944

CLIPTON THE TECHNICAL SECTION TELELY PROGRESS REPORT

JUNE 28 - JURY 5, 1944

Pile Operation

The new fan was started early in the week and has ircreased the total air flow by about 20%." at the same time approximately 200 partial plugs were removed from the tubes in the 100 to 150° C temperature zone described and diagramed in the report of June 7-20. These partly plugged channels were largely in the bottom and top portions of this zone. As a result of the increased flow of air in these channels the metal temperatures dropped from 10 to 30° C and thereby increased k to a point where it was necessary to insert all four shim rods to within 59 of the center of the pile and to have the two control rods at about the same relative position. This large amount of absorber acted as a poison and lowered the temperatures on that half of the pile so that the only slugs at 200° C are in a half moon shaped some in the other

This situation is being corrected by an adjustment of the present poison columns and possibly by the addition of others. It is expected that this will again result in a symmetrical series of temperature zones with the shim rocs out and a power output of 2200 KW. As soon as the second fan is in use the remaining partial plurs vill be removed and the poisoning again adjusted to restore satisfactory temperature isotherms. The predicted pover output with both fans is 2400-2600 KW.

It is the opinion here that the new funs cannot raise the pressure drop through the pile enough to endanger the roof. It is expected that the unblocking of channels will allow all the air available to pass through the pile at a safe pressure drop. As a safequard a



ORO113601



manometer and mercoid have been arranged to function between 57 to 61 inches. This situation was discussed by Mr. R. W. Smith With Mr. Tebb.

Problem No. 124-XIE - Corresion of Hanford Tubes and Slugs
Under Radiation

One of the two water tubes has been operating 7 days with a charge of 40 un orded slugs, 32 in the graphite and four in each shield wall. The second water tube contains 20 each of zinc bonded and AlSi bonded slugs 16 of each alternated in the graphite and four in the shields. This latter tube has been in operation for 4 days. The water for both tubes contains 2 ppm K2Cr2O7, 40 parts SiO2 as silicate and about .1 ppm iron and is being held at a pH of 7.5 and a temperature of 82-86° C. Peroxide to the extent of 2 x 10-5 N is being injected near the tube entrances. The velocity of 6 ft. per second leaves the exposure at about one fifteenth that of Hanford at 250,000 kW. No samples have been taken for bichromate decomposition as the exposure is too wild but some will be taken later when the power level is increased with the new fans.

Prior to charging the tube with the bonded slugs these were given an autoclave test to show leas in the jackets. In this test six slugs were placed in a short section of ribbed tubing and several such pieces of tubing were inserted in a section of 8ⁿ pipe flanged off at both ends and connected to 100# (3280 p 1640 c) steam pressure. The autoclave was drained with a trap so that the slugs were damp but not submerged in water. The test was continued for 22 hours after which the slugs were cooled with a flow of water and removed for examination.

all of the ginc bonded slugs surveved the test with no bulges but one alsi bonded slug failed in each of the two tests with 25 slugs. The first failure caused the tube to swell and split for an inch and when the tube was cut off and the break in the slug cleaned with water, there was a cavity in the uranium about an inch in diameter and over half an inch deep. The jacket was bulged out to complete a spherical This break occurred about an inch and a half from the shape. bottom of the can and there was a crack almost all the way around the bottom at the corner* The second fullure caused a tab to lift on the jucket over an area of about 1 sq. cm. This break was near the weld end and there were other smaller bulges near the edge of the can. The remaining slugs vers all in good condition after the test. It appears that the autoclave method is very severe and slugs passing it should be of good quality.

*See photograph in attached Engineering Section report.





Decomposition of Dichromate by Radiation

The work has consisted of a series of runs at a spread of pH values from 6 to 8 and for times of exposure equal to Hanford conditions. The method being used is the single exposure type where a small sample is scaled in a glass container, placed at the center of the pile for the desired time and then removed and analyzed. The method used to obtain the values given in the report of June 7-20 consists of flowing a considerable sample (5 gallons) of water through a capillary tube in the pile at such a rate as to equal Hanford exposures. Samples of exit water are taken from time to time for analyses. Although not complete the results from the two methods agree in general except that the static exposure method shows some breakdown even at pH 8. Further work must be done before the results are corclusive.

Problem No. 124-X14E - Radiation Versus Film Polymation

When analyzed the solutions resulting from film removal from the two capitlary takes by exalic acid appeared to be a rgely from although they were not run for aluminum. Chromius was not present in large enough amount to give a positive result.

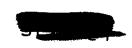
After cleaning, the two tubes were operated for 4 days with Hunford water to which 2 ppm K₂Cr₂O₇ and 40 ppm SiO₂ had been added. The pH was held at 7.5 and the from content was the same as in the previous run, that is all ppm. There was an increase in pressure drop in both tubes of about the same degree (10-12%). Several samples for dichromate decomposition showed about 704 ppm an amount consistant with earlier data. After running to exhaust the supply of water the tubes were again cleaned with oxalic acid. A third run has now been started with synthetic W water again containing 2 and 40 ppm of K₂Cr₂O₇ and SiO₂ respectively and all ppm from. The pH is being held at 8 and has varied from 7.8 to 6.2. A few samples for dichromate breakdown have indicated that this occurs even at pH 8. Further samples will be considered.

Problem No. 124-X12E - Measurement of Corrosion Currents and Potentials

In one experiment run for 36 hours the potential was measured between a slug of 28 and a 728 clad tube with proper insulation between them. This was done both with a slug near the centre of the pile and with one in the shielding. The potential with the radiated slug started at -.058V and did not become neutral for 32 hours after which it rose to \$\neq\$.006V at 36 hours when a breakdown stopped the experiment. The slug in the shield







started at -. 105V, reached zero in 19 hours and was +. 040V at 36 hours. In neither case, therefore, was the tube protective to the jacket at first but ir both cases it became so with time. This is a condition that was encountered frequently in the previous tests and the agreement between the results is sufficient to show that radiation played no part.

Testing Wethod for Locating a Swelled Slug in an Operating Pile

This work is still on an exploratory basis. The background in a charged tube in the SMX unit with normal water flow has been traversed for the magnitude of various frequencies and the experiment is being repeated with a slug carrying known bulges located at the centre of the tube. Pickup in this case is by a contact microphone fastened to the end cap.

If no characteristic frequencies are caused by the bulge the experiment vill be repeated with the pickup in the water inside the tube to eliminate the effect of the tube walls.

In case both methods fail an attempt will be made to transmit a characteristic high frequency down the water annulus and determine if it reflects back from a known bump somewhat in the matner of

SMX

The screen test has been almost completed. It was shown that the 50 mesh header screen will hold back any particles that could plug the conical tube screens even when the latter are blocked for their top halves to close the largest openings. With the firest sand used (65 - 100 mesh) the 50 mesh screen gradually plugged until with 10 grams the pressure drop was 300%. Considerable fine sand passed this screen and while it was found in the tubes there was none in

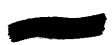
Further and more accurate work with water emulsions of 60 Texaco soluble his shown that the use of such emulsions during charging rear peralt replacement of the spline and hand crank charger with a much simpler device operated by one man. "hile evaluating a second type of oil an attempt will be made to design at attachment to permit hand charging by the use of an oil emulsion.

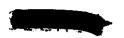
SEPAR STOP PLANT OFERATION

Cell Line-Up

Cells 5 and 6 were put in service with run 150. The "clinton" pro-







cers then in use consisted of the steps shown in column A of the table below. It was planned to start the "half Hunford" scheme with run 170 on July 6, 1944, following column B below. Adoption of the full "Hanford" process, column C below, will presumably take place about August 1.

Scheme	. A	В	· C
Wf.p. Cell 2 Cell 3 Cell 5 Cell 6 Cell 4 Storage Cell 6 Cell 5 Cell 5	No extraction by-product product by-product LaF ₃ product	Yes extraction Ce=Zr by=product product by=product LaF3 product	Yes extraction Ge-Zr by-product product Ge-Zr by-product Bi product 8 runs held by-product Li F3 by-product Li F3 product

Pre-Extraction Treatment

Formic acid was replaced by NaNo₂ starting with run 166 on 7-3-44. The extraction solution was made 0.1 M in NaNo₂ (41.5 lbs of NaNo₂ added with the 2400 lbs of dilution water from the dissolver) and held for ½ hour at 40° C before adding standard amounts of H₂SO₄, Bi, and H₃FO₄. The first run showed a 3.1% loss in the extraction waste solution (0.9% before skimming) while the next two each had 1.1% loss. This is far below the 20 to 20% previously found when HCOOH was omitted. Sodium nitrite will be adopted as standard unless difficulties arise.

Lower H3PO4 Concertration in Second By-Product Frecipitation

Rurs 159-163 were made with 0.02 instead of 0.1 M ${\rm H_3\,PO_4}$ in the Cell 6 by-product precipitation. The objective was to lower the amount of phosphate going to room D with the product LaF3, and so improve the oxidation of the room D two-timer while using less rirconium. It was deemed necessary, however, to return to 0.1 M H3PO4 when the 41 R analyses (reduced solution before LaFz precipitation) dropped to 12.7, 46., 39,4, 28.0, and 68.3% of total product for runs 159-163 respectively. D1-P analysis (HNC, solution of metathesized cake) of pair 158-9 showed only 52.7% which agreed with 41 R liqures in suggesting a product holder in 41 tank. It was thought that the lover H3PC4 concentration might have left sufficient bismuth in solution to cause bismuth oxalate which is known to carry product well to precipitate when the reducing agent was added. The D1-P analyses on pairs 160-1 and 162-3, however, both showed 12C and 110% rield, indicating pick-up of the hold-up. This theory is not water-tight, hovever, as at the same time lover Hgf04 was started in the second by product step, the new cells

DECLASSIFIED⁵



5 and 6 were put in service. Nevertheless, after returning to C.1 M H3PO4 in cell 5, the 41R sample showed 84.4% yield for run 164 and 122% for run 165. Since this value was 99.2% for run 158, the five low percents coincide exactly with the period of low H3PO4.

Lover HF Concentration in Cell 4

Eleven runs using 0.5 N HF for the LaFz product precipitation in Cell 4 showed an average loss of 1.2%. This is alightly greater then the former average of about 0.8% at 1.0 N HF, but the less corrosive conditions rould appear to justify continuance of the lower amount.

Oxidation in Room D

The difficulty in oxidizing recent pairs of rurs in room D, which necessitated the use of extra dichromate, zirconium, and time, has not appeared since the change to 0.02 M H3FO4 in Cell 6. If the trouble reappears when the 0.1 M H3FO4 runs reach room D, further tests will be necessary. The change being held in reserve, that is washing the LaF3 product cake ir centrifuse 42, should fill the need since this cake is not washed under present procedures.

Omission of Zirconium in Room D

Since recent isolation studies in 706-A have been handicapped by zirconium associated with the room D concentrate delivered for isolation, it has been proposed to emit the zirconyl nitrate from one or more eight-timers. It is felt that any zirconium leaking through from the two-timer (where its use now appears nocessary) would probably be removed during the eight-timer steps.

RESEARCH AND DEVELOPMENT

Slug Dissolving

Problem No. 201-X248 - Solution of W Slugs

Both 15% NaOH - 13 NaNO3 solution and 10% haOH - 20% NaNO3 have been compared in jacket removal tests. The former was selected for the last revision of the W flowsheet because of the possible precipitation of alumina on storing jacket solutions made with 10% NaOH. The following table summarizes the data:

Result of treatment

Rus & N. OH/S PANO

mola CH/mol Al

A B

Ia

15/13

1.25

no ppt no ppt syrup

PROMUAGII (PD				desult of treatment			
Run	% Na OH/% Na NO3	mols OH/mol Al		A	В	C	
Ib Ic ITa ITb IIc	15/13 10/20 15/13 15/13 10/20	2.5 1.2: 0.83 1.67 0.83	o	no ppt no ppt no ppt no ppt no ppt pot	no ppt no ppt	syrup solid	

(Treatment A - cool and let stand four days)

(Treatment B - evaporate to 80% of volume, cocl, and let sturd 4 days)

(Treatment C - evaporate to 30% of volume, and cool)

The indication is that neither combination will precipitate at the OH:Al ratio of 1.25. Since the 10/20 mixture reacted less vigorously and both mixtures suspended the precipitate equally well, the 10/20 combination will be recommended for 205 Building trials and the W flowsheet.

Extraction

Problem No. 271-X18AS - BiPO4 Precipitation Studies

Three types of extraction strike have been found to reduce waste losses in extractions at W product concentrations. These were compared in 3 liter scale "DS" runs. In one series of runs the reverse strike (Bi last) with 7 N B1 reagent solution and the combined strike (2/3 of Bi, all H3PO4, and then other 1/3 Bi) gave waste losses equivalent to the two hour H3FO4 (direct) strike. In another series, however, the H3PO4 strike gave about one-third the losses of the other methods. It appears that the reverse strike gives somewhat erratic results and the long H3FO4 strike is preferable.

Problem No. 203-X25S - Improvement in Decontamination

Pission product analyses of cake solutions from a standard laboratory extraction and from an extraction using fluosilicic instead of sulfaric acid showed very similar distributions of gamma activity. Since the total gamma activity was 3 or 4 times less with H2SiF6, it appears that a hold-back effect was exerted upon lanthanum as well as with zirconium and columbium where it was expected. Decostamination of barium was between 20 and 60 times better with H2SiF6 than with H2SO4. Relatively large amounts of H2SiF6 are necessary in extraction, however, since enough must be added to complemable the uranium.



Decontamination

Problem No. 203-X258 - Improvement in Decontamination

The potential difficulty of plugging jets and lines and tanks with BaSO4 from BaSO4 scavenging has not been solved. Zirconium salts are not satisfactory in dissolving large quantities of BaSO4. Greater attention is therefore being given the substitution of PbSO4 for scavenging active barium, since this salt can be more readily dissolved.

H2SiF6 for semi-works trials has been received on the plant, but tests will be delayed because of the greater importance of the current semi-works program.

Pission product analyses after a Ce-Er scavenged by-product precipitate, extraction and I Ce-Tr scavenged decontamination cycle indicate the chief gamma desponent to be lanthanum. This, and the level of activity, compares with similar observations after extraction and two Ge-Er scavenged decontamination cycles. As previously sisted, it thus appears that the same number and kind of alternate steps after quite similar decontamination regardless of viice is made first. A final experiment will include a Ge-Tr by-product, nor all extraction, and two decontamination cycles with Ge-Fr in by-product steps and Hesife in product steps. Further work will then avait availability of semi-sorks equipment.

Seil Rorma

The first three runs after the recent Hanford coassover experiments, 192-4, involved Bi. Be. CE, and Ir precipitation in the first half and Ce alone in the second half of the by-product precipitate of the first cycle. The second cycle was similar except that no BaSO4 was added. The average describmination factor through the second cycle was 106. The next three runs similar except that 35° C instead of 75° C was used for digestion after Ba and Bi precipitation, showed overall factors in the neighborhood of 104. The next three runs involving no Ba but Ce and Ir in both falves of both cycles and 40° C digestion, cave indications of low factors (104). It was decided, therefore, to make the following three runs, 201-3, with Ce and Er in both parts of by-product precipitates are with 75° C digestion. This will be useful for comparison with 205 Building runs not starting with similar conditions. The steps in those cycles are briefly as follows: exidize at 5 % HNO3, dilute to 1.25 % HYO3, add Bi solution, digest at 75° C, cool, add Na2Cr2O7, 464 Ce 2r solution, digest 30 minutes at 30° C, centrifuge, add more NaBiO3, add Ge-Zr solution, digest 15 minutes at 30°, and recentrifuge.

DECLASSIFIED - --



After run 203, it is passed to use plant 54P solution (BiPO₄ product cake after one decontamination cycle) from "half Hamford" runs a starting material for intensive study of the second deconta invition cycle and the crossover. It is unfortunate that the time was too short to make two cycle semi-works runs with Ce alone in the second half and Ba omitted in the first half of the first cycle, since it is not now clear whether Ba or Ce alone was responsible for the factors of 100 in runs 167-170 and 192-4. Sufficient data are probably available, however, to justify later plant trials of Ce alone in the second half. This of course would not be expected to demonstrate 100 decontamination until the full Hamford process cums are under tay in the 205 Building.

Problem No. 242-(21.8 - Aquipment Decontamination

Preliminary tests have been made with the 16 square foot parels painted with Tapogarth + CaCl2 films. When contaminated with neitralized semi-works vaste UNE solution, withing reduced the activity to background from a level of about 100 times background. With UNE solution and extraction product cake solution from 205 Building, the activity was reduced from about 300 times background to 3-5 times background.

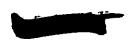
It is believed better results are obtained if the film is applied no two costs with some time allowed for partial drying. It was found a 2 roat film could be removed by ten cycles of & minute spraying with a fine mist and 2.5 minute soaking. The total time was 30 minutes and the vater consumption was 2 gals, per sq. ft.

Bulk Reduction

Problem No. 203-1255 - Improvement in Decontamination

Some publicity has been given to the possibility of using lanthanum oxalate for the by-product precipitate in the crossover. Laboratory tests have not shown as efficient gamma removel as when LaF3 is used. This conclusion is substantiated by specific fission product analysis of a product cake solution obtained by precipitating La(OH)₃ after the by-product La₂(C₂O₄)₃, and dissolving the former in H1O₃. Over 4O₆ of the gamma activity was not due to La, Ce, or Zr. Suspicion is directed toward an unknown rare earth-like cation which may be carried by LaF3 and La(OH)₃ but not by La₂(C₂O₄)₃. The existence of such a material has been hinted at occasionally in the past, but little definite information seems available. One other fact hampers the development of a crossover cycle is volving La₂(C₂O₄)₃ by-product and La(OH)₃ product pre-





cipitates, that is the very large amount of KOH that is necessary to raise the pH of the oxalate buffered system to a point the re the product-oxalate complex is destroyed and La(OH), is precipitated. The indications are that the amount of KOH that T would require is far above the present U. >, production and probably above the productive capacity.

Problem No. 202-X22S - Flocuulation of Process Precipitates

Evidence has been obtained in the laboratory that from the point of view of case of certrifuging the precipitate the degree of agitation is much more important during LaF3 precipitation from during the subsequent digestion. Tith either pre-or co-formed LaF3, removed by centrifuging is most complete when agitation is vigorous (comparable to that in the plant) during the strike. This indicates that the present plant pre-agitation is the most satisfactory known.

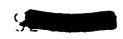
Problem No. 211-X238 - Product Isolation at W

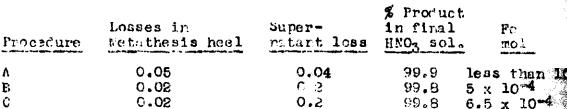
Two semi-works tests of the straight carbonate metathesis have been conducted in the BJ equipment. In both runs a large loss (56.5% and 20.9%) was encountered in the metathesis heel. The lostes in the KOH-K2CO3 supermetants were much less than was expected. Since the actual metathesis with K2CO3 has rarely given trouble in the neutine isolation procedure, and since the supermatant losses mentioned above were small, a laboratory study is being started using straight tank rather than a combination of bowl and tank metathesis and somewhat larger volumes. It is anticipated that further BJ runs will be made after new conditions are rorked out in the laboratory.

Problem No. 213-X67C - Hanford Isolation Studies

Three variations of the K₂CO₃ metathesis were tried on an aliquet of isolation batch 2%. Procedure a was the previous "standard" method is volving treatment of the LaF₃ cake (centrifuged from slurry water) with successive portions of 45% and 20% K₂CO₃ solution. Procedure B on the T scale would consist of adding solid K₂CO₃ to the 195 gallon LaF₇ slurry (from cell E of 224 Building) until the solution was 30% K₂CO₃. Procedure C was similar but a volume of 400 instead of 195 gallons was assumed for the T scale. all three involved the use of amnonium sulfide to remove also from the K₂CO₃ solution. The following table summarizes the results;

DECLASSIFIED





These results indicate the possibility of using much larger volumes than was formerly thought safe. No peroxide precipitations were made since the results would have been meaningless in the absence of knowledge of the zirconium content of the solutions. A method for this analysis is now under study and isolation work will be considerably handics pred until a method is perfected.

Isolation

Problem No. 213-X9C - Isolation of 49 from Room D Concentrates

Rater 22, mentioned last week as started in stainless steel, was completed in glass after incomplete oxidation was found in stainless. Improper heat treating of the new vessel is suspected of contributing to the oxidation difficulty. An aliquot of batch 23 is being processed in stainless to check the results.

Problem No. 211-X238 - Hanford Froduct Isolation

A promising lead has been uncovered to remove iron and other impurities that cause high peroxide solubility. The suggestion consists of making a product lodats precipitation instead of the first peroxide. Since this compound has a very low solutility, it can be precipitated from somewhat larger volumes than can the peroxide. It is possible that solution of the lodate might be simpler than the method formerdy used, mamely reduction with SO2 and dissolution in acid, precinitation of the hydroxide and dissolution of this in acid since new developments indicate that product locate may netathesize readily with caustic. It is not yet known, chowever, is the presence of impurities in plant solutions will rulse the solubility of the iodate. The metathesis procedure is also uncertain. Sufficient infortation should be available in another week to indicate viether this lead should be dropped or followed actively.

Problem No. 213-X67C - Harford Isolation Studies

Data have been obtained on the filtration of product peroxide. The first experiment used three centimeter diameter Pyr x ninetered glass crucibles of the "ine" and "medium" grade. As the case was not more than 0.5 cm thick in of ther case it was



DUH-1978

note a the rule of experience of chance of illusting the a the rule of experience of and the weather that the tention of according to the first eller and the first eller and the middle of the chartest of th

THE TOAL FIVILIER - CLIPTON

WETCARD ---

west teatonth. hooriging the state of the st

DECLASSIFIED