OP-59 Revised

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PROJECT #536

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K-X-Teller [illegible]

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File ____________________

Route List

1. L. Squires
2. C. H. Gruenwalt
3.
4.
5.

Date 10-10-44

Subject Clinton TNX Tech. Section Weekly Prog[illegible]

Report- Oct. 5 to Oct. 10-, 1944. (Separation [illegible] Proce[illegible]

To File

From Kirst

Copy No. [redacted]orthington

INV. C [illegible]

BEFORE READING THIS DOCUMENT, SIGN AND DATE BELOW:

Hood Worthington	10/20	Geo. H. Watt	11-17-44
D. H. Pelerin	10/27	[illegible]	11-18-44
B. T. Perkins	11-4-44	[illegible]	11/21/44
J. L. Dreher	11-4-44	M. E. Ballon	11/22/44
[illegible]	[illegible]	D. C. Lincoln	11/28/44
J. B. Work	11/4/44		
E. H. Turk	11/4/44		
D. R. Miller	11/6/44		
L. F. Ninne	11/6/44		
H. R. Hoekstra	11/6/44		
Wm. Johnson	11/9/44		
S. R. Gaarder	11/8/44		
[illegible]	11/8/44		
John W. [illegible]	11/11/44		
[illegible]	[illegible]		

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4-4-02

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OP-59 Revised

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Route List

1. ~~HW~~
2. ~~RWCrane~~
3. ~~DFB~~
4. ~~JAW~~
5. CHGamertsfelder

~~MKaltmas~~

~~PFGast~~

~~CWJWende~~

~~?. Spain~~

CI Bradford

Date 10-10-44

Subject Clinton TNX Tech. Section Weekly Prog

Report Oct. 5 to Oct. 10, 1944. (Pile Operati

To File

From Kirst

[redacted]

Copy No. H Worthington

INV. 9-47

BEFORE READING THIS DOCUMENT, SIGN AND DATE BELOW:

Hood Worthington	10/20
L. Squires	10/20
[illegible]	10-26
[illegible]	[illegible]
J. A. Wheeler	1944/11/8
C116	11/9
W. K. Woods	11/10/44
CW Wende	11/11
Paul F. Gast	11/10
J. B. Niles	11/17
E. Kirchner	11/18
Skuminsky	11-20
[illegible]	11 21 44
W. E. Webb	11/22/44
D. G. Toom	11/22/44

CP Cahill	11-24-44
G Andresen	11-24-44
S G Bartoff	11-27
[illegible]	12-16-44
L Bradford	12/28/44

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OP-59 Revised

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PROJECT #536

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Route List

1. ~~L. Squires~~
2. ~~C. Topenwalt~~
3. ~~McAcker~~
4. ~~C.H.J.~~
5.

Date 10-10-44

Subject Clinton TNX Tech. Section Weekly Progress Report Oct. 5 to 10, 1944. (Isolation)

To File

From Kirst

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[illegible]	10/20
J.E. Willard	10/25
[illegible] Watt	10/27
M. Lindner	10/28
~~L. Squires~~	11/9/44
[illegible]	11/23/44
P.J. Beaton	11/23/44
J. Woike	11/25/44

[redacted]

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This document consists of 11 pages. [redacted] figures.

[redacted]

1. R. E. DeRight
2. R. E. DeRight
3. H. Worthington
4. R. L. Doan
5. M. D. Whitaker
6. M. D. Whitaker
7. S. W. Pratt
8. W. O. Simon
9. W. O. Simon [redacted]

E. I. DU PONT DE NEMOURS & COMPANY

WILMINGTON, DELAWARE

Explosives Department - TNX

October 10, 1944

CLINTON TNX TECHNICAL SECTION WEEKLY PROGRESS REPORT

OCTOBER 5 - OCTOBER 10, 1944

The 49 process has now developed to a point where little further information of immediate interest to Hanford can be obtained at Clinton. Consequently this will be the last of the regular weekly TNX Clinton reports that have been issued since last October. At the same time since the results from the future program here should be made available to interested parties, the same number of copies of all reports from here that have been attached in the past will be sent to those now receiving them, namely, R. E. DeRight (2), W. O. Simon (2), H. Worthington (1). It is assumed that requests for special work required by difficulties arising at Hanford will be handled directly with Mr. Whitaker.

This being a final report, it seems well in cases where possible to indicate the future program and comment in general on the importance of it to Hanford.

Pile Operation

The repaired fan is now back in service and the power level is around 4200, which on the basis of an average down time of 10% gives a daily rate of 3900 KW. There are definite indications of a higher level as atmospheric temperatures drop. The present estimate is that during the winter the operating rate will be 4700 and the daily average 4300 KW. This is based on a maximum metal temperature of 250° C at the control point. The present flux distribution is good and there are no plans for altering it by further poison changes and it is expected that the current metal mass will also be maintained.

Two lines of work are under consideration for increasing the power output. The first of these involves the addition of a

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fine water spray to the cooling air from which an increase of 10% is expected. The second change comprises a more complete seal of the shield plugs at the charge face to prevent drawing in warm building air and to allow more cold air to be used. This is being considered more to conserve building heat than to increase power level as the probable power increase is not over 1%.

Neutron Thermometer

The 48 hour comparison with an ion chamber at constant load could not be made as the chamber galvanometer became erratic and required overhaul. The comparisons already made, however, show that such a device is as satisfactory as a chamber. In fact the use of four in series at the corners of the pile is thought to be the most reliable electronic method for power measurement. It is now planned to evaluate the neutron thermometer by connection to the Esterline Angus that originally had an ion chamber located so as to control the regulating rods automatically. Some thought is also being given to using four properly located thermometers in series to actuate the integrating KWH meter.

Power Level by Air Monitoring

Work is still continuing along this line with some success. The chief trouble, however, is with a variable air flow. This results both from an unsteady inlet air pressure and a variable discharge suction. This latter is caused by changes in the main cooling air flow as the activated air after passing the Beckman is drawn into the pile rather than discharged to the atmosphere. This method appears less attractive than the use of four neutron thermometers but enough more work will be done to reach a definite conclusion.

Activity on Surface of Canal Water

The expedient of sweeping the surface with a cheese cloth mentioned last week reduced the activity materially by removing the oil then on the surface. This latter, however, continues to accumulate and movements of buckets in the water tend to bring more activity to the surface.

The bulk of the original oxide has now been removed by a small pump with a hose extension on the suction side. This was used as a vacuum cleaner to remove all visible oxide from the floor of the deep pit and the floor of the canal is now being cleaned. The discharge of the pump is returned to the canal overflow and thence to the large settling basin.

A further attempt was made to clean the canal by the use of Orbit

(cardinol). This was done by holding the inlet water at 5 gals. per minute and adding an emulsion of 14 lbs. Orbit in water with agitation to give a cloudy appearance to the water. After standing 40 hours the water flow was increased materially to flush the canal. As a result of this treatment the activity of the exit water dropped by three fold. If the activity increases later due to pickup from the mattresses, this treatment will be repeated.

Tests to Demonstrate Xenon Poisoning

Several experiments were made to substantiate the Hanford experience and as a report covering them is being issued this week. No attempt will be made here to give a complete description. The final experiment, however, furnished a definite proof of the poisoning effect. In this the pile was operated for 24 hours at 4000 KWH in order to accumulate the poison to the equilibrum amount. When this had been done the pile was shut down and cooled as rapidly as possible, this taking five hours. All dampers were then closed to prevent air flow by natural draft and the critical point (K = 1) was determined by control rod movement using a power of 100 watts. It was necessary to insert the control rod gradually and this gave a smooth curve over a period of 21 hours. At the end of this time 46 channels were pushed and recharged with new metal, which change, gave a vertical drop in the curve. No further measurements were taken after this metal change.

At this writing it is not possible to express the equilibrum poison effect per 1000 KW in "inhours" as two different values had been obtained for rod calibration. The first of .7 inhours/inch is based on small changes in period at 100 watts rating, while the other (1.3) was obtained by determining period for a wider spread of settings, that is for a wider spread of periods.

In addition to the two different calibration values the uncorrected decay curve fits a 15 hour rather than a 9.6 hour period. It is expected that these difficulties will be reconciled by further calculations and experiments now in progress to a point where the report to be issued this week will present much more definite conclusions. It can be said, however, that the poisoning effect appears to be confined to fission elements with short half lives.

Problem No. 124-X1E - Effect of Radiation on Corrosion of Tubes and Jackets

The tube at 85° C operated satisfactorily last week bringing the total time in service to 48 days. It is now planned to continue, unless trouble occurs, until the last of October and then discharge the slugs for examination and weighing after about 70 days of operation. No plans have been made beyond this point at pres[illegible] and there would appear to be but limited merit in further tests o[illegible]

this nature. If they were stopped by slug failure no knowledge would be gained and to be of any value they would have to run for so long that Hanford experience would probably be much more representative. The main point of interest has been the effect of radiation on corrosion rate and this has been amply proved to be negligable.

Problem No. 124-X14E - Effect of Radiation on Film Formation

No further results were obtained last week. This line of work also appears to be of limited value to Hanford as it has been demonstrated that radiation has no effect on film formation. In addition the amount of dichromate decomposed has not only been demonstrated by this work but is in agreement with other results here and at Chicago.

Monitoring 205 Stack

Air has been drawn through the sampling line and equipment for almost a week after solution of the last metal change. Over this period the residual iodine activity dropped slowly to almost the instrument background. After following one more metal solution charge the equipment will be turned over to the Health Physics Group for routine operation.

This method of monitoring is considered satisfactory for Clinton where the gross activity is low. The chief fault of the equipment lies in a 20% variation in air sample size this being about as close as the flow can be regulated. A variation of 10% is considered to be much more satisfactory and possibly necessary for Hanford.

New Slug Coating

The sodium aluminum silicate (from the Bureau of Standards) coating is being evaluated further in a water tube containing a new lot of steel dummies coated with a thicker (1 mil) and somewhat different composition. The previous test with steel slugs showed numerous rust spots after several weeks exposure to water at 85° C and the present slugs will be kept under similar conditions until November 1 before being examined.

A method has now been developed by which it appears possible to apply a continuous coating to heavy metal. The slugs are cleaned, etched in nitric acid, washed, dried and a coat applied as a spray. When dry, this is baked on. Coated heavy metal slugs for test are expected shortly.

While the film is thick enough to prevent fission recoils from entering the water its utility will depend on preventing an increase in water activity through pin holes long enough to hold

the activity in the exit water below the tolerance limit and still form a practical amount of product in the metal. This coating is not an immediate possibility but it has many desirable features if it could be made to give adequate protection.

Detection of Bulged Slugs

Work is continuing on the method involving the use of a continuous column of slugs in electrical contact and insulated from the tube. It has been possible to cause an insulated slug to bulge and to detect it before it became too tight to push. Detection is accomplished by completing a current between the insulated slug and the tube. At present, it is planned to accomplish insulation by holding sections of mica to the slug at either end by a thin aluminum band. One end cap of each tube would have an insulated terminal extending inward to contact the last slug. Leads from these would then be brought to a scanning device the other side of the circuit being completed through a wire to the shielding. One problem involved is the disposal of the insulation during metal solution. Another is the insurance of adequate insulation without displacing the slugs so as to alter materially their present concentricity in the tube. It is also possible that bringing all the wear at two small areas might, in view of the weight of the slugs, cause the bands to cut through. In general, however, this appears to be the most promising and easiest applied of the various schemes being studied and it has been suggested that this work be followed actively until a definite proposition can be submitted.

The use of high frequency vibrations initiated at one end of a tube and monitored at the other, with a definite effect resulting from the interference of a bulge, has been found to be unsatisfactory. No reliable and reporducable effects could be found.

At present the possibility of using a beam of light is being studied. This appears to offer major difficulties. In the first place, the annulus is narrow and long and constant and perfect collimation of a beam of light would be difficult. Furthermore, this method would require a transparent end in one end fitting and a light source in the other. Finally visual observation would be unreliable due to the human factor while the use of photography would be extremely tedious. A very great number of pictures would be required and since the expected effect would be small the procedures of exposure, developing and interpretation of the films would have to be rigidly standardized. Finally, it is doubtful if this method could be used with the pile in operation.

It is understood that a trial will be made later in SMX of the method by which a slug column is moved a short distance and then returned to its original position with a measurement of the force required. The necessary end fittings will be provided by Chicago.

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SEPARATION PROCESS

A nine timer using the standard Hanford flowsheet process with 60 day metal has just been completed with the following results;

Decontamination Factors

	Ext.	1st By-Prod.		1st Prod.		2nd By-Prod.		2nd Prod.	
Run	Step	Step	Cum	Step	Cum	Step	Cum	Step	Cum
227	6	40	240	9.9	2360	7.5	17560	19	334000
228	9	35	325	10.5	3400	14.7	50000	9	450000
229	4.8	62	300	12	3600	2.1	7700	10.2	788000
230	8	46	365	18.5	6800	5.3	35800	23.8	850000
231	7	41	290	42.5	12300	2.2	27300	113	3100000
232	5	70	350	8	2800	17.8	50000	11.0	550000
233	8	32	255	15.7	4000	4.5	18000	11.3	203000
234	8	37	300	23	7000	2.2	15300	30	460000
235	6	43	257	26	6700	-	NS	-	900000
Weighted Avg. Composite	6.2	48	300	13.3	4000	4.7	18800	26.5	500000 490000

Measured Yield Losses

	Ext.	1st By-Prod.		1st Prod.		2nd By-Prod.		2nd Prod.	
Run	Step	Step	Cum	Step	Cum	Step	Cum	Step	Cum
227	3.2	3.1	6.3	4.2	10.5	3.6	14.1	1.9	16.0
228	1.2	3.3	4.5	2.1	6.6	5.7	12.3	1.2	13.5
229	1.3	4.3	4.6	2.1	6.7	4.2	10.9	0.8	11.7
230	1.8	4.1	5.9	1.8	7.7	4.0	11.7	0.5	12.2
231	1.5	4.0	5.5	2.2	7.7	3.8	11.5	0.9	12.4
232	2.4	3.6	6.0	3.0	9.0	3.9	12.5	0.6	13.1
233	1.2	3.6	4.8	1.7	6.5	4.0	10.5	0.7	11.2
234	2.5	2.7	5.2	1.7	6.9	3.7	10.6	0.9	11.5
235	1.5	2.2	3.7	1.0	4.7	3.8	8.5	1.0	9.5
Avg.	1.84	3.43	5.27	2.2	7.47	4.1	11.57	.93	12.5

This series was double scavenged in each cycle with Ce and Zr and no fluosilicate was used in the product precipitations. It will be noted that the decontamination was satisfactory with a maximum of 3,100,000, a minimum of 203,000 and a weighted average of 500,000 through extraction and two cycles. The composite was 490,000. The yield losses averaged 12.50 overall with all values about as expected.

Comparison of Demonstration Runs

So far six series of runs have been made to demonstrate the Hanford process or slight modifications of it. The weight averaged decontamination factors and the arithmetic average yield losses from

these series are summarized in the following tables.

Summary of Data for Decontamination Cycles of W Runs in 205 Bldg.

Conditions

	1st Cycle		2nd Cycle		
Run Nos.	By-Prod.	Prod.	By-Prod.	Prod.	Metal
186-193	Bi;Ce-Zr twice	Bi	Bi;Ce-Zr twice	Bi	100/60
194-201	Bi;Ce-Zr; Ce	Bi	Bi;Ce-Zr;Ce	Bi	100/60
202-209	Bi;Ce-Zr; Ce	Bi	Bi	Bi+$SiF_6^=$	100/30
210-217	Bi;Ce-Zr twice	Bi	Bi;Ce-Zr twice	Bi	100/30
218-226	Bi;Ce-Zr twice	Bi + $SiF_6^=$	Bi	Bi+$SiF_6^=$	100/30
227-235	Bi;Ce-Zr twice	Bi	Bi;Ce-Zr twice	Bi	100/60
255-263	Bi	Bi + $SiF_6^=$	Bi	Bi + $SiF_6^=$	100/60

Weighted Average Cumulative Gamma Decontamination Factors

		1st Cycle		2nd Cycle		
Run Nos.	Extraction	By-Prod.	Prod.	By-Prod.	Prod.	Composite
186-193	6.5	218	4450	5160	54200*	383000
194-201	6.1	195	2600	8880	184000	207000
202-209	8.8	162	4000	6000	180000	188000
210-217	8.2	112	4700	10330	837000	630000
218-226	7.6	163 (2.4)	5140 (1.4)	7300 (1.4)	255000 (3.5)	400000 (6.5)
227-235	6.2	300 (4.8)	4000 (1.3)	18800 (4.7)	500000 (24.6)	490000 (2.6)
255-263	7.0	32 (.5)	830 (.11)	6000 (.25)	130000 (.26)	160000

*Low due to recontamination of samples

Average Cumulative measured Yield Losses (in percent)

		1st Cycle		2nd Cycle	
Run Nos.	Extraction	By-Prod.	Prod.	By-Prod.	Prod.
186-193	2.9	6.6 (3.7)	8.9 (2.3)	13.3	15.4
194-201	2.4	5.3 (1.9)	6.3	9.3	10.1
202-209	2.0	5.3 (4.3)	6.0	6.7	6.9
210-217	1.9	5.6 (3.1)	8.8 (3.2)	12.7	13.5
218-226	1.6	5.2 (3.6)	6.3 (1.1)	7.3 (1.0)	7.9 (0.4)
227-235	1.84	5.27 (3.43)	7.47 (2.2)	11.57 (4.1)	12.5 (0.93)
255-263	2.5	4.2 (1.7)	4.5 (0.3)	4.8 (0.3)	5.0 (0.2)

It will be noted that series 186-193 and 227-235 are checks of the flowsheet process with 100/60 day metal and that series 210-217 is with the same process and 100/30 day metal. All three of the series (25 runs) give very satisfactory decontamination and there is no difference between 30-and 60 day metal. The arithmetic average of all three gives a factor of 621,000. The overall losses for the three series are in good agreement with an average value of 13.8%.

Omission of Zr in the second half of the by-product precipitates (one quarter of total) (runs 194-201) showed a lower decontamination factor (207000) and a lower loss (10.1%) with 100-60 day metal.

When three quarters of the zirconium and half the cerium scavengers were omitted (runs 202-209) by using Ce-Zr in the first half and Ce only in the second half of the first by-product precipitate with no scavengers in the second by-product but fluosilic acid in the second product precipitate the factor was slightly lower (188000) and the yield loss was reduced to 6.9%.

Finally when the first cycle was fully scavenged (runs 218-226) the second by-product was straight bismuth phosphate (half normal zirconium and cerium) and fluosilicic acid was used in both product precipitates the factor rose to 400,000 and the overall loss to 7.9%.

It may be concluded from the above data that the flowsheet process gives the best decontamination at a somewhat higher loss as operated at Clinton. If the semi-works runs at Hanford product and by-product levels are accurate, however, this loss should be under 10% at full Hanford product concentration. There appears, therefore, to be little justification for using any of the modifications since they all result in lower decontamination factors and only promise a lower yield loss that may not be demonstrable at Hanford.

Additional runs are to be made at Clinton that may be considered as a part of a further process demonstration. Since a specific process has been recommended, however, and flowsheets have been issued the results of these later runs are not of primary importance. Furthermore, judging from the effect of the variables already tried, there is little possibility that any desirable improvements will be shown.

Crossover

The data for the crossovers of five of the six series of runs already discussed may be summarized as follows:

		Average Losses			
Series	Bi By-Prod.	LaF_3 By-Prod.	LaF_3 Prod.	Total	
186-193	1.5	1.25	2.2	4.95	
194-201	1.0	1.5	1.4	3.90	
202-209	0.3	1.0	1.0	2.30	
210-217	0.90	1.6	0.6	3.10	
218-226	0.38	1.5	1.0	2.38	
227-235	0.9	Not yet completed	1.5	3.70	
			1.3		
Group Avg.	0.8	1.37	1.24	3.41	

The above losses are so consistant (41 runs) that there is little doubt that the crossover will be satisfactory at Hanford.

Neutralization of Wastes

At the request of Hanford tests have been made in 205 Building to determine if there is any difference in the power required for agitation when neutralizing waste metal solution with caustic plus carbonate instead of straight caustic. No difference in power was found.

This was not requested. It was desired to know the power - time relation during neutralization to determine if a max. existed at time of max. precipitation of solids.

ISOLATION

204 Operation

The nine timer (218-226) was treated in 204 in three lots of three charges each.

Lot 1 comprising runs 218, 219, and 220 contained no recycled supernatant and the product concentration was too low to give a precipitate. After destroying the peroxide in the supernate in Room D it was divided into three parts and returned to charges 224, 225, and 226 prior to the LaF_3 by-product precipitation.

Lot 2 containing charges 221, 222, and 223 plus recycled supernate from 216 and 217 gave a precipitate and the solubility in the supernate was 86 mgs. This solution, after destruction of the peroxide, will be returned to later charges.

Lot 3 consisting of charges 224, 225, and 226 plus returned supernate from 218, 219, and 220 showed a solubility of 79 mgs. The supernate will be returned to later charges.

The combined first precipitates were divided into two portions for the second precipitation. This was entirely satisfactory with supernate solubilities of 52.5 and 19.7 mgs respectively. There appears to be little doubt of the satisfactory operation of this portion of the process.

Equipment entirely similar to that at Hanford for carrying out the first precipitation is expected to be ready for operation here later in the week. When this is available the process will be standardized along the following lines. Two or three charges will be combined after the first metathesis in Room D at ¼ volumes and sent to 204 Building. Here the solution will be put through the first precipitation under Hanford conditions and the product saved. The supernate will be returned to Room D for peroxide destruction and return to the process ahead of the LaF_3 by-product precipitation of later charges. The first precipitates will be combined into larger amounts for final precipitation but not at Hanford volumes.

The above procedure should not only simplify Clinton operations but provide a continued demonstration of the most critical part of the Hanford isolation process.

Peroxide Destruction

The supernates from 204 are now transferred to Room D and analyzed for peroxide with standard permanganate. The solution is then agitated and held at 15-20° F while being treated with a solution of $NaNO_2$. In order to insure complete peroxide destruction two spot test indicators are used. The first of these ammonium vanadate

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(1.5 grams of 25 cc of 8 N H_2SO_4) shows red with peroxide changing through pink to yellow with no free peroxide or nitrite and greenish blue with excess nitrite. Sodium nitrite solution is added with spot testing until the pink color disappears. When the peroxide is destroyed as shown by this test a slight excess of nitrite is insured by the use of a second indicator: benzidine dehydrochloride (.1 gms plus .25 cc DM aniline in 30 cc of 6 N acetic acid). This is colorless in the absence of nitrite and turns pink as free nitrite is added. More nitrite solution is then added, with spot testing, to a definite pink color. This excess nitrite is then destroyed by adding permanganate to a permanent pink color.

So far it has been found that only 60-80% of the nitrite required based on analyses for peroxide is necessary to remove all the peroxide. This is thought to be due to decomposition while agitating and waiting analysis, a period of about 30 minutes.

TECHNICAL DIVISION - CLINTON

W. E. Kirst

W. E. KIRST

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