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200 Area Reports for Technical Progress Letters #52 thru 78 (7-5-45 thru 12-31-45)

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AUTHOR
M. F. Acken

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200 AREAS
June 29 Through July 5

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INV. 2-47

Canyon Buildings (221)

Metal Dissolving (B)

The second dissolver (3-5-R) has been placed in service in the B Canyon. Three buckets of metal (approximately 2500 lbs.) were added for a heel. With this heel, the dissolving times of the first three charges were 6-1/4, 5, and 6-1/2 hours, respectively, approximately equal to those obtained with the regular dissolver (4-5-L). Upon the completion of three more dissolvings, 3-5-R will be held as stand-by for 4-5-L.

Solids in Product Solutions (B and T)

The presence of undissolved solids in the product solutions (8-4-P, 14-4-P, 17-4-P) has been detected frequently, but only rarely have these caused process difficulties. The solids have been of two types: high turbidity solutions (greater than 1g/l) in which the solids have analyzed over 50% bismuth, and low turbidity solutions (less than 0.1g/l) from which the solids have not been analyzed. The solution of the extraction cake frequently contains small amounts of undissolved matter throught to be carried through from the dissolver (slug bonding residues) and small amounts of columbium and zirconium (fission elements) precipitated during metal dissolving or extraction. These have not caused any process difficulties.

The solutions of the first and second cycle product cakes have contained, in addition to infrequent undissolved bismuth phosphate, small quantities of a material not immediately identified. It has been observed that the silico fluoride solution, upon standing, will deposit a slime which is insoluble in 50% nitric acid. Upon the possibility that this is the correct explanation of the solids, the silico-fluoride make-up procedure at the B Canyon has been altered to provide a 24-hour settling period with a side tank draw-off (10 inches above the bottom) to prevent adding this slime to the process solution.

Bismuth Phosphate Metathesis-KOH Washing (T)

Three runs (T-5-06-D-9, -10, -11) have been completed in the T Canyon in which second cycle product cake was converted, in the centrifuge bowl, to bismuth hydroxide before dissolving in a reduced quantity of nitric acid (Production Test

SE-221-T-PA-6). The objective is to provide a means for the Concentration Building to operate at 40% standard process volume. The net effect is not known at this time since the runs have not yet progressed through the Concentration Building.

Based on the average of the three runs, the second cycle product waste, including the metathesis waste and washes, increased slightly, from 0.34% (average of last 10 runs) to 0.49%. The present manipulative procedure still places too much load on the centrifuge motor, causing the thermal over-load switch to open. On subsequent runs, the procedure is to be modified by the elimination of part of the load on the centrifuge motor in an attempt to eliminate the "cut-outs".

Precipitator Sprays (B and T)

Experiments have been made in both the B and T Canyons on the performance of the precipitator sprays (squirters) in the second cycle product precipitator (17-1). These tests indicate that less temperature rise and probably more consistent operation of the sprays results from operating at 60-70 pounds instead of the full pressure of about 100 lbs./sq.in. Both canyons will operate the squirters at 60 ± 10 lbs./sq.in. when the quantity of acid added to the precipitator for dissolving the centrifuged cake is insufficient to reach the agitator blades (approx. 2300 lbs.); the circulation produced by the squirter is the principal means of dissolving the precipitate heel remaining in the precipitator.

Cell Inspection (T)

All process cells in the T Canyon have been inspected from the crane or deck level, depending upon the hazard. The most consistent observation was the severe rusting of unprotected surfaces. The air blowing procedure for cooling jets [illegible]doubtedly has increased this corrosion problem. The use of alternate jets, wherever available, will be adopted as soon as the maintenance work can be done and should reduce the corrosion rate in the cells.

The exposed parts of the skimmer on the second cycle product centrifuge (17-2) were so badly rusted that treatment with penetrating oil and a brass hammer was required before it could be used on Run T-5-06-D-7.

Concentration Buildings (224)

Reduced Process Volumes

Production Test No. S.E.-221-T-PA-6 was started on Run T-5-06-D-9. This test involves KOH metathesis of the second cycle $BiPO_4$ product cake in Section 17 and dissolution of the metathesized cake in 40% of the former standard volume of HNO_3. The object of this test is to provide smaller process volumes for the Concentration Building. These smaller volumes permit the carrying out of the two LaF_3 product strikes at 120mg/l La each, instead of at 80mg/l, for the same total weight of La (2.5 lbs.).

One run only, T-D-9, has thus far been completed in the three-run test series. The LaF_3 product waste (E-3-WS) for this run was 1.0%, corrected for recycled product to a performance basis (actual weight of product present).

Increased La Addition

Production Test No. S.E.-224-B-PA-1 was started on Run B-5-06-B-8 at B Area and is continuing through Run B-13. This test involves the addition of 50% more La to

[redacted]

each LaF_3 product strike in the 60% volume process. This process also results in 120mg/l La per strike, obtained at the expense of increasing the total La to 3.75 lbs.

The LaF_3 waste loss (E-3-WS) data thus far obtained are shown below. The runs thus far made have produced an average two-centrifugation loss of 1.3%. This is approximately 0.5% lower than the previous B Area average of 1.8% for two 80mg/l strikes.

LaF_3 Product Waste Losses (Test No. S.E.224-B-PA-1)

Run. No.	mg. La/ liter/strike	% E-3-WS Losses Cent.#1	Cent.#2	After HNO_3 Wash
B-5-06-B-8	120	6.2	0.89	1.0
B-5-06-B-9	120	8.6	1.3	1.5
B-5-06-B-10	120	6.8	1.2	1.2
B-5-06-B-11	120	7.8*	1.3*	1.5*

* Corrected for 9.5% recycle added to LaF_3 by-product

Some difficulty was encountered in the first two runs in the slurry removal of the LaF_3 product cake from E-2 centrifuge to F-1 metathesis tank. On Run B-5-06-B-9, in particular, the Beckman meter reading on the supposedly empty E-2 centrifuge was halved by adding an extra 250 lb. water slurrying to the regular cake removal procedure. The F-1-PS assays (LaF_3 product slurry prior to metathesis) contained 81% and 97% product respectively before and after the extra bowl slurrying. This additional wash has been incorporated in the procedure for the remainder of the heavy-cake runs and no difficulty is being experienced.

Recycling Tests

Production Test No. SE-224-T-PA-6 is still being continued. This test involves the recycling of the equivalent of the isolation supernatants from two charges to the LaF_3 by-product of each production run in Cell D. The weight of La thus supplied to the by-product (5.0 lbs.) makes up the LaF_3 cake entirely, with no additional La supplied.

The data for the runs thus far made are shown in the tabulation below. For the 60% standard volume runs, no noticeable increase in the LaF_3 by-product losses (D-4-BP) has been obtained, the average being 0.78%. For the 40% volume runs, however, the D-4-BP loss has increased to an average of 1.02%. Although the weights of the LaF_3 by-product cakes were slightly increased during these latter runs, due to the return of the isolation supernatants from the 50% extra La runs at B Area, it is felt that the increased losses are due to the more concentrated centrifuge heels after skimming. The LaF_3 cake in D-2 is washed by displacement at present and it is proposed to replace this with the former method of slurry washing to improve the displacement of the high-product heel from the D-2 bowl.

The LaF_3 by-product decontamination factors have remained unaffected on both volume scales.

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LaF₃ By-Product Losses and Decontamination

Run No.	Process Volume	% 8-1-MR Recycled	Lbs. La	Decontamination Factor	% D-4-BP Loss	
					Uncorrected	Corrected
T-5-06-B-3	60% Std.	6.3	5.0	12.7	0.65	0.61
T-5-06-B-4	60% Std.	6.5	5.0	14.8	1.1	1.03
T-5-06-D-5	60% Std.	5.6	5.0	11.2	0.82	0.78
T-5-06-D-6	60% Std.	6.2	5.0	12.4	0.81	0.76
T-5-06-D-7	60% Std.	8.0	5.0	7.8	0.88	0.81
T-5-06-D-8	60% Std.	7.2	5.0	11.2	0.77	0.72
T-5-06-D-9	40% Std.	7.4	5.0	15.0	1.1	0.99
T-5-06-D-10	40% Std.	7.9	5.25	10.8	1.0	0.92
T-5-06-D-11	40% Std.	10.0	5.44	--	1.27	1.15

*Corrected to performance basis for amount of recycle added.

Metathesis Losses

It has been discovered recently in the laboratory that chromium has a pronounced detrimental effect on both the carrying and separation of product by $La(OH)_3$ in the metathesis operation. Samples of the LaF_3 by-product effluent (D-3-OS) and the LaF_3 metathesis slurry (F-1-PS) are being analyzed for chromium in the current series of runs, and an attempt to obtain a plant correlation with the 15% KOH metathesis losses (F-7-WS) is to be made.

Further work has shown that manganese has a beneficial rather than a harmful effect on metathesis. Since most of the crhomium comes from the $Na_2Cr_2O_7$ holding [illegible]idant added prior to the cross-over $BiPO_4$ precipitation, it would be desirable [illegible] substitute $KMnO_4$ for the $Na_2Cr_2O_7$ holding oxidant. Preliminary confirmatory tests of this proposal are now being carried out in the Cold Semi-Works.

Waste Tank Solids

The depth of solid precipitate on the bottom of the Waste Settling Tank (361) was measured as 2.0 ft. on May 13. On June 29, after eighteen additional charges had been processed, the cake depth was again measured and found to be 2ft.4inches, an increase of 4 inches. The liquid free-board to the overflow is still 12 ft. 8 inches. In addition, the precipitate was found to be very loosely packed and quite capable of being slurried and jetted out to the Waste Farm.

Impurity Backgrounds

Since the start of the more extensive washing of the $BiPO_4$ product precipitation tank (17-1) in the B Canyon Bldg. by longer circulation of the dissolving acid through the distributor, more insoluble impurities appear to be appearing at the Concentration Bldg. This is indicated by the empty tank Beckman readings or "background". The oxidation tank (C-4) Beckman background has risen from a former level of 700 to 1500×10^{-14} amps. This is almost four times the T Area C-4 background. The A-4 background at B Area confirms this rise, since it has also risen from its former level of 300 to 700×10^{-14} amps. It is expected that the present leveling off of this contamination pick-up will be retained or reversed as the clean-out of 17-1 proceeds.

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Product Centrifuge Beckman Readings

The Beckman readings on the LaF_3 product centrifuge (E-2) and the $La(OH)_3$ prod[illegible]t centrifuge (F-2) have been too low to be accurately measured with the original G.E. tube installation approximately 4 ft. from the centrifuge casing. Recently new tubes have been installed at both centrifuges in both T and B Areas at a distance of about 4 inches from the casing to improve the sensitivity of measurement. At B Area both the new and the old tubes are connected to the Beckman meter so that direct comparisons are available. Some recently obtained data are as follows:

Product Centrifuge Beckman Meter Readings*

Run No.	LaF_3 Prod.Centrifuge(E-21)				$La(OH)_3$ Prod.Centrifuge(F-2)			
	4 ft.from bowl		4 in.from bowl		4 ft.from bowl		4 in.from bowl	
	Full	Empty	Full	Empty	Full	Empty	Full	Empty
B-5-06-B-8	14	1	150	25	35	0	320	2
B-5-06-B-9	11	0	125	24	--	0	350	0
B-5-06-B-10	13	2	155	28	28	1.5	290	0
B-5-06-B-11	7	0.5	81	18	--	--	--	--
T-5-06-D-7			135	7			415	0
T-5-06-D-8			135	16			400	5
T-5-06-D-9			100	0.5			300	0

*All readings quoted in amsp. x 10^{-14}.

A factor of approximately ten in sensitivity improvement has been obtained by [illegible]e change in tube placement. The E-2 empty bowl readings are now measurable. The [illegible]2 empty bowl readings, while still nearly zero, are now more reliably so.

Isolation Building (231)

Product Accounting

All tanks and lines (except N-1 First Nutsche anf F-2 Sump Tank in Cells 3 and 4 were flushed repeatedly with 1N HNO_3 during the past 2 weeks in preparation for necessary maintenance work. Of the 140.8 units missing by Cell 4, cleanout 16 recovered 147.5 (72.6 from Cell 3 and 74.9 from 4), while cleanout 17 picked up about 58.1 from Cell 4. The total amount unaccounted for is thus only 19.7 units, although Cell 3 is short 68.2 units while Cell 4 is over 48.5 units. This anomaly is unimportant when considered in the light of the large total amount of product handled in each cell.

Control of S-1 (Still) Evaporations

Evaporation to the desired concentration, 225g/l, is controlled by observing the volume of condensate collected in R-1. Since the adjustment tank (AT) and condensate receiver (R-1) volumes always total more than the vacuum-receiver (PR-3) volume charged to the still, an empirical table is used to determine the volume to collect in R-1 for a given batch. An effort has been made to improve the accuracy ot the table by tabulating AT and R-1 volumes for 20 most recent runs in Cell 3 and 30 in Cell 4. The study indicates "effective" PR-3 volumes of 9.47 and 9.20 liters for cells 3 anf 4, respectively, as compared with calibrated volumes of 9.345 and 9[illegible]03 liters.

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Cold Semi-Works

Oxidation with Lead Oxide

Further tests were made in the Semi-Works in order to evaluate the use of Pb_3O_4 as a stustitute for $NaBiO_3$ in the oxidation step. As mentioned in the last report, an average 1.5% by-product loss was obtained in two runs when oxidations were made in 7.5N HNO_3 solutions using 0.01M Pb_3O_4 and 0.008M $KMnO_4$ concentration. The conditions for these runs were as follows: (1) the oxidations were made for 2 hours at 60°C, (2) the $KMnO_4$ was added after 1.5 hours of the oxidation period had elapsed, and (3) additional $KMnO_4$ (equivalent to 0.0003M on the diluted solution) was added during the by-product precipitation.

In Run AP-35, the additional $KMnO_4$ was omitted and a by-product loss of 2.1% was obtained. In Run AP-36, the additional $KMnO_4$ was omitted and the 7.5N HNO_3 solution was made 0.008M in $KMnO_4$ at the start of the 2-hour oxidation period, with the resultant by-product loss of 1.85%.

The results of these two runs indicate that by omitting the $KMnO_4$ in the by-product precipitation an increase of 0.5% in by-product loss is obtained..

There was some indication that more slurrying was required to remove the by-product precipitate from the bowl when the $KMnO_4$ was added at the start of the oxidation period than when added near the end of oxidation.

Replacement of $Na_2Cr_2O_7$ by $KMnO_4$ as Holding Oxidant

Since the presence of chromium tends to increase the metathesis waste losses, it is desirable to maintain the concentration of this metal at a minimum figure. As a step in this direction, a series of Semi-Works runs are being made in which $Na_2Cr_2O_7$ is replaced by $KMnO_4$ as the holding oxidant in the cross-over cycle.

The oxidations are made in 5N HNO_3 solution using 0.01M $NaBiO_3$ concentration and 1-hour oxidation time at 50°C. At the end of the 1-hour period, the oxidized solution is made 0.01M in $KMnO_4$ and then diluted for the $BiPO_4$ by-product precipitation. The effluent from this step is made 0.01M in $KMnO_4$ prior to the LaF_3 by-product precipitation.

Analytical results on the first completed run showed a 0.6% loss in the $BiPO_4$ by-product step.

Since the $BiPO_4$ by-product cake was satisfactorily removed when using HNO_3 and H_2O_2, the cake removability is to be checked without the use of the supplemental peroxide.

Replacement of Ammonium Fluosilicate by Ammonium Fluomolybdate

One exploratory tracer run was made in the Semi-Works in which ammonium fluomolybdate was substituted for ammonium fluosilicate in the product precipitation step. In this run the $BiPO_4$ cake removal, cake solution, and product carrying appeared to be similar to results obtained with ammonium fluosilicate.

Process Chemistry

Alternate Procedure for Treatment of Isolation Building (231) Peroxide Waste Solutions

[redacted]

[redacted]

The waste solutions from the peroxide precipitation steps, after destruction of the peroxide, are transferred to the Concentration Building for recycling in the process. The volume of these solutions (about 150 liters per run) is objectionably large and four transfer cans are required for each batch. A project already approved for the installation of a large transfer tank mounted on a truck could be abandoned if the volume of solution to be recycled could be reduced about four-fold.

Attention has therefore been given to the development of an alternate method for treatment of these solutions in order to recover the product in a comparatively small volume. A promising procedure, which has been worked out on a laboratory scale, involves the partial neutralization of the waste solutions (to 0.5N or slightly less) with 25% KOH solution, addition of oxalic acid to precipitate lanthanum oxalate, addition of more KOH to make the solution slightly alkaline, gravity settling of the precipitate (which is rapid) decantation of the supernatant, washing of the precipitate with water, and final dissolution in HNO_3 and destruction of the oxalate with $KMnO_4$. In the laboratory runs, carrying of product under these conditions has been satisfactory and losses in the supernatant and wash solutions have been low (1.5 - 2%). On the basis of the laboratory scale tests, the final solution volume to be recycled per run would be about 30 liters or more than a four-fold reduction in volume. Preliminary work indicates that it may also be feasible to leave the precipitate in the tank and bring in another charge thus making it possible to combine several lots before dissolution.

More rapid decomposition of H_2O_2 occurs in alkaline than in acid solutions and effervescence at room temperature is excessive. Tests made at lower temperatures (5° - 7°C - attainable in the cooled catch tanks) showed that effervescence was decreased, but it was not possible to determine from the laboratory scale tests whether it had been decreased sufficiently so that satisfactory settling of the precipitate is not prevented by the escaping gas bubbles. Such tests are to be made on a plant scale.

Product Losses in the Metathesis Waste Solutions

It was reported last week that the presence of chromium was found to have an adverse effect both on product carrying by $La(OH)_3$ and on relative ease of separation of this precipitate from the KOH metathesis slurry. Another impurity which may be present is manganese. However, tests indicate that it does not interfere with carrying and actually improves the centrifugation characteristics of $La(OH)_3$, probably by a weighting effect.

Tests have also been made on the effect of chromium and manganese on carrying by LaF_3 and on relative centrifugation of this precipitate but no effect on carrying was found and centrifugation properties were adversely affected only to a very slight extent. Accordingly then, by the elimination of chromium from the process by substituting $KMnO_4$ for $Na_2Cr_2O_7$ as the holding oxidant in the cross-over cycle oxidation step (provided significant amounts of chromium are not introduced through corrosion), it would be expected that metathesis waste losses would be decreased but that losses in the LaF_3 product precipitation step wastes would not be altered.

Tests on the use of $KMnO_4$ instead of $Na_2Cr_2O_7$ as the holding oxidant in the cross-over have been made in the Cold Semi-Works and satisfactory results have been obtained.

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M. F. Acken

M.F.Acken, Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.

MFA:jd

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July 14, 1945

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INV. 2-47

200 AREAS
July 6 Through July 12

Canyon Buildings (221)

Coating Removal (B and T)

Removal of the aluminum jackets by the slow caustic addition procedure is being continued in the B Canyon (Production Test SE-221-B-PA-1), while in the T Canyon the temperature control procedure is being followed. In general, the slow addition method permits a higher vacuum to be maintained in the dissolver during the reaction.

Extraction (B and T)

The transfer jet between metal solution storage and the extraction precipitator (4-7 to 8-1) is a high capacity jet (75 gal./min.). With such rapid transfer any difficulty with the weight factor meter or the gang valve which results in not turning the jet off promptly causes a considerable excess of solution to be received, with no way to return it to storage. On a recent run in the B Canyon (B-5-07-B-2) the gang valve stuck open and before it could be closed approximately 1100 additional pounds of solution were received into the precipitator. No additional dilution was made and the extraction waste loss was high, 3.2% (see below). In the T Canyon (T-5-07-B-2) the weight factor meter stuck momentarily with the result that the extraction weight of metal solution was high by approximately 2200 lbs. Additional dilution was made to compensate for this and the extraction waste loss was normal (0.66%). Replacement of this 75 gal./min. jet with a 20 gal./min. jet is being considered.

The high waste loss on run B-5-07-B-2 of 3.2% is thought to be due in part to the higher UNH concentration (25.5-26%) and in part to the metal solution used:

Run No.	Dissolver Soln.	UNH Conc.	H Conc.*	Waste Loss
B-5-06-B-13	4-L	24%	1.05g/l	0.58%
B-5-07-B-1	3-R	24	1.10	1.7
B-5-07-B-2	3-R	25.5	1.16	3.2
B-5-07-B-3	3R/4L = 2	24	--	1.0

* Total acidity before bismuth addition.

[redacted]

Higher acidity in the dissolver solutions was suspected but the above data fail to show a difference great enough to explain the increased waste loss.

Solutions obtained from the first dissolving in Dissolver 3-5R in the B Canyon and all dissolving in 3-5R in the T Canyon have given normal process performance.

Decontamination (B)

Experiments made in the Cold Semi-Works have indicated the feasibility of substituting the 24% bismuth solution for the 14% bismuth solution, thereby requiring only one bismuth solution in the Canyon Buildings. A decision was made about a month ago to evaluate this substitution in the B Canyon. However, their stock of 14% solution was such that a large number of runs would have to be made before a tank and header could be used for the 24% solution. On the basis of present inventories, the use of the more concentrated solution cannot be evaluated before the August series of runs.

Waste (T)

The first cycle waste tank (241-107) has been equipped with a dry sleeve into which an ionization chamber can be lowered. Meter readings vs depth have been obtained, July 6, which indicate that active solids have built up to a depth of about 1 foot, with less compacted slime above this for about 1.5 feet.

Samples of neutralized first and second cycle wastes as well as samples from the respective 241 Tanks have been taken for study of future waste disposal procedures. Samples (500-ml.) of supernatant liquid from Tanks 241-110 (second cycle waste) and 241-107 (first cycle waste) gave readings of 2mr/hr and 40mr/hr respectively at 4 inches with no shielding.

Alternate Jets (B and T)

Piping modification to permit the use of both the regular and alternate jets in emptying the precipitators is in progress in the B Canyon and are planned for the T Canyon.

Periscope Exposure (T)

Measurements of the radiation received by the periscope lens system have been discontinued, since the order of magnitude has been found to be low. No accurate correlation between metal properties and total periscope radiation has been found, but an approximation, good to a factor of 2, is 2.5 r/metric ton of metal charged.

Concentration Bldgs. (224)

Reduced Process Volumes

Production Test No. S.E.-221-T-PA-6 has been carried through Runs T-5-06-D-9 to D-11 and is to be extended through Runs T-5-07-B-1 to D-5. This test involves KOH metathesis of the second cycle $BiPO_4$ product cake in Section 17 and dissolution of the resultant $Bi(OH)_3$ cake in 40% of the standard volume of 60% HNO_3. The object of this test is to provide smaller process volumes for the Concentration Building. These smaller volumes permit the carrying out of the two LaF_3 product strikes at 120mg/l La each, instead of the usual 80mg/l, for the same total weight of La (2.5 lbs.).

For the three runs thus far completed, the cross-over $BiPO_4$ by-product losses

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(A-4-BP) appear to be only slightly increased, if at all, from the 60% volume process levels. The 40% volume runs average 0.36%, as compared to 0.28% at T Area and 0.33% at B Area for the 60% volume process. No change in the cake washing procedure is to be made at present.

The LaF_3 by-product waste losses have increased, however, to an average of 1.02% (corrected for recycling to a "performance" basis) from a T Area average of 0.78% and a B-Area average of 0.74% for the 60% volume process. It is believed that incomplete displacement of the more concentrated centrifuge heel in the cake washing process is the cause. Accordingly, the next five runs at T Area will contain two supplementary slurry washes of the LaF_3 by-product cake (250 lbs. 1N HNO_3 washes) in addition to the regular 500 lbs. of 1N HNO_3 precipitator-centrifuge displacement wash. In addition, since the eventual Cell D process might involve the recycling of a two extra La charges (3.75 lbs.) into a 40% volume process, the next five runs at T Area will be carried out with 7.5 lbs. of La recycled to each LaF_3 by-product to determine if the revised cake washing procedure is capable of producing and maintaining low losses.

The LaF_3 product waste losses (E-3-WS) were reduced to an average of 1.0% (corrected to a "performance" basis for recycling) from the former T Area average of approximately 1.4%. Run T-5-06-D-10 is not included in this average. In this run, the La for the entire two-strike precipitation cycle was added in the first strike (100% extra). A second strike with the usual weight of La(1.25 lbs.) was made with additional La, resulting in an overall excess of 50% extra La. The low final E-3-WS waste, 0.79%, can be treated as a preview of the losses to be expected if both the extra La and the reduced volume processes are combined.

The performance data for the 40% volume test runs are as follows:

40% Volume Waste Losses (Test Number S.E.-221-T-PA-6)

Run.No.	Remarks	% Waste Losses(corrected to"performance"basis				
		$BiPO_4$ B.P. (A-4-BP)	LaF_3 B.P. (D-4-BP)	LaF_3 Prod.(E-3-WS) Cent.#1	Cent.#2	After HNO_3 Wash
60% Vol.Ave.	1.25-lbs.La/strike	0.28	0.78	--	--	1.4
T-5-06-D-9	1.25-lbs.La/strike	0.42	0.99	6.6	(1.2)	1.0
T-5-06-D-10	1.25-lbs.La/strike #1 1.25-lbs.La/strike #2	0.33	0.92	3.3	0.73	0.79
T-5-06-D-11	1.25-lbs.La/strike	0.34	1.15	4.7	0.94	0.99

Increased La Addition

Production Test No. SE-224-B-PA-1 has been carried through Runs B-5-06-B-8 to B-13 and is to be extended through Runs B-5-07-B-1 to B-6. This test involves the addition of 50% more La to each LaF_3 product strike in the 60% volume process. This is another method of obtaining 120mg/l La per strike--obtained by adding 3.75 lbs. La total instead of 2.50 lbs. Both types of La concentration increases are designed to decrease the LaF_3 product waste losses (E-3-WS).

The results of the first four runs were reported in the last Progress Report. The six run over-all average LaF_3 product loss has remained unchanged at 1.3%, a decrease of 0.5% from the previous B Area average of 1.8% for two 80mg/l La strikes.

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Although an attempt has been made to determine if any hold-up of LaF_3 product cake in E-2 centrifuge has been caused by the use of additional La, inspection of the metathesis slurry assays (F-1-PS) and final product solution yields (P-1) has not supported any conclusions, due to the erratically high final yields obtained on these runs. The Beckman meter readings on the supposedly empty E-2 centrifuge are being carefully watched for each run and extra water flushing used if the readings are in excess of 30×10^{-14} amps.

Pertinent yield and loss data for these test runs are given below:

Data for 50% Extra La Process Runs (Test No.SE-224-B-PA-1)
% 8-1-M Product Assays

Run No.	$BiPO_4$	Prod.Sol.	$BiPO_4+LaF_3$ Losses	LaF_3 Product Losses			LaF_3 Meta. Losses	Combined Met. Loss	Final Yield
	17-4-P	C-4-0	A-4+D-4-BP	Cent.#1	Cent.#2	After Wash	F-1-PS	F-7 and F-9-WS	P-1
B-5-06-B-8	93	92	1.0	6.2	0.89	1.0	80	0.7	87
B-5-06-B-9	102	105	0.9	8.6	1.3	1.5	97	1.0	112
B-5-06-B-10	94	99	1.1	6.8	1.2	1.2	93	1.0	109
B-5-06-B-11	96	97	1.1	7.8*	1.3*	1.5*	92*	1.0	97*
B-5-06-B-12	94	99	1.0	6.3	0.98	1.1	89	1.1	88
B-5-06-B-13	100	99	1.1	6.9	1.2	1.4	110	0.9	111

* Corrected to performance basis for 9.5% recycle.

June Process Performance

The overall average Concentration Building performance for the June series of runs at both T and B Areas are summarized in the tabulation below. The two areas are identical in performance insofar as total waste losses are concerned with the T Area approximately 2% higher in both overall measured yield and material balance.

June Summaries for Concentration Bldg.Performances

Run No.	% 8-1-MR Product Assays											Bldg. 224 (% of C-4-0)			Over-all (% of 8-1-MR)			
	17-4-P	C-4-0	A-4-BP	Re-Cycle	D-4-BP	E-1-R	E-3-WS	F-1-PS	F-7-WS	F-9-WS	P-1	Yield	Loss	Mat. Bal.	Yield	221 Loss	224 Loss	Mat. Bal.
T-5-06	99	97	0.31	7.6	0.87	--	1.2	93*	0.79	0.39	98*	101	3.7	105	98	3.3	3.6	105
B-5-06	94	96	0.33	--	0.74	95	1.3	97	0.79	0.40	96	100	3.7	104	96	3.3	3.6	103

* Corrected for recycle

Special Recycle Run

Recently a special recycle run (at 40% standard volume) was carried out with approximately 31 lbs. of La in the LaF_3 by-product, in order to forward all product hold-up in the E-4 reservoir to isolation as quickly as possible. Although this run contained 50% more La in the by-product than any previous "master" recycle run, no changes in procedure were used beyond skimming the LaF_3 by-product cake (in D-2) to a 40-gal. heel instead of the usual 30-gal. heel for an eight charge recycle run (20 lbs. La).

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A LaF_3 by-product loss (D-4-BP) of 3.3% was obtained, but was not unexpected because of the large cake. A decontamination factor of approximately 12 was obtained in the LaF_3 by-product, high enough to indicate no great LaF_3 leak-through in centrifuging and washing. The Beckman meter on D-2 centrifuge indicated no hold-up of cake after removal of the LaF_3 by-product from the bowl. The LaF_3 product waste (E-3-WS) was extremely low at 0.72%. As usual, the metathesis waste losses were higher than for regular production runs, the reasons for which are still obscure.

The performance data are as follows:

Performance Data for Special Recycle Run (T-5-07-R-1)

Recycle Basis	% E-4-RC Product Assays										Final Yield
	Oxidized La Soln.	LaF_3 B.P.	Reduced Prod. Soln.	LaF_3 Prod. Wastes			LaF_3 Meta. slurry	Meta. Waste	Meta. Waste	
				Cent. #1	Cent. #2	Final				
E-4-RC	D-1-O	D-4-BP	E-1-R				F-1-FS	F-7-WS	F-9-WS	P-1
100	102	3.4	--	5.2	0.51	0.72	--	1.1	0.70	115

Isolation Building (231)

Effect of Increased La Concentration in F-10-P Solution Building

Runs B-5-06-B-8 through B-5-06-B-13 and run T-5-06-D-10 were received in the Isolation Building with about 50% more than the former "standard" weight of lanthanum. Laboratory work has indicated an increased solubility of product peroxide in the first precipitation when the lanthanum concentration is increased. This effect was masked by the variables associated with plant-scale decanting, however, as measured by the total weight of product recycled. The latter averaged 9.0 grams (exluding run B-12 on which 23.7 grams were recycled due to faulty decanting) per batch for six of the seven runs with extra La, as compared with 9.1 grams per batch for five control runs with standard La.

Acidity of F-10-P solution received from Concentration Building

The acidity of the F-10-P solutions, as measured in the first precipitation tank in the Isolation Building has averaged 1.76N for twelve runs from T Plant as against 1.86N for fourteen runs from B. Eight of the T runs required acidity adjustment whereas only five of the B runs were below the limit of 1.8N. A further change in T Plant quantity of HNO_3 seems indicated.

Acidity of First Peroxide Cake Solution

This determination (P-2 acidity) was dropped as routine in the middle of the May series of runs. To ascertain if recent increases in product content per batch caused the acidity at this point to fall below the desired 1.5N in HNO_3, samples from runs B-10 and 11 and T-9 and 10 of the June series were assayed for acidity. Values of 1.96, 1.52, 1.48, and 1.62 (order same as runs above) indicate satisfactory control.

Dummy Run (Cell 1) of oxalate method of treating wastes before recycling.

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The method worked out in the laboratory and described in last weeks report was given a preliminary trial on a synthetic waste solution (without product) in precipitator P-1 of Cell 1. All steps proceeded as expected from laboratory experience. Considerable evolution of oxygen occurred after sufficient KOH was added to give the brown $Fe(OH)_3$ coloration which indicated the end-point. This bubbling was made more severe by the addition of 150ml of 25% KOH to bring the pH from about 3 to about 6. Settling of the precipitate was not complete.

In view of the marked changes in effervescence which can result from slightly increased basicity and the attendant problems of poor settling and possibly hazardous foaming, it has been concluded that this method of treating wastes is not to be recommended for plant operation.

Additional laboratory work is planned along lines which do not involve the processing of alkaline peroxide wastes.

Cold Semi-Works

Replacement of Sodium Dichromate by Potassium Permanganate

A series of four Semi-Works runs was completed in which $Na_2Cr_2O_7$ was replaced by $KMnO_4$ as the holding oxidant in the cross-over cycle. The oxidations were made in 5N HNO_3 solution using 0.01M $NaBiO_3$ concentration and 1-hour oxidation time at 50°C. At the end of the 1-hour period the oxidized solution was made 0.01M in $KMnO_4$ and then diluted for the $BiPO_4$ by-product precipitation. The effluent from this step was made 0.01M in $KMnO_4$ prior to the LaF_3 by-product precipitation. Analytical checks made during the $BiPO_4$ by-product step indicated that the $KMnO_4$ concentration dropped from 0.002M to 0.0007M during this step.

The average $BiPO_4$ by-product step loss on three runs was 0.7% while a higher loss was obtained on the fourth run due to H_2O_2 contamination.

The removal of $BiPO_4$ by-product cake from the bowl was satisfactory without the use of supplemental H_2O_2.

The overall results showed that the replacement of $Na_2Cr_2O_7$ by $KMnO_4$ can be made satisfactorily.

Cleanout of Semi-Works Equipment

A cleanout of the Semi-Works tanks and centrifuges is in process prior to placing the equipment in stand-by condition.

Process Chemistry

Alternate Procedure for Treatment of Isolation Building (231) Peroxide Waste Solutions

In the report for last week a procedure was described for recovery of product from Isolation Building peroxide precipitation waste solutions in a comparatively small volume of solution for recycling to the Concentration Building (224). This procedure was based on precipitation of lanthanum oxalate, neutralization of the solution and dissolution of the settled precipitate in HNO_3 and destruction of the oxalate with $KMnO_4$. A critical point in the procedure was whether the rate of decomposition of H_2O_2 would be too rapid in the slightly alkaline solution to prevent satisfactory settling or to present a hazard due to spray or to foaming out of the tank. Tests made in a plant tank on 38-liter scale using simulated process solution (without product) showed that some effervescence was encountered. Supplementary laboratory tests indicated that the rate of H_2O_2 decomposition was very sensitive to pH changes near neutrality and that such close control of the end-point of the neutralization would be necessary, in order to avoid excessively rapid H_2O_2 decomposition, that the method does not appear satisfactory for plant use. Further work on precipitation from strongly alkaline peroxide solutions is, therefore, being discontinued and attention will be given to procedures based on precipitation of product carriers from acidic or buffered solutions as a means of reducing the volume of solution for recycling. Another possible line of attack involves destruction of the peroxide prior to making the solution alkaline.

Washing of $BiPO_4$ Extraction Cakes with NH_4HF_2 Solutions

Previous experiments have shown that Zr-Cb activities can be effectively removed from $BiPO_4$ extraction step precipitates by washing with a 10% NH_4HF_2 solution. This solution, however, was found to be too corrosive for use in plant equipment. Subsequent experimental work on corrosion inhibitors showed that addition of $CrO_4^{=}$ to the solution decreased the corrosion rate on 25-12 Cb steel to a tolerable level.

Additional tests have now been made to determine the effect of added chromate on the removal of Zr-Cb activities from $BiPO_4$ extraction precipitates. These washing experiments showed that the presence of chromate (10% NH_4HF_2-5% K_2CrO_4) markedly reduced the effectiveness of the wash. Only about one-fourth as much activity was removed by each NH_4HF_2-K_2CrO_4 wash as with the comparable NH_4HF_2 washes in the control run. Accordingly this method of effecting decontamination does not appear to be of further interest.

Potassium Permanganate as a Holding Oxidant in the Cross-Over

As reported previously, laboratory experiments indicated that the metathesis waste losses were increased by the presence of chromium. The eliminate of $K_2Cr_2O_7$ as a holding oxidant in the preceding oxidation step, therefore, might possibly decrease these losses. The alternate procedure using $KMnO_4$ instead of $K_2Cr_2O_7$ as a holding oxidant has been evaluated in the Cold Semi-Works. In these tests, oxidation in 5N HNO_3 was with the usual concentration of $NaBiO_3$ (0.01M) and the solution was made 0.01M in $KMnO_4$ just prior to addition of the dilution water. The results obtained indicated satisfactory performance in that $BiPO_4$ by-product losses were normal, about a third of the permanganate remained at the end of centrifugation, the by-product cakes were removed cleanly from the centrifuge bowl with the customary number of treatments and appeared normal in every respect. No further laboratory or Semi-Works tests appear to be necessary prior to plant tests.

Characterization of Activity in Certain Process Solutions from Run T-5-06-D-5

An examination of solutions D-1-O (oxidized solution prior to LaF_3 by-product precipitation in the cross-over) and P-1 (isolation process starting solution) was conducted to determine whether or not the 40-hr. La activity coming through in these solutions was "orphaned" or "Ba-supported".

Measurements of the gross activity of the D-1-O solution showed that (1) the beta ray had an initial half-thickness of 45mg/cm^2, (2) the beta activity decayed initially with about a 13-day half-life, and (3) the gamma activity decayed initially with about a 13-day half-life. These results would suggest that Ba-supported La is controlling the situation relative to all activities present (after about 7 days of decay before experiments were started).

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The gamma activity in the P-1 solution from this run decayed with about a 14-day half-life, which again indicates that Ba was the controlling factor as far as gamma radiations were concerned. The beta ray absorption curve showed the characteristics expected for 13.5-day Pr.

A comparison of the gamma counts per unit of product for the two solutions showed that the gamma activity present (presumably Ba-La) was decontaminated by a factor of about 18 by the LaF_3 by-product precipitation in the cross-over.

M. F. Acken
M.F.Acken - Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.Div.

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July 21, 1945

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200 AREAS
July 13 Through July 19

Canyon Buildings (221)

Coating Removal (B and T)

It has been decided to adopt the slow caustic addition procedure for coating removal (Production Test S.E. 221-B-PA-1) as standard for both B and T Canyons.

Extraction (B)

The high extraction waste losses reported last week appear to be due to some abnormality in the metal solution. Neither free nitric acid nor iron content was high enough to account for the increased loss;

Run No.	Dissolver Soln.	UNH Conc.	H Conc.	Fe Conc.	Waste Loss
B-5-06-B-13	4-L	24%	1.05g/1	--	0.58%
B-5-07-B-1	3-R	24	1.10	--	1.7
B-5-07-B-2	3-R	25.5	1.16	0.0029M	3.2
B-5-07-B-3	3R/4L=2	24	--	--	1.0
B-5-07-B-4	4-L	24	--	0.0025M	0.58

Decontamination (T)

The first cycle scavenger by-product (13-4-BP) waste loss on Run T-5-07-D-3 was abnormally high, 17.1%, for no known reason. The bismuthate and dichromate used in the preceding oxidation step were checked and found to be within specification; also, the same materials were used simultaneously for the oxidation step in Section 14 on Run B-2, with satisfactory results. All chemicals entering the Section 13 Precipitator were checked for the presence of reducing agents, which were found to be absent. Oxidations on the subsequent runs, D-4 and D-5 were normal (approximately 99%).

The high scavenger by-product on Run D-3 was re-worked by the procedure used for reworking the same waste on Run T-5-04-B-3, but without comparable results. The product was solubilized and separated from the cerium and zirconium satisfactorily, by using ammonium silicofluoride, but difficulty was encountered in the subsequent oxidation. The by-product (no scavengers) from the re-work (Run D-3-A) of the

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original scavenger by-product (Run D-3) contained about 50% of the charge and was re-worked as Run D-3-B. Oxidation was again incomplete, approximately 65%, but the by-product was discarded (2.7% of the original D-3 charge).

The cause of this oxidation difficulty is not known and is being investigated. One possibility which is being investigated is the presence of a silicofluoride salt which behaves similarly to bismuth phosphate, precipitates at low acidity and dissolves at high acidity, and could therefore, inhibit the oxidation. A summary of the pertinent data is presented in the following table.

	Run #(T-5-07-)				
	D-3	D-3A	D-3B	D-4	D-5
Oxidation in	12-7	13-4	13-4	12-7	12-7
Acidity (theoretical), g/1	8.0	7.5	7.2	7.7	--
Acidity (by analysis)	--	7.9	7.4	7.0	7.9
Appearance before oxidation	Clear(8-4)	1.0g/1	1.0g/1	Clear	Clear(8-4)
Prod. on insoluble residue-performance basis - %	--	7	5	--	--
% Unoxidized, performance	(17-?)	53	36	0.8	1.3
1. In soln., performance	--	--	25	0.8	1.3
2. On insoluble residue, performance	--	--	.25	--	--
By-product waste, performance	17.1	53	30	1.4	2.4
$NaBiO_3$	←Same Bbl.→				
$Na_2Cr_2O_7$	←Same Solutions→				
Time, Temperature, and Agitation	←Standard→				

Runs D-3A and D-3B were combined by metathesizing each in the first cycle product centrifuge and dissolving in half of the usual amount of acid. The acid was added directly to the centrifuge, no attempt being made to clean the precipitator. Run D-4 is also being metathesized so that the dissolving acid, diluted in the precipitator until it reaches the agitator thereby dissolving more completely the heel from runs D-3A and D-3B, will dissolve the cake without difficulty.

Wastes (B and T)

The temperature of the metal waste continues to increase more rapidly than that of the other tanks. A summary of data is presented below:

Tank Temperature (F°)

Area	Date	101 Metal Waste	104 Empty	107 1st Cycle	110 2nd Cycle
T	4/18	77	61	80	80
T	5/21	93	62	83	83
T	6/19	108	67	90	88
T	7/17	118	70	94	88
B	4/23	62	51	57	60
B	5/15	75	52	68	58
B	6/19	97	55	75	71
B	7/17	110	57	80	78

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Reserve Capacity

Tanks	Type Waste	Gallons per run*	B Area Gallons	B Area Runs	T Area Gallons	T Area Runs
X-101- 2, -3	Metal	5900	1,357,000	230	1,200,000	203
X-107- 8, -9	Ctng.& 1st Cycle	4800	1,370,000	285	1,235,000	257
X-110- 11,-12	2nd Cycle	3600	1,420,000	395	1,265,000	351

* Based on 1.5 tons per run, as per procedure of 6/15/45

Alternate Jets (B and T)

Piping modifications to permit the use of both the regular and alternate jets in emptying the precipitators has been completed in the B Canyon and work is being started in the T Canyon. It is planned to incorporate this change into the procedures, beginning with the next runs in each area.

Concentration Buildings (224)

Production Tests

Two production tests are being continued. Both have the same objective: to decrease the LaF_3 product waste losses by increasing the lanthanum concentration of the product strike. SE-221-T-PA-6 in the T Area involves a 40% standard volume process with the **reduced** volume obtained by KOH metathesis of the second cycle $BiPO_4$ product cake in Section 17. SE-224-B-PA-1 in the B Area involves the addition of 50% extra La to each LaF_3 product strike. Both processes result in 120 mg/l La per strike.

Reduced Volume Runs

The 40% volume runs are being continued through runs T-5-07-B-1 to D-5. In addition, 7.5 lbs. instead of 5 lbs. of lanthanum are being recycled to each LaF_3 by-product as this additional La might be involved in the eventual Cell D process. In the previous 40% volume runs higher than normal LaF_3 by-product losses (1.02% vs. 0.78%) had resulted presumably because of the more concentrated centrifuge heel. To reduce this loss, Runs T-5-07-B-1 and B-2 were made with two slurry washes of the LaF_3 by-product cake (250 lbs. of 1N HNO_3 each) in addition to the regular 500 lbs. 1N HNO_3 precipitator-centrifuge displacement wash. These slurry washes were added directly to the centrifuge bowl from the operating gallery. The by-product waste loss (D-4-BP) was reduced to 0.55%. The LaF_3 product waste loss, however, increased from 1% to 1.9% probably because the very low HF content of the by-product washes resulted in LaF_3 being carried over into the effluent, which is then in a form difficult to separate during the LaF_3 product centrifugation. In Run T-5-07-D-3 the displacement wash and the two bowl slurry washes were replaced by a 500-lb. 1N HNO_3 precipitator - two 250-lb. centrifuge slurry washes. The resulting waste losses were 0.72% and 1.1% respectively for the by-product (D-4-BP) and product wastes (E-3-WS). This process will be continued for Runs T-5-07-D-4 and D-5.

The performance data for the current series of runs are as follows:

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40% Volume Waste Losses (Test No. SE-221-T-PA-6)

Run No.	Wash Procedure for LaF_3 By-Product Cake	% Waste Losses (Corrected to perfomance Basis)				
		$BiPO_4$ B.P. A-4-BP	LaF_3 B.P. D-4-BP	LaF_3 Prod. Waste (E-3-WS) Cent. #1	Cent. #2 + Wash	Cent. #3
T-5-07-B-1	1 displacement wash plus 2 slurry washes added directly to bowl.	0.34	0.50	9.0	1.5	--
B-2	1 displacement wash plus 2 slurry washes added directly to bowl.	0.37	0.51	9.6	1.9	1.0
D-3	2 slurry washes added via the precipitator.	0.38	0.67	4.5	1.0	
60% Vol. Ave.	Precipitator-centrifuge displacement wash.	0.28	0.78	--	1.4	--

Increased La Addition

Production Test No. SE-224-B-PA-1 has been continued through runs B-5-07-B-1 to B-5.

The results of the initial six runs were reported in the last progress report. The waste losses of the current 5 runs continue to be low and are given in the following tabulation.

50% Extra La Process Waste Losses (Test No. SE-224-B-PA-1)

Run No.	$BiPO_4$ BP A-4-BP	LaF_3 BP D-4-BP	LaF_3 Product Wastes (E-3-WS) Cent. #1	Cent. #2 + wash
B-5-07-B-1	0.31	0.83	6.1	1.2
B-5-07-B-2	0.29	0.64	6.2	1.1
B-5-07-B-3	0.38	0.65	6.1	1.2
B-5-07-B-4	0.27	0.68	6.8	1.1
B-5-07-B-5	0.41	0.82	7.6	1.4

The LaF_3 product centrifuge (E-2) Beckman readings have been followed closely during the removal of the 150% La product cakes. During initial runs the standard E-2 cake removal procedure did not always clean the bowl completely. The cake removal procedure has been modified to include a second slurry wash of 200 lbs. of water. This still allows the metathesis to be carried out at normal volume. The data are obscured by the fact that the processing of the first large LaF_3 product cake coincided with the change in the Beckman tube location to increase its sensitivity; therefore, no background for the empty bowl had been established. To aid in determining the E-2 background and as an added precaution the extended cake removal procedure has been followed by additional washings. The extra washes necessitate increasing the metathesis charge weight from 2400 lbs. to 2700 lbs.

A summary of pertinent yield figures and E-2 full and empty Beckman readings is as follows:

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Yield Figures and E-2 Beckman Readings

Run No.	17-4-P	E-1-R	E-2 Beckman		F-1-PS	P-1
			Full	Empty		
B-5-06-B-8	93	91	150	25	80	87
B-5-06-B-9	105	--	125	24	97	112
B-5-06-B-10	94	94	155	28	93	109
B-5-06-B-11	96	105 (1)	81	18	102 (1)	107 (1)
B-5-06-B-12	94	96	77	20	89	88
B-5-06-B-13	100	99	77	20	110	111
B-5-07-B-1	95	96	79	4	123	112 (2)
B-5-07-B-2	91	91	48	7	89	92 (3)
B-5-07-B-3	95	98	84	17	91	93
B-5-07-B-4	98	93	81	22	89	--
B-5-07-B-5	103	102	--	21	--	--

Notes: (1) uncorrected for 9.5% recycle
(2) Uncorrected for 3% 8-1-MR product left behind in F-10
(3) Includes 3% 8-1-MR product from Run B-1 left behind in F-10

The details of the E-2 cake removal with 50% extra lanthanum are given in the following tabulation:

Beckman Readings for E-2 Product Cake Removal (150% LaF_3 Cake)

Run No.	E-2 Empty Before Cent. (x10^{-14})	E-2 Full (x10^{-14})	Standard Cake Removal Procedure	E-2 Empty (x10^{-14})	Extended Cake Removal Procedure	E-2 Empty (x10^{-14})	Additional Cake Removal	E-2 (x10^{-14})
B-5-06 B-8	4*	150	700 lbs.water flush 250 lbs.water slurry 150 lbs.water flush	25	--	--	--	--
B-9	--	125	Repeated	40	--	--	250 lb. slurry 150 lb.flush	--
B-10	--	155	Repeated	33	--	--	250 lb.slurry	28
B-11	--	81	--	--	700 lb.water flush 250 lb.water slurry 200 lb.water slurry 150 lb.water flush	18	--	--
B-12	19	77	--	--	Yes	20	--	--
B-13	18	77	--	--	Yes	20	--	--
B-5-07								
B-1	12	79	--	--	Yes	4	--	--
B-2	4	48	--	--	Yes	7	--	--
B-3	7	84	--	--	Yes	17	200 lb.slurry 150 lb.flush	16
B-4	16	81	--	--	Yes	22	200 lb.slurry 150 lb.flush	19
B-5			--	--	Yes	21	as above	18

* After acid washes

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Isolation Building (231)

Effect of Increased La Concentration in F-10-P Solution

Runs from B Concentration Building continue to contain 50% more lanthanum than the former standard amount (and that currently used at T). No process difficulties in the Isolation Building have been encountered. Possible reasons for the recent high assays of the solution (P-1) are being investigated. At the present writing, it cannot be said that the high assays (P-1) are associated with the increase in La concentration in Concentration Building product solutions. Also, the 231 Building cell variable is being removed from consideration by running some B Plant runs in Cell 4, and T runs in 3 instead of all B runs in 3, and T runs in 4 as formerly.

Concentration of Final Product Solutions

The nominal concentration of final product (AT) solutions has been increased from 225g/l to 250g/l. This change, made July 7, is intended to reduce the frequency of intermediate evaporations necessitated by AT concentrations below 200g/l. This is especially desirable since intermediate evaporation in the new Cell 6A equipment requires the subsequent handling of a potentially contaminated adapter plug when the can is reloaded in a process hood.

Volume of PR-3 Tank

The means used to control the volume of solution obtained after evaporation in the Still (S-1) is to charge a known volume and measure the volume of distillate collected in Receiver R-1. The vacuum receiver, PR-3, therefore, is fitted with an overflow pipe which leaves a calibrated volume (about 9 liters) to be charged to S-1. Due to the increase in heel left in P-2 (after decantation) as product content per batch has increased, the PR-3 capacity has been exceeded on several occasions and some rich solution overflowed to ST-2 tank from which it had to be recovered. Several minor changes have been made to avoid this. Since one run during the past week had such a large P-2 heel that almost a liter of solution overflowed PR-3, it has been decided to use 95% HNO_3 instead of 60% for dissolving the P-2 cake. This will save about a liter of volume and, along with increased operational emphasis on decanting to the lowest P-2 heel possible, may avoid the PR-3 volume limitation in future runs. This change was made with Runs T-5-07-D-3 and B-5-07-B-3.

Cleanout No. 18 (Cell 3)

A cleanout of Cell 3 was made in an effort to locate about 200 "missing" units (based on P-1 assays). Only about 20 units were obtained. This also throws doubt on the high P-1 assays mentioned previously.

Treatment of Peroxide Waste Solutions

As reported previously, procedures for reducing the volume of solution to be recycled from the Isolation to the Concentration Building, based on precipitation of a carrier from alkaline solutions containing H_2O_2, were unsatisfactory due to rapid decomposition of H_2O_2 in the alkaline solutions. This difficulty has been overcome by first destroying the H_2O_2 with $NaNO_3$ prior to complete neutralization. The present revised procedure, by which satisfactory recovery of product has been obtained on a laboratory scale with an indicated recycle volume per batch (plant scale) of less than 40 liters, involves the following steps. A prescribed volume of KOH is first added to the waste solution to lower the acidity to about 0.5N, the H_2O_2 is then destroyed by addition of a slight excess of 20% sodium nitrate solution, oxalic acid is then added to precipitate lanthanum oxalate, after which the solution is made alkaline with KOH. After agitation for 15 minutes the precipitate is allowed to settle, the supernatant decanted, the precipitate washed and again settled and finally dissolved in HNO_3 and the oxalate destroyed with $KMnO_4$.

Cold Semi-Works (321)

The acid cleanout of all cell equipment, including tanks, centrifuges, dissolver, samplers, and jet transfer lines was completed. The tanks (except A-8, the neutralizer for miscellaneous active wastes), centrifuge samplers and cell floors were monitored and found to be satisfactory.

All the weigh tanks except the one supplying NaOH to A-7 and A-8 Tanks were cleaned together with the lines leading from the weigh tanks to the cell equipment.

The make-up tanks and the solution supply systems except 50% NaOH were cleaned and flushed with water.

The instruments except those affecting A-7 and A-8 Tank operation are being placed in stand-by condition.

Process Chemistry

Disposal of Decontamination Cycle Wastes

A laboratory study has been initiated to determine whether sufficient activity is removed with solids which settle out so that the supernatant solutions could be transferred to dry wells after a short aging period. If this is possible, it would allow the tank storage space assigned to these wastes to be used for those from the extraction and first cycle, thereby appreciably increasing the waste storage facilities.

For the initial appraisal of this problem samples of solutions were obtained from the first and second decontamination cycle waste neutralizing tanks (15-8 and 15-9) and from the corresponding waste storage tanks (107 and 110). Gross β and γ activity and product content were determined on the supernatants after gravity settling (over night) and after centrifugation. Data are given below:

Source of Solution	β Counts/Min./cc (1)		γ Counts/Min./cc (2)		Micrograms of Product per liter
	Gravity Settled	Centrifuged	Gravity Settled	Centrifuged	
15-8 (3) (First cycle waste neut. tank.	6.3×10^6	6.8×10^6	8100	9000	
15-9 (Second cycle waste neut. tank.	5.3×10^4	5.6×10^4	31	32	
107 -First Cycle storage tank.	2.3×10^6	2.9×10^6	11,000	11,500	39
110 -Second Cycle storage tank.	4.9×10^4	4.9×10^4	30	26	15

Notes on following page:

(1) Second shelf, minimum absorber
(2) Second shelf, standard counting conditions
(3) Because of sampling difficulties, this sample is not representative. Also, the amounts of sample from the by-product and product steps were such that there was about three times as much sample from the product step.

Solids settled out of the solutions rapidly and the above data show that the amount of activity remaining in solution after gravity settling was essentially the same as remained after centrifugation. Further tests are to be made to identify the major activities present in the solutions.

The amount of activity that can be run into dry wells has not been defined as yet so no definite conclusion can be drawn as to feasibility of the procedure. The amount of product remaining in the supernatant solutions was surprisingly high.

M. F. Acken
M.F.Acken, Acting Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.Dept.

MFA:jd

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INV. 9-47

July 28, 1945

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200 AREAS

July 20 Through July 26

Canyon Building (221)

Coating Removal (T)

The coatings have been successfully removed from two 3-ton batches of slugs by the newly adopted procedure involving the slow addition of caustic to the boiling solution.

Decontamination Cycles (T)

The metathesis of bismuth phosphate to bismuth hydroxide by the use of 35% KOH (Production Test SE-221-T-PA-6) has been completed on twelve product cakes; three first cycle cakes (in 14-2) and nine second cycle cakes (in 17-2). Mechanically, the operations were feasible. However, this method added at least 2 hours to the cycle time and occasionally caused the centrifuge motor to kick out during the metathesis, metathesis washing, or cake dissolving steps. Chemically, the metathesis was sufficiently complete to result in complete cake solution but caused an increase of about 0.4% product in the product wastes:

Effect of Metathesis on Product Wastes

Product Cake	Without Metathesis		With Metathesis		
	No. of runs	Ave. Waste	No. of runs	Ave. Waste	Increase
First Cycle (14-2)	10	0.37%	3	0.79%	0.42%
Second Cycle(17-2)	10	0.34%	9	0.71%	0.37%

On the basis of these increased waste losses and the failure to obtain greater reduction in the Concentration Building losses, the metathesis procedure has not been adopted as standard but will be maintained as a procedure for use on special occasions when it may be necessary or desirable to dissolve a cake in less than the standard quantity of acid.

Centrifuging the metathesis slurry and subsequent washings an additional 5 minutes before skimming failed to reduce the loss.

Concentration Buildings (224)

40% Volume Runs

Test SE-221-T-PA-6 is being discontinued. This test involved a 40% standard volume process with the reduced volume obtained by KOH metathesis of the second cycle $BiPO_4$ product cake in Section 17. The concentration process waste losses for these 40% volume runs are given in the tabulation below. The results indicate that even with a modified washing procedure for the LaF_3 by-product cake, the 40% volume process results in little change in total product waste losses for the concentration process. There are, however, some attendant reductions in Concentration Building time cycles due to decreased centrifuging time. The 40% volume process is being discontinued in favor of adding 50% additional lanthanum in the product strikes to decrease Cell E waste losses due chiefly to the mechanical difficulties and higher waste losses encountered in the Canyon Building.

40% Standard Volume Process vs. 60% Standard Volume Process
(All wastes on a performance basis corrected for recycle)

Runs	$BiPO_4$ BP A-4 BP	LaF_3 BP D-4 BP	LaF_3 Product E-3WS Cent.#1	LaF_3 Product E-3WS Cent.#2	Remarks D-2 Washing
Ave.60% Vol.Runs	0.28	0.78	--	1.4	displacement
40% Vol. Runs					
T-5-06-D-9	0.42	0.99	6.57	1.1	displacement
T-5-06-D-10	0.33	0.92	3.3*	0.8	displacement
T-5-06-D-11	0.34	1.15	4.7	0.99	displacement
T-5-07-B-1	0.34	0.50	9.0	1.5	displacement + bowl
T-5-07-B-2	0.37	0.51	9.6	1.9**	slurry
T-5-07-D-3	0.44	0.84	5.5	1.2	precipitator
T-5-07-3A	0.47	0.87	6.1	4.2***	centrifuge slurry
T-5-07-4	0.33	0.62	6.3	1.3	centrifuge slurry
T-5-07-5	0.33	0.81	5.9	1.2	centrifuge slurry

* All La added in first strike - 50% additional used for second strike.
** Recentrifuged to give 1%
*** Low level kitten runs

The details of the washing procedures for the LaF_3 by-product cake in conjunction with the 40% standard volume process were discussed in the last weekly progress report. The table following contains a summary of the waste losses as affected by modifications in the washing procedure. All losses are calculated to a performance basis for recycling.

Waste Losses vs. LaF_3 By-Product Washing Procedure

Runs	D-4 BP LaF_3BP	E-3-WS LaF3 Product Waste	1N HNO_3 Washing Procedure: 500-lb. Precipitator Centrifuge Displacement wash.	Two 250-lb. bowl slurry washes	500-lb. precipitator-two 250 lb. centrifuge slurry washes.
Ave.of 60%Vol. Runs	0.78	1.4	Yes	No	No
40% Vol.Runs					
T-5-06-D-9,10,11	1.02	1.1	Yes	No	No
T-5-07-B-1, 2	0.50	1.7	Yes	Yes	No
T-5-07-B-3, 4, 5	0.76	1.2	No	No	Yes

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To evaluate the effect of the precipitator-centrifuge slurry wash for the 60% volume process and because of the increased La in the LaF_3 by-product due to recycle, the TA will continue to use the slurry washing procedure during the 4 remaining runs in the July series.

150% La Runs

The procedure used in test SE-224-B-PA-1 is being adopted as the standard process for both B and T Areas. This involved the addition of 50% extra La to each LaF_3 product strike. The following table records the average product waste losses for this process as compared to the 60% standard volume process.

Waste Losses

150% La vs. 100% La in 60% Standard Volume Process

Runs	$BiPO_4$ BP A-4 BP	LaF_3 BP D-4 BP	LaF_3 PW E-3-WS (two centrifugations	Metathesis Waste F-7WS	Wash Waste F-9-WS	Remarks
B-5-05-B-3 to B-5-06-B-7	0.34	0.62	1.9	0.83	0.41	100% La
B-5-06-B-8 to B-5-07-B-5	0.30	0.75	1.2	0.70	0.27	150% La

An acid wash has been run in Cell E (LaF_3 product cell) B Area which has been useful in obtaining more information concerning product holdup in the Cell E centrifuge (E-2) after cake removal. The analytical results showed a pickup in E-2 of only 7.7% which can be compared with a 5.7% pickup during a previous acid wash made after B-5-06-D-7. Unfortunately the Cell E Beckman required maintenance work during the test which makes Beckman readings questionable. As near as can be determined, the E-2 Beckman reading went from 15×10^{-14} to 7×10^{-14} amperes during the course of this wash.

To obtain additional evidence as to the effect on the Beckman reading of a partial charge in E-2, jetting will be interrupted after 10% of the next charge has been transferred to E-2. Beckman readings will be made after skimming and after slurrying this small cake into the bottom of the bowl.

Isolation Building (231)

Increased La Concentration in F-10-P Solution Received from Concentration (224-B) Building

Runs from B Plant with the 50% extra La previously discussed have continued to perform satisfactorily in the Isolation (231) Building.

Concentration of AT Solutions

Since increasing the objective from 225 to 250g./l. for the AT final product adjustment tank concentration, sixteen runs have been process without obtaining a concentration below 200g./l. This has meant that no sample can required an intermediate evaporation with the attendant hazard of removing a potentially contaminated adapter plug assembly for the final loading after evaporation.

Dummy Runs (Cell 1) of Oxalate Method of Treating Wastes before Recycling

A modification of the method referred to in last weeks report shows considerable

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promise. The method consists of adding KOH to make the waste about 0.5N in HNO_3, then destroying H_2O_2 with $NaNO_2$ as in present plant practice (using clarification of solution as end-point), precipitating La with oxalic acid, and then adding KOH to definite alkalinity. This technique gave good settling and yields in the laboratory and good settling in the first dummy run trial in Cell 1. Using 25% KOH and 20% $NaNO_2$, the total volume of treated waste was 58 liters on a scale of 0.3 full plant volume. This would be 193 liters on full scale which is too high for CT-1 capacity. The second dummy run is now in progress with 50% KOH and 40% $NaNO_2$ being used. A final volume of about 160 liters is hoped for.

A further change is being made in the present run to reduce the volume of the solution obtained by dissolving the settled lanthanum oxalate in HNO_3 and destroying oxalic with $KMnO_4$. In order that this volume will always be contained in one can, solid $KMnO_4$ (instead of 4% solution) will be added equivalent to 80% of the oxalate present and the last 20% titrated with 4% $KMnO_4$ to a MnO_2 end-point. If this run proceeds as expected, one further dummy run will be made followed by a production test to try the method on a waste from a regular run.

Process Chemistry

Recovery of Product from First Cycle By-Product Precipitate

In Run T-5-07-B-3 approximately 17% of the product was lost in the scavenger by-product precipitate of the first cycle. In reworking of this precipitate to recover product a procedure was used comprising the usual dissolution in HNO_3 (with added H_2O_2) followed by addition of SiF_6^{--} to solubilize $ZrPO_4$ and the precipitation of a $BiPO_4$ product precipitate which was dissolved in HNO_3 and then carried through a normal second decontamination cycle. In this cycle a high loss occurred in the by-product precipitate and agin in the same step in a rework of this by-product precipitate. As a result of this plant experience, a laboratory investigation was made to determine the cause of the difficulty.

The scavenger by-product precipitate contains at least 1.5 times the amount of bismuth used in the product precipitation steps. The laboratory tests using simulated plant solutions showed that the Bi-Zr-Ce phosphate by-product precipitate dissolved satisfactorily in the amount of HNO_3 and SiF_6^{--} used in the plant. The $BiPO_4$ product precipitate from this solution, however, did not dissolve completely in the flow-sheet quantity of nitric acid, but complete dissolution was obtained when 1.5 times the flow-sheet volume of acid was used. Satisfactorily complete oxidation of product in such solutions was obtained and losses in the subsequent $BiPO_4$ by-product precipitates were low.

Accordingly, it is recommended, should it be necessary to make other recovery runs, that the product precipitate be metathesized so that it can be dissolved satisfactorily in 2600 lbs. of 60% nitric acid.

No satisfactory explanation can be offered for the initial 17% loss encountered in the first by-product of the plant run.

Disposal of Decontamination Cycle Wastes

As a preliminary appraisal of the possibility of saving of waste storage space by dry well disposal of supernatants from second decontamination cycle wastes after gravity settling to remove solids, data were presented last week on product content and gross gamma and beta activity in samples of solutions from first and second

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cycle waste storage tanks. The data obtained applied to solutions from which solids had been removed by gravity settling and by centrifugation. Further analyses of such supernatant solutions have been made to determine the elements accounting for the observed beta and gamma activity. Data are given below.

Source of Solution	% of total beta activity due to		% of total gamma activity due to	
	Rare Earths	Ba or Sr	Rare Earths	Columbium
107 (First cycle waste storage tank)	45	15	3.6	43
110 (Second cycle waste storage tank)	78	0.5	22.0	33

Fluomolybdate as a Substitute for Fluosilicate

Previously reported data have shown that better decontamination is obtained in the $BiPO_4$ product precipitation steps by the use of fluomolybdate in place of fluosilicate. Further experimental runs have confirmed this finding. These runs were made starting with "dead" UNH solution to which was added inactive isotopes of fission element, Zr-Cb tracer, and product. In the product precipitation step with fluomolybdate the gamma decontamination factor was about six times that in the same step of the control run with fluosilicate.

Fission Product Studies

Continued studies of fission products separated from 30 g./ton, 60g./ton and 130g./ton plant dissolver solutions have shown that the controversial long-lived antimony activity does exist and that it is a nuclear isomer of a stable isotope Sb^{121} or Sb^{123}. From decay measurements over a relatively short period, the half life value is estimated to be 400 to 600 days (previously estimated values, 250-400 days). Radiations associated with the activity include an approximately 0.6 Mev. gamma ray, a 26 Kev. X-ray, and 0.5^{+} Mev. electrons. From the observed relative intensities of these components from aluminum absorption curves and from critical x-ray absorption measurements with Ag, Cd and In absorbers, it is postulated that the gamma ray, emitted during isomeric transition, is partly internally converted, giving rise to monoenergetic electrons and the characteristics K. x-rays of antimony.

A number of fission products are now being isolated from plant dissolver solutions to be used for mass spectrograph studies and for calibration relative to use of the instrument in analyzing fission product mixtures. For this work a total of about 3×10^9 d/min. of Ce^{144} has been isolated with about 1 mg. of carrier. Also, about 8×10^8 d/min. of Ru^{106} has been concentrated in a small volume and is ready for final isolation. Approximately 200,000 c/m of 2-3 year Eu activity have been isolated for further study of methods for obtaining larger quantities of this activity.

M. F. Acken
M.F.Acken, Acting Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.Dept.

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August 4, 1945

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200 AREAS
July 27 Through August 2

Canyon Buildings (221)

Extraction Step (B)

During two recent runs (07-B-11 and 12) in the B Canyon, the extraction strike was inadvertently made over 2 hours instead of 3, resulting in slightly higher waste losses (1.1% and 1.5%). During Run B-12, the reduced solution was also held for approximately 6 hours at 85°C, due to adjustment in batch size; however, no oxidized product was found in the extraction waste.

Due to analytical difficulty on Run B-11, a fictitiously high product content was indicated which led to a decision to split the charge. This was done by withholding part of the slurry in the precipitator until the major portion of the charge was out of the centrifuge, then centrifuging the "heel" and holding it in the centrifuge for combination with Run B-12.

Decontamination Cycles (B)

The 14% bismuth solution normally used for product precipitation in the decontamination cycles was replaced, on an equal bismuth basis, with the 24% solution used in the extraction step on Run B-5-07-B-8. No adverse effect was noted in either waste losses or ease of cake removal from the centrifuge. Further runs are planned in B Canyon during August to demonstrate the use of the 24% solution.

Transfer Jet, 4-7 to 8-1 (T)

In order to obtain closer control of the weight of metal solution transferred from storage to the extraction precipitator, the 75 gal./min. jet has been replaced by a nominal 20 gal./min. jet.

Use of Alternate Jets for Emptying Precipitators (B and T)

On the basis of 29 measurements in the B Canyon and 11 measurements in the T Canyon, the normal jetting procedure empties the precipitator in the majority of cases. However, in a few cases a heel is left which is picked up by the alternate jet: in the B Canyon, one 200 lb. heel, two 80 lb. heels, and the remainder below

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60 lbs; in the T Canyon, one 110 lb. heel, one 80 lb. heel, and the remainder below 60 lbs.

Concentration Buildings (224)

Spare 26-inch Centrifuge

The spare 26-inch centrifuge (F-22) has been installed at both T and B Areas. A new dip tube and jet line from precipitator F-1, separate drain line to catch tank F-9, return jet line to precipitator F-1, and jet line to product receiver tank F-10 have been installed. The tachometer, overload relay, and main power line have been equipped with double-throw switches and the F-2 manometer connected to F-22. Installation at U Area is being started.

Cell B Changes

The tie-in of Cell B as a spare fluoride cycle cell has been completed at both T and B Areas. The new line installations include (a) an HF sparger in precipitator D-1 (1-inch 25-12 dip tube to within 9 inches of tank bottom); (b) a B-3 to E-1 line, to provide for use of Cell B for the LaF_3 by-product; (c) a D-3 to B-1 line; and (d) a B-2 to F-1 line, both to provide for the use of Cell B for the LaF_3 product precipitation. Installation at U Area is being started.

Recycling

Since both the T and B Area runs now utilize 3.75 lbs. of lanthanum in the LaF_3 product strikes, recycling of the isolation supernatants to the T Area alone results in 7.5 lbs. of lanthanum being precipitated in the LaF_3 by-product step, if a charge per day is run in each area. Occasionally the product content of production runs approaching the LaF_3 by-product step may be too high to permit additional product, however. To provide against an accumulative build-up of recycle solution in the E-4 reservoir, resulting from infrequent omission of recycling, 8.5 lbs. of lanthanum will be recycled into each production run at T Area, beginning with Run T-5-07-D-5.

In addition, variable amounts of lanthanum will be returned from Isolation if the new recovery methods are adopted in the Isolation Building (231). It will be necessary to add lanthanum to all Isolation acid wash recovery runs, in order to precipitate product-carrying lanthanum oxalate. This makes it more desirable to have available on short notice a lanthanum assay of the solution in the E-4 reservoir, rather than rely on the present method of estimation by counting recycled charges. A rapid method for lanthanum determination is to be furnished to accomplish this.

LaF_3 Cake Washing

The LaF_3 by-product cakes at T Area (8.5 lbs. La) will continue to be washed by the two-batch precipitator-centrifuge slurry wash with 1N HNO_3, in an effort to maintain low by-product waste (D-4-BP) losses. The single batch precipitator-centrifuge displacement wash (5.5 lbs. La) will be continued at B Area. If the slurry wash at T Area does not cause any noticeable increase in the LaF_3 product waste (E-3-WS) losses because of LaF_3 fines carry-over from the by-product, it is to be tried in test at B Area.

Cell E Acid Wash

The acid wash, carried out in the LaF_3 product precipitation cell (Cell E)

[redacted]

which was designed to measure product hold-up in the centrifuge (E-2) has been completed. This run was carried out by slurrying 5000 lbs. of approximately 4N HNO_3 through the centrifuge (E-2 to the solution tank (E-4). The E-2 Beckman activity reading was reduced from 15×10^{-14} to 8×10^{-14} amps. and 7.7% of product in a normal run was picked up. The acid wash solution was returned to the precipitator (E-1), diluted to 1N HNO_3, a single 2.5 lb. La strike carried out, the LaF_3 product cake carried through a regular metathesis, and the final product solution (F-10-P) was returned to E-4 for recycling.

The product balance and pick-up are as follows:

Cell E Acid Wash (Run B-5-07-E-AW)

Process Solution		Cumulative % Prod.	%Prod. Pick-up
Initial E-4 heel		0.9	--
Acid wash in E-4 after E-2 flush		8.6	+7.7
LaF_3 product waste	(E-3-WS)	0.7	-0.7
LaF_3 product slurry in F-1	(F-1-PS)	14.3	+6.4
15% KOH metathesis waste	(F-7-WS)	0.12	-0.1
2% KOH metathesis waste	(F-9-WS)	0.04	-0.04
Final product solution	(E-4-RC)	17.8	+3.6

150 % Lanthanum Runs

Continuation of the use of 50% extra lanthanum in the LaF_3 product strikes at B Area has maintained low waste (E-3-WS) losses, averaging 1.2%. Recent data are given below:

LaF_3 Product Waste Losses-150% La Runs

Sample	B-5-07-B-6	B-7	B-8	B-9	B-10
After Cent. #1	6.0	6.9	6.1	6.5	7.3
After Cent. #2 (final)	1.15	1.07	1.23	1.3	1.2

Removal of the extra-weight LaF_3 product cakes from the centrifuge, E-2, is suspected of being incomplete, as indicated by either LaF_3 product slurry assays (F-1-PS) or the E-2 Beckman readings. Progress of the development of modified cake removal procedures has been traced in recent reports. During the recent series of runs, product assays have failed to indicate any increase of product hold-up in the centrifuge. Increasing empty bowl Beckman readings have indicated otherwise, however. Another cake removal procedure has been tried on one run thus far with appreciable success, and is being continued. In outline this procedure is as follows:

1) Add 700 lbs. of water to E-2 via sprays, jetting continuously to F-1.
2) Add 250 lbs. of water to the centrifuge (E-2) and slurry from 0 to 110 RPM 4 times.
3) Rotate E-2 at 10 RPM for 15 minutes.
4) Slurry to 110 RPM and jet to the metathesis tank (F-1).
5) Add 200 lbs. of water, slurry from 0 to 110 RPM 5 times, and jet to F-1.
6) Add 150 lbs. of water via sprays, jetting continuously to F-1.

The test data and product assays are given in the following tabulations:

LaF$_3$ Product Cake Removal from E-2

Run No.	E-2 Beckman (x10^{-14} amp.) Start(MT)	Full	End(MT) (1)	(2)	Cake Removal Procedure
B-5-07-B-6	8	140	15		Standard: (1) 700 lb. spray (2) 250 lbs.- 3 slurryings, (3) 200 lbs.- 3 slurryings, (4) 150 lb. spray.
B-5-07-B-7	15	95	20		
B-5-07-B-8	18	110	26	26	(1) Standard
B-5-07-B-9	22	110	34	35	(2) Extra 200 lbs. - 3 slurryings plus 150 lb. spray.
B-5-07-B-10	26	74	4.5		Method outlined in text above

Product Balances-150% La Runs

Run No.	% Product 17-4-P	E-1-R	F-1-PS	P-I	% Product Losses A-4-BP	D-4-BP	E-3-WS	F-7-WS	F-9-WS
B-5-07-B-6	99.5	97.5	97.5	97	0.31	0.42	1.15	0.92	0.22
B-5-07-B-7	98.5	95.5	91.2	93	0.31	0.78	1.07	0.66	0.20
B-5-07-B-8	97.2	97.2	100	96	0.30	0.82	1.23	0.86	0.26
B-5-07-B-9	95.7	93.8	89.0	103	0.26	0.68	1.3	0.91	0.22
B-5-07-B-10	95.3	93.9	99.5	--	0.21	0.70	1.2	--	--

The reduction of the E-2 Beckman reading in Run B-10 by the use of the modified cake removal procedure appears to be substantiated by the high F-1-PS product assay obtained.

A semi-quantitative check on the correlation of the E-2 Beckman readings with product hold-up has been obtained by jetting 10% of the LaF$_3$ product precipitation slurry from E-1 to E-2, skimming the bowl, and measuring the Beckman reading. A reading of 23x10^{-14} amp. was obtained with the cake on the bowl wall and 18x10^{-14} amp. when slurried to the bottom of the bowl. The initial empty bowl reading was 8x10^{-14}.

Isolation Building (231)

Oxalate Method of Treating Wastes before Recycling

Further dummy runs have been made, following the procedure outlined in last week's report and using 50% KOH, 40% NaNO$_2$, and solid KMnO$_4$. Of the five dummy runs completed by the latest version of the method, two were unsatisfactory because of poor settling of the "lanthanum oxalate" precipitate. It is suspected that lanthanum hydroxide was actually responsible for the poor settling, but it is not known whether extra lanthanum was present, too little oxalic acid was added, or some other factor was responsible. The plant runs are being actively followed and paralleled with laboratory runs using plant solutions. The method is felt to be inherently sound and should soon be demonstrated sufficiently by dummy runs for moving on to the production test which is planned.

Volume of PR-3 Tank

As discussed previously, the use of 95% HNO$_3$ was adopted at Runs T-5-07-D-3

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and-B-5-07-B-3 for dissolving the second peroxide precipitate. This was intended to keep the total volume of second cake solution as low as possible in order to avoid exceeding the capacity of PR-3 Vacuum Receiver from which the still is charged. With the smaller batch size to be processed in current and future runs, this problem is no longer as critical as formerly. Starting with Runs T-5-07-D-6 and B-5-07-B-6, therefore, 60% HNO_3 is again being used routinely; a volume check is made before adding the acid and 95% HNO_3 may be used at the supervisor's discretion.

Differences between HEW and consumer assay of Shipments

It was thought that incomplete removal of product from the sample can by the consumer might be responsible for the difference in assay. Therefore, a number of returned cans were flushed with 6% HNO_3 which was then assayed to determine the amount of product returned in the can. Although the amounts found were large by H.I. standards, they were far too small to be a factor in the discrepancy under discussion. Thus, a total of only 0.011 grams was found in 6 cans with a maximum of 0.006 grams in one.

The balance and weights used to weigh sample cans before and after loading with final product solution (AT) might also be suspected of inaccuracy. A thorough check has revealed no sources of error of the magnitude under discussion.

Consideration of the current operating procedure has revealed a source of error that may account for part or all of the discrepancy between HEW and consumer assay of shipments. Before the concentrated product solution is transferred from the still to AT (the adjustment tank), the practice has been to send a 25-ml. flush of 6% HNO_3 through the vacuum receiver to AT and drop it to the recovery tank ST-2. In this operation a small volume of solution is trapped below the AT tank ahead of the valve in the line leading to WT (the weigh tank). When the product solution is subsequently brought to AT and then dropped to WT and thence to the sample can, this small volume flows ahead of the actual AT solution and is weighed into the sample can as if it were part of the AT solution assayed for production content.

The volume of holdup probably varies from run to run and from cell to cell. Two checks made in Cell 2 (which is uncontaminated) showed that 6 grams and 4.5 grams, respectively, of solution were trapped as described above. The first solution was apparently strong HNO_3 from cell flushing while the second was water added for the test. The total volume of the two solutions was 9-ml. This indicates that about 4.5-ml. were held up each time. If such a volume of 6% HNO_3 were included in the kilogram of AT solution normally loaded to a can, the error would be 0.45%. If the volume of the holdup were larger (as it may be in Cells 3 and 4), the percent error would be greater. Further, the error would not show up on every sample can but only on cans containing the first portion loaded from each run. A check of the latter will be made when data are available.

The 6% HNO_3 wash has been omitted from all runs after T-5-07-D-5 and B-5-07-B-8.

Process Chemistry

Fluomolybdate as a Substitute for Fluosilicate

Product carrying tests in the $BiPO_4$ product precipitation step, carried out in the presence of fluomolybdate, indicated that waste losses were of the same order as in a control run with fluosilicate. Not enough tests have been run, however, to determine whether there may be minor differences.

The specific decontamination of lanthanum in the product precipitation step has been determined using lanthanum tracer. The decontamination factor for lanthanum through the step in the presence of fluomolybdate was in the same range as previously obtained in similar tests made with fluosilicate present. The addition of either reagent decreases the decontamination of lanthanum.

Recovery of Product from First Cycle By-Product Precipitates

As reported last week, laboratory tests indicated that the difficulty encountered in the rework of a first cycle by-product precipitate was probably caused by incomplete dissolution of the comparatively large $BiPO_4$ product precipitate in the volume of nitric acid used. Spectrographic analyses of the $BiPO_4$ product precipitate from the laboratory test run showed that it contained less than 1% of Zr, Ce, or Si so that it does not appear that a sufficient amount of a fluosilicate compound was co-precipitated with the $BiPO_4$ to interfere with subsequent oxidation. In this rework procedure a $BiPO_4$ product precipitation is made from the HNO_3 solution of the scavenger by-product precipitate in the presence of sufficient fluosilicate to solubilize the zirconium phosphate scavenger. This precipitate is then dissolved in HNO_3 and carried through a standard second decontamination cycle. In the plant rework run a high loss was encountered in the by-product precipitate of this cycle.

Solubility of $Bi(OH)_3$-$BiPO_4$ Mixtures in 32% HNO_3

In the last run of each series in the plant it is planned to clean out the precipitators (used in the $BiPO_4$ product precipitation steps of the decontamination cycles) by putting the flowsheet quantity of 60% HNO_3 into the tanks and diluting down to 32% to increase the volume sufficiently to obtain adequate agitation for dissolution of all residual $BiPO_4$. In order then to insure complete dissolution of the product precipitate cake in the dilute acid (32%), with an adequate margin of safety, it is planned to metathesize the $BiPO_4$ cake to $Bi(OH)_3$ by a KOH wash. Laboratory solubility tests indicate that stable solutions are obtained if 60% of the total $BiPO_4$ is converted to $Bi(OH)_3$. These tests were made on the basis of an assumed accumulation of an amount of $BiPO_4$ equivalent to about 40% of that used in one first cycle product precipitation step, an amount considerably in excess of that likely to be encountered.

Disposal of Decontamination Cycle Wastes

It was reported previously that samples of solutions taken from the first and second decontamination cycle waste storage tanks still contained significant amounts of product not carried on solids which settled out. The high product content of these solutions has been verified by cross-check analyses. Additional samples from both areas have been obtained for additional product and gross fission product assays.

M. F. Acken

M. F. Acken, Chief Supervisor
Sep.Eng.Div. 200 Areas Tech.Dept.

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August 13, 1945

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200 AREAS

August 3 Through August 9

Canyon Buildings (221)

Time Cycles (T)

Revisions in production schedules have increased the number of batches to be processed per month. Because of this fact and the desirability of providing the maximum amount of downtime for maintenance and unforseen delays, further efforts are being made to reduce the unit time cycles. Accordingly, some minor changes in operating technique and the sequence of operating steps have been made to obtain an estimated minimum cycle of 18 hours or less:

Section	Cycle time in Hours 8	13	14	16	17
"Old" Minimum	22-1/2	19	20	15	18-1/2
"New" Minimum	17-1/4	17	16-3/4	14	15-1/2

The major time saving results from sampling the product waste solutions (Sections 8, 14, 17) on completion of centrifugations (-3W sample) instead of on completion of cake removal (-3WS sample). This permits an analysis of the waste to be obtained by the time the precipitator is empty and ready to receive another charge. As was predicted on the basis of previous experience, the "-WS" analysis averages about 0.10% higher than the "-W" or present "throw-away" analysis.

Additional time saving was effected in Section 8 by the concurrent addition of metal solution and reagents to the precipitator, in Section 13 by a combination of jacket and sparger heating plus starting the scavenger precipitation concurrent with the completion of cooling, and in Section 14 by a combination of jacket and sparger heating.

As indicated below, initial operation with the shortened procedures has resulted in appreciable time saving in Sections 8, 13, and 14. It should be pointed out that the actual "working" time cycle will probably average approximately 10% longer than the "estimated minimum" cycle through maintenance delays and other unforseen complications. It is also probable that the new estimates regarding sections time cycles

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represent nearly the minimum obtainable with present process conditions and reflect continued work in this direction since the plant start-up.

Run	Cycle Time in Hours Sect. 8	 Sect. 13	 Sect. 14
Old Minimum	22-1/2	19	20
07-D-8	23	20-3/4	19-1/2
07-D-9	24-1/4	23	18-1/4
08-D-1	24	22-1/4	17-1/2
New Minimum	17-1/4	17	16-3/4
08-D-2	19-1/4	19	17-1/4
08-D-3	18	18-1/4	16-1/4
08-D-4	19	17	17-1/4
08-D-5	19	19	18
08-D-6	19-1/4	18-3/4	

Extraction (B)

During the extraction precipitation of Run 08-B-3, the agitator (8-1) cut out. Operation was resumed after the unit was greaded. The product slurry was digested for 3 hours at 75°C. The waste loss was slightly lower than normal.

Oxidation (B and T)

The acidity at which the product cake solutions are oxidized (in 12-7 and 14-4) was reduced from 7.5N to 6.5N on runs T-5-07-D-6 and B-5-08-B-1. A possible reduction in the product lost in the scavenger by-product precipitate may be indicated although an extended demonstration will be required to prove this point.

Decontamination Cycles (B and T)

The 24% $BiONO_3$ solution used in the extraction step has replaced the 14% solutions in the B Canyon for making the product precipitations in the decontamination cycles. This change was made with Run B-5-08-B-1 and no effect has been observed in the performance of the process.

During the recent series of runs in the T Canyon an appreciable number of decontamination cycle product cake solution samples have been found to contain turbidity to the extent of 0.05 to 0.5 gram/liter. The major source of this turbidity has been traced to the small amounts of nitric acid insoluble material in the ammonium silico-fluoride and ferrous ammonium sulfate solutions and is introduced into the system by the addition of these reagents. A laboratory check of the turbidity produced in 10N nitric acid by the addition of a proportional amount of the centrifugable material from representative samples of these reagents is in agreement with turbidities encountered in plant product cake solutions. Existing analyses indicate that the amount of product associated with the insoluble material which gave rise to the turbidity is essentially constant and somewhat independent of the amount of the residue. The variation in the amount of turbidity is attributed to the degree of settling which has occurred in the reagent storage tanks at the time of addition. Several schemes for the elimination of the insoluble material from the reagent solutions are under consideration.

A summary of the data concerning the turbidity and crud analyses obtained on recent samples is given below. No consistent correlation of these data with the subsequent by-product losses is possible.

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Run No.	Turbidity 14-4 -P	Product On Insoluble Residue	Turbidity 17-4-P	Product on Insoluble Residue
T-5-07-D-6	Clear	--	1) 0.5 g/l 2) 0.2 g/l	0.20%
D-7	Clear	--	0.05g/l	--
D-8	Clear	--	0.4 g/l	--
D-9	Clear	--	0.1 g/l	--
T-5-08-D-1	1) 0.5 g/l 2) 0.2 g/l	0.20%	1) 0.3 g/l 2) 0.3 g/l	0.20
T-5-08-D-2	0.05g/l	--	1) 0.2 g/l 2) 0.1 g/l	
T-5-08-D-3	Clear	--	Clear	--
T-5-08-D-4	Clear	--	0.5 g/l	0.14
T-5-08-D-5	Clear	--	Clear	--

Concentration Buildings (224)

Process volumes are 60% of start-up volumes, and 50% extra lanthanum (3.75 lbs. total) is being used in the LaF_3 product precipitation. Major differences consist of recycling of 8.5 lbs. of lanthanum to the LaF_3 by-product and a slurry wash of the resulting centrifuge cake at T-Area, as compared to the use of 5.5 lbs. of fresh lanthanum in the by-product and a displacement wash of the cake at B Area.

LaF_3 By-Product

The use of 8.5 lbs. of lanthanum in the LaF_3 by-product, added completely as recycle, has not caused any increase in the by-product waste loss (D-4-BP) at T Area. A two-batch precipitator-centrifuge slurry wash of the LaF_3 by-product cake is being used in these runs to keep waste losses due to cake hold-up at a minimum.

A standard 5.5 lb. lanthanum cake is in use at B Area. It is obtained by adding fresh lanthanum from the gallery. A straight precipitator-centrifuge displacement wash is being used in these runs.

The waste losses (D-4-BP) are summarized in the tabulation below for recent runs. The 8.5-lb. cake with slurry wash averages 0.62%, the 5.5-lb. cake with slurry wash averages 0.65%, and 5.5-lb. cake with displacement wash averages 0.74% for product loss. All loss calculations were corrected for recycling to give the results on a performance basis.

LaF_3 By-Product (D-4-BP) Waste Losses

Run No.	% 8-1-M Prod. Recycled	Lbs. La in By-Prod. Cake	% D-4-BP 8-1M Basis	% D-4-BP Perf. Basis (Corrected)*	Type B.P. Cake Wash
T-5-07-D-6	12.9	8.5	0.65	0.58	Slurry(2-batches 1N HNO_3)
T-5-07-D-7	6.3	8.5	0.75	0.70	Slurry(2-batches 1N HNO_3)
T-5-07-D-8	8.2	8.5	0.80	0.74	Slurry(2-batches 1N HNO_3)
T-5-07-D-9	None	5.5	0.63	0.63	Slurry(2-batches 1N HNO_3)
T-5-08-D-1	8.2	8.5	0.66	0.61	Slurry(2-batches 1N HNO_3)
T-5-08-D-3	None	5.5	0.68	0.68	Slurry(2-batches 1N HNO_3)

LaF_3 By-Product (D-4-BP) Waste Losses (Cont'd.

Run No.	% 8-1-M Prod. Recycled	Lbs. La in By-Prod. Cake	% D-4-BP 8-1M Basis	% D-4-BP Perf.Basis (Corrected)*	Type B.P. Cake Wash
B-5-07-B-6	None	5.5	0.42	0.42	Displacement(1-batch 1N HNO_3)
B-5-07-B-7	None	5.5	0.78	0.78	Displacement(1-batch 1N HNO_3)
B-5-07-B-8	None	5.5	0.82	0.82	Displacement(1-batch 1N HNO_3)
B-5-07-B-9	None	5.5	0.68	0.68	Displacement(1-batch 1N HNO_3)
B-5-07-B-10	None	5.5	0.70	0.70	Displacement(1-batch 1N HNO_3)
B-5-07-B-11	16	7.0	0.97	0.83	Displacement(1-batch 1N HNO_3)
B-5-07-B-12	1	5.75	0.95	0.94	Displacement(1-batch 1N HNO_3)

* Corrected to performance basis for amount of recycled product.

LaF_3 Product Precipitation

The use of 50% extra lanthanum (3.75 lbs. total) in the Cell E LaF_3 product precipitations has been continued at both T and B Areas. Final product waste losses (E-3-WS) have continued to average well below the desired standard of 1.5%, averaging 1.20% at B Area and 0.77% at T Area, corrected for recycling to a performance basis.

It is noticeable, however, that the same differential of approximately 0.4% between T and B Area losses is still being obtained as was the case when losses were at higher levels (1.4% at T Area vs. 1.8% at B Area). The significant difference seems to lie in the efficiency of the second strikes. At T Area, the product content of the first effluent is reduced by a factor of 7.5 in the second strike, while the corresponding factor at B Area is only 5.6. The product content of the first effluents at the respective areas is not markedly different. The reasons for this are believed to be mechanical but are unknown at present.

LaF_3 Product Cake Removal

Completeness of removal of the extra-weight LaF_3 product cakes from the centrifuge E-2 is being studied at both T and B Areas. The centrifuge Beckman meter readings and the LaF_3 product metathesis slurry (F-1-PS) assays are being used as the measures of cake removal. The extensive slurrying procedure being used at B Area was described in the Progress Report of August 1. This procedure involves (a) a 700-lb. water flush with the bowl sprays, (b) four slurryings from 0 to 110 RPM with 250 lbs., (c) a 15-minute rotation at 10 RPM and fifth slurrying with the same water, (d) five slurryings with 200 lbs. and (e) a 150-lb. flush with the sprays. This method is designated as "Method A". It has been successful thus far in producing empty bowl Beckman meter readings below $7x10^{-14}$ amp.

At T Area, "Method B" is being used. This method is simpler, involving (a) a 700-lb. flush with the sprays, (b) three slurryings from 0-110 with 250 lbs., (c) three slurryings with 200 lbs., and (d) a 150-lb. flush with the sprays. This method performed satisfactorily for 3 runs, producing empty bowl Beckman readings of $7.5x10^{-14}$ amps. On the fourth run, however, the reading increased to $18x10^{-14}$ and has remained at $15x10^{-14}$ since then. It appears that incomplete removal is largely due to the failure of the initial 700-lb. spray occasionally to perform its usual nearly complete cake stripping and this step in the cycle may have to be modified.

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Beckman Meter Records

A set of curves recording Beckman meter readings for certain critical process points is in use and will be continued as a record of full and empty vessel readings for monitoring purposes.

Isolation Building (231)

Oxalate Method of Treating Wastes Before Recycling

Twenty dummy runs in Cells 1 and 2 have been completed. Minor changes in Runs 11, 13, 15, and 20 have led to a procedure that is felt to be satisfactory insofar as a dummy run can demonstrate. The last run was made in the large catch tank CT-1, of Cell 2 at the volumes which would be required in a regular plant run. Conditions for this run may be outlined as follows:

1) About 96 liters of combined first and second cycle supernates are treated with 10.5 kg. of 50% KOH to give acidity of about 0.5N.
2) Forty percent $NaNO_2$ is added until all hydrogen peroxide is destroyed (about 25 kg. required).
3) Oxalic acid equivalent to a 50% excess over the amount of La present is added (about 38 kg. of 9.2% $H_2C_2O_4 \cdot 2H_2O$).
4) Fifty percent KOH is added until the white precipitate ($La_2(C_2O_4)_3$) turns red-orange due to $Fe(OH)_3$ precipitation (about 10 kg. required).
5) A small fixed excess (3 kg.) of 50% KOH is added to give a definitely alkaline solution (total volume = 180-l.).
6) After 30 minutes settling, the supernate is decanted to leave a volume of 10 to 15 liters.
7) 65 kg. of water are added with agitation.
8) After 30 minutes settling, the supernate is again decanted to about the same volume.
9) The precipitate is dissolved in 17 kg. of 60% HNO_3.
10) Solid $KMnO_4$ equivalent to about 80% of the oxalate is added (about 1 kg. required).
11) 4% $KMnO_4$ solution is titrated in until a brown precipitate (MnO_2) indicates all oxalate destroyed. (about 5 kg. are required; the total volume to be recycled to 224 is then 25 to 40 liters).

One additional dummy run at the full volume scale in Cell 2 is planned to check the observations made on Run 20. A production test can then be made with 1/3 of full scale charges in P-1 tank (precipitator) of Cell 3 or 4.

Size of P-1 Sample

For several months the official P-1 assay has been based on the "bulk" sample (about 10g.) although "peanut" samples (0.3 g.) were also taken in triplicate on each run. The latter have not agreed with each other and have almost always been higher than the bulk sample values.

The concensus has been that evaporation in the sample cup while the peanut sample is being removed by pipette caused the higher assay. Minor changes in equipment and technique have not eliminated the trouble. Confidence in the bulk sample value was reinforced by taking two successive bulk samples on Run B-5-07-B-11. These samples checked very closely whereas the three peanuts on the same run were again high and inconsistent. Although the peanut sampling technique is safer than the bulk, it has been necessary to discontinue the former and continue the latter; this was done with Runs T-5-07-D-9 and B-5-08-B-1.

Undissolved Solids in AT Solution

Plant runs T-5-07-D-8 and 9 have been sampled to check the earlier finding that the use of "Becco" hydrogen peroxide has reduced the suspended solids in final product solution (AT) below 100 mg. per 80 grams of product. These two runs were sampled in duplicate from the adjustment tank (by pipette) and from the weigh tank (by draining from bottom outlet). The values found from these respective duplicates were 42 and 44 mg. per 80 grams of product on T-5-07-D-8. Values for T-5-07-D-9 are not yet available.

Thread Seizure in Sample Cans

Instances have been reported where the consumer had difficulty in unscrewing the filter cap assembly from a sample can. Measures taken to prevent this in the future include: (1) a careful check and lubrication of all parts of each sample can involving threaded stainless steel; (2) use of two (instead of one) gaskets below the filter cap in the present can assembly; and (3) a proposed can design change to replace the threaded fit of filter cap into can by a pressure fit employing the lid of the can carrier and its thumb screws to seat the cap tightly. A design change has also been proposed to replace the threaded retainer ring in the filter cap assembly with a snap ring.

Cell Cleanouts

In one cleanout, material in sump tank (ST-2) of Cell 4, consisting mostly of overflow solution, from the receiving tank (PR-3) was sent to PR-3, charged to the still S-1, and evaporated down for loading to a can via the adjustment tank (AT) and the weigh tank (WT). The purity on the AT solution was 95.3%, barely satisfactory; the low value probably arose from impurities in ST-2 from previous solutions held there after less than two peroxide cycles.

In Cleanout No. 20, Precipitator P-1 in Cell 3 was washed ahead of Run T-5-07-D-6 in order to remove any heel left from Run No. 5. The material recovered was sent to ST-2 and blended off, with other solution in ST-2 from PR-3 overflow, in Runs 6, 7, and 8.

In No. 20, the nutsche (N-2) in Cell 4 was leached, backwashed, and pre-coated after Run B-5-07-B-12. This was last done before B-5-07-B-1.

Process Chemistry

Decontamination Cycle Wastes

Additional samples of first and second decontamination cycle waste solutions taken from waste storage tanks in both 200 Areas have been examined for gross beta and gamma activity and product content. The values obtained are summarized below and apply to supernatant solution after removal of solids by gravity settling.

Source of Solution	β Counts/min./cc (2nd shelf)	γ Counts/min./cc (2nd shelf)	α Activity Counts/min/cc	µg/l	pH of Solution
Storage Tank, 107 (first cycle) West Area	2.0×10^6	1.0×10^4	2220 (2030)*	31	10.2

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Source of Solution	β Counts/min./cc (2nd shelf)	γ Counts/min./cc (2nd shelf)	α Activity Counts/min/cc	μg/l	pH of Solution
107 East Area	3.2×10^6	1.8×10^4	2320 (2470)	33	10.2
Storage Tank 110 (second cycle) West Area	6.9×10^4	53	1320 (1430)	19	9.8
110 East Area	7.0×10^4	35	600 (980)	8.5	9.6

* Cross-check by Control Laboratory (222-B)

The values given above on the samples from the 107 and 110 Storage Tanks in the West Area are essentially in agreement with those reported previously on first set of samples taken. The above data show that activity levels in the corresponding tanks in the two areas are not significantly different. Tests indicate that most of the product is in the Pu(IV) valence state and also that the alpha activity present is due to product.

Fluomolybdic Acid and Ammonium Fluomolybdate

In order to obtain information as to the physical characteristics of fluomolybdic acid and ammonium fluomolybdate, samples of these materials were prepared by evaporating solutions of molybdic acid ammonium molybdate in water and HF. The free acid is a dense, crystalline non-hygroscopic material that is subject to slight discoloration, but the solutions of which are clear and colorless. The ammonium salt is also a dense crystalline material and appears more color-stable than the free acid.

Fluomolybdate solutions, used in previously reported tests comparing the effectiveness of fluomolybdate and fluosilicate in improving decontamination in the $BiPO_4$ product precipitation steps, were prepared simply by adding the calculated stoichiometric amount of HF to molybdic acid or ammonium molybdate suspended in water.

Radio Assay of Films Deposited from Cooling Water on Slugs in the 105 Building

An attempt is being made through Radioassay to obtain further information concerning the composition of films deposited on slugs from the cooling water. The first approach will be to obtain as complete a characterization as possible of the activities present in the films removed from slugs as they are pushed from the pile. The first objective of this study is to determine whether any differences in film composition can be found which might account for the generally more rapid increase in pressure drop in 100-F than in 100-D or 100-B.

Characterization will be made by obtaining decay and absorption curves on the samples both with and without chemical separation. The separation scheme to be used has been tested in a preliminary way and is being used in the assay work which is now in progress on the first samples which were obtained from 100 D.

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Tracer Preparation

Some time ago smear samples were taken from one of the Canyon cell covers for assay of activities present. The portion of the sample not used at that time was retained for future recovery of ruthenium tracer. The total activity which has been recovered and concentrated and is available for use is about 9 x 10^8d/min. distributed as follows:

42 day Ru	52.5%
1 year Ru	46.5%
gamma background	1%

M. F. Acken
M.F.Acken, Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.Dept.

MFA:jd

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#1 R.M.Evans
#2 R.M.Evans
#3 W.O.Simon for The Area Engineer
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#5 W.O.Simon for The Area Engineer
#6 W.O.Simon for The Area Engineer
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#7 N. Hilberry
#8 N. Hilberry
#9 N. Hilberry
#10 W.O.Simon-B.H.Mackey-700 Area
#11 M.H.Smith
#12 L.Squires

#13 300 Area File
#14 Pink Copy
#15 Yellow Copy
#16 M.F.Acken
#17 W.C.Kay - J.E.Cole
#18 R.S.Apple
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200 AREAS
August 10 Through August 16

Canyon Buildings (221)

Acid Wash (B)

An acid wash (B-5-08-AW-1) has been made in the B Canyon, following the same procedure used for the June wash. The improved performance of the acid sprays in the product precipitators (14-1 and 17-1), resulting from longer circulation at lower steam pressure, is apparent from the very small amount of product recovered from these vessels. The sampling points and the product found, based on the preceding run, are tabulated below:

B-5-08-AW-1 Sample Designations	Sample Description	% of Preceding Run (B-5-08-B-7) Starting Solution (8-1-MR)
8-1-P	Wash Acid in 8-1 precipitator	0.14*
8-4-P-1	Wash Acid after transfer through 8-1 and 8-2 centrifuge	0.20
8-4-P-2	Wash Acid after agitating in 8-4 solution tank	0.40*
12-7-P-1	Wash Acid after agitating in 12-6 solution tank	0.72*
12-7-P-2	Wash Acid after agitating in 12-7 solution tank	0.72*
13-1-P	Dissolving Acid in 13-1 precipitator	0.01
13-4-BP	Regular waste on AW Run	0.05
13-3-P	Product Sample on AW	0.79*
14-1-P	Wash Acid in 14-1 precipitator	3.9
14-4-P-1	Wash Acid after 14-1 and 14-2 centrifuge	1.7
14-4-P-2	Wash Acid after agitating in 14-4 solution tank	1.7
14-4-P-3	Regular sample on AW	2.6
14-3-WS	Regular sample on AW	0.01
16-1-P	Dissolving Acid in 16-1 precipitator	0.02
16-4-BP	Regular sample on AW	0.06
16-3-P	Product sample on AW	3.3 *

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B-5-08-AW-1	Sample Description	% of Preceding Run (B-5-08-B-7) Starting Solution (8-1-MR)
17-1-P	Wash Acid in 17-1 precipitator	4.4
17-4-P-1	Wash Acid after 17-1 and 17-2 centrifuge	1.5
17-4-P-2	Wash Acid after agitating in 17-4 solution tank	1.5
17-4-P-3	Regular sample on AW	4.4*
17-3-WS	Regular sample on AW	0.02

* Cumulative values

37 Recovery

A second run made to recover Np^{237} from a metal waste has been processed through the B Canyon. No difficulty was experienced in re-extracting the extraction waste from Run B-5-07-B-7. The product content of the charge was very low:

Assay Point	% of Precding Run (B-5-08-B-7) Starting Solution (8-1-MR)
8-4-P (Extraction Product Solution)	0.53
14-4-P (1st cycle product solution)	0.53
17-4-P (2nd cycle product solution)	0.6

Ferrous Ammonium Sulfate (T)

Use of the Metalloy ferrous ammonium sulfate, although containing more acid insoluble matter (0.025%) than the previous material, has been made without difficulty on several runs. At least 9 hours settling, plus a standpipe draw off, has given clean first and second cycle product solutions.

Concentration (224) Buildings

LaF_3 By-Product

The substitution of a displacement wash of the 8.5 lb. LaF_3 by-product cake in centrifuge D-2 for the double-batch slurry wash at T Area caused the by-product waste loss (D-4-BP) to rise from an average of 0.60% to 1.16%. It appears desirable to retain the more involved method of slurry washing at least for these larger LaF_3 by-product cakes.

There appears to be little difference in waste losses for the 5.5-lb. La cakes as affected by either slurry or displacement wash. T Area, with a slurry wash, averages 0.58%; B Area, with a displacement wash, averages 0.66%.

LaF_3 Product

The continued use of 50% extra lanthanum (3.75 lbs. total) in the Cell E LaF_3 product precipitations has averaged 1.2% at B Area and 0.80% at T Area for product waste (E-3-WS) losses. Run T-5-08-D-7 was given two full displacement washes because of operating difficulties and the resultant E-3-WS loss was 1.6%. This loss was reduced to 0.69% by recentrifugation and combination of the product recovered with Run D-8 in the centrifuge E-2.

Recent data are as follows:

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LaF_3 Product Waste Losses (E-3-WS)

Run No.	% E-3-WS Losses* Cent. #1	Cent. #2 plus Cake Wash
T-5-08-D-3	5.29	0.72
T-5-08-D-4	4.60	0.65
T-5-08-D-5	4.74	0.88
T-5-08-D-6	5.5	0.91
T-5-08-D-7	5.97	1.62 (0.69)
T-5-08-D-8	5.45	0.81
T-5-09-D-9	4.71	0.76
B-5-08-B-1	--	1.08
B-5-08-B-2	--	0.95
B-5-08-B-3	--	1.29
B-5-08-B-4	--	1.22
B-5-08-B-5	--	1.23
B-5-08-B-6	--	1.26
B-5-08-B-6	--	1.21

* Corrected to performance basis for recycling.

LaF_3 Product Cake Removal

Variable quantities of product continue to be held up in the E-2 centrifuge in both areas despite all attempts to develop a more satisfactory cake removal procedure.

Inspection of the Beckman readings during the cake removal cycle at both Areas makes it evident that the major part of the cake removal is accomplished by the initial 700-lb. spray and that the involved slurrying accomplishes little additional cake removal. Unfortunately the initial 700-lb. spray is not always as efficient in cutting the major portion of the cake off the wall and/or breaking it up in small enough lumps for it to be easily jetted out of the bowl. The slight additional removal effected by the final 150-lb. spray indicates that more water spray alone may not necessarily solve the problem.

Consideration is being given to the use of a higher pressure spray, such as is used in F-2 in order to effect more complete cake removal.

The E-2 Beckman data for recent runs are given below:

Centrifuge E-2 Beckman Readings ($x10^{-14}$ amps.)

Run No.	Start Empty	Full	After 700-lb Spray	After 250-lb Slurry	After 200-lb Slurry	After 150-lb Spray	Cake Removal Procedure*
T-5-08-D-3	14	94	20	--	18.5	14	Method B
T-5-08-D-4	15	95	26	--	22	18	Method B
T-5-08-D-5	19	72	24	--	16	15	Method B
T-5-08-D-6	15	73	24	--	17.5	15	Method B
T-5-08-D-7	14	56	20.5	--	17	16	Method B
T-5-08-D-8	14	145	12	--	8	7	Method B
T-5-08-D-9	7	135	20	--	12.5	12.8	Method B

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Run No.	Start Empty	Full	After 700-lb Spray	After 250 lb Slurry	After 200-lb Slurry	After 150-lb Spray	Cake Removal Procedure*
B-5-08-B-1	3.5	90	13	7	5	4	Method A
B-5-08-B-2	4	74	22	19	8	9	Method A
B-5-08-B-3	9	100	25.5	23	16	16	Method A
B-5-08-B-4	15	76	27	22	14	14	Method A
B-5-08-B-5	13	92	--	--	--	18	Method A (No 10RPM slurry)
B-5-08-B-6	16	83	--	--	--	21	Method A
B-5-08-B-7	20	85	--	--	--	22	Method A

* As given in last weeks report

Cell E Acid Wash

Prior to the arrival of the Canyon Building acid wash run at B Area (B-5-08-AW-1, an acid wash of the E-2 centrifuge (B-5-08-EAW) was carried out as follows: (1) three portions of approximately 250 lbs. of 60% HNO_3 each were successively added to and slurried in E-2 and then skimmed to catch-tank E-3; (2) approximately 700 lbs. of 60% HNO_3 was then passed through the bowl, overflowing to E-3; and finally (3) 13,000 lbs. of water were sprayed into the bowl and jetted continuously to E-3.

A product assay of the wash solution in E-3 showed a pick-up from E-2 equivalent to 23.1% of the 8-1-MR of Run B-5-08-B-7. The E-2 Beckman reading dropped from 22×10^{-14} to 5×10^{-14} amps. during the acid wash and from then down to 0 during the water flush. Rework of this acid wash solution by the addition of a single shot of 1000 lbs. of 1% lanthanum salt solution resulted in a loss of 0.83%. The LaF_3 metathesis slurry (F-1-PS) assay of 27.7% showed a still further pick-up in the E-2 to F-1 line or in the F-1 heel. The E-2 Beckman returned again to zero after removal of the rework LaF_3 product cake.

F-1-PS Product Assays

Recently the T Area LaF_3 metathesis slurry product assays (F-1-PS) have frequently been erratically high, indicating in at least one run a product yield as high as 250% at this point. It is suspected that poor or incomplete mixing of the slurry in F-1 and/or non-representative sampling are the causes of these irregular assays.

Tests have indicated that sampling immediately after receipt of the product cake slurry from centrifuge E-2 may cause high and non-consistent assays, possibly due to the presence of large lumps of cake. Additional agitation appears to produce lower and more consistent assays. These sampling studies are being extended in an effort to obtain confirmatory data and to establish the minimum agitation time required.

Time Cycles

An evaluation of the time cycles involved in Cell E and F operations has been made recently as follows:

Process Time Cycles

Cell	Estimated Minimum Cycle	Remarks
E	(a) $19\frac{1}{2}$ hours	Includes E-1-R(temporary) and E-3-WS-1 Samples
	(b) 17-1/4 hours	Includes discarding of E-3-WS waste on basis of WS-1
	(c) 15-3/4 hours	Item (b) plus entry of next charge after cake wash
F	15-3/4 hours	[redacted]

On the basis of these estimates it appears that a practical time cycle of 18 hours for the Concentration Building is feasible, assuming values about 10% above the minimum estimates to provide for maintenance and other unforseen delays.

Isolation Building (231)

Oxalate Method of Treating Recycles

Two additional full scale dummy runs have been made in Cell 2 as Numbers 21 and 22. The purpose of Run 21 was to check the satisfactory operation of Run 20; the maximum volume was 178 liters and the final cake solution volume was 29 liters. The former value is felt to be typical of the volume which should result in the average or "normal" plant run. It does not leave a very large margin of safety for "abnormal" runs since the large catch tank (CT-1) would overflow at about 200 liters. It is therefore desirable to investigate means of lowering this maximum volume still further; addition of solid KOH, use of a mixed KOH-$NaNO_2$ solution, or use of KNO_2 instead of $NaNO_2$ (former more soluble) are being considered.

The purpose of Run 22 was to determine whether the decant line for CT-1 could be located on the high side of the sloping bottom. It was found that the cake settled with about the same slope as the bottom of the tank. Decanting to the cake level on the high side of the tank left about three liters of supernate which could then be removed by decanting from the low side. If mechanically feasible, therefore, it will be desirable to install the proposed dip pipe in such a manner that it will drain the low side of the tank.

The final cake solution volume obtained in Runs 20, 21, and 22(22.8, 29, and 26 liters respectively) is probably smaller than it will be possible to obtain routinely with a built-in decanting system. On these runs, decanting was done by means of a hand-directed Saran dip tube with which it was possible to leave extremely small heels (9.5, 7.8, and 9.0 liters per run, respectively). The results indicate, however, that use of solid $KMnO_4$ for 80% of the permanganate requirement will result in volumes conveniently handled in one RC can under routine operating conditions.

Control of Batch Size

With the present system of sampling and analysis there is no opportunity to check batch size between the P-1 assay (of filtered $La(NO_3)_3$ solution received from 224 Building) and the AT assay (of concentrated product solution to be charged to sample can). In case an appreciable fraction of one run is left behind, it may be combined with the following run before a low AT assay on the first run is received. It would seem desirable to sample P-2, either before the second precipitation or after dissolving the second cycle peroxide precipitate, to permit earlier and better diagnosis of such holdups.

Undissolved Solids in AT Solutions

The AT sample of Run B-5-08-B-2 was found to contain 100 mg. of solids per 80 g. batch of product, while the WT sample contained 120mg./batch. Two conclusions may be drawn from these results and the results reported last week on Run B-5-07-B-8 (erroneously reported last week as T-5-07-D-8) (42 mg./batch from AT and 44mg./batch from WT): (1) the low solids content previously reported for AT solutions from early runs employing "Becco" H_2O_2 has continued after 2 months operation; (2) there is essentially no difference in the amount of solids by sampling AT by pipette or by sampling WT via the drain spout.

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Process Chemistry

Waste Disposal Studies

Further study of supernatant liquors (after removal of solids by settling) from the first and second decontamination cycle waste storage tanks (109 and 110) has shown that the solutions contain a considerable amount of dissolved bismuth and that the amount is dependent upon the pH of the solution. Bismuth content and pH of each of four samples are given below.

Source of Solution	pH	Mg. Bi/l.
107 West Area	10.23	343
107 East Area	10.18	427
110 West Area	9.78	54
110 East Area	9.58	30

Upon acidification of these solutions, precipitation of what is presumably bismuth hydroxide occurs. Data given below show that by neutralization of a sample of solution from the second cycle storage tank (110 West Area) to a pH of 6, about 95% of both the alpha and beta activity was carried by the precipitate. The solutions after adjustment of pH were allowed to stand overnight before centrifuging.

pH	alpha Activity c/m/cc (52% Geom.)	Beta Activity c/m/cc (2nd shelf)
9.8 (as received)	1300	6.9×10^4
8.2	980	5.0×10^3
6.0	63	2.9×10^3

Titration of a sample of unneutralized waste from the second cycle, using a Beckman pH Meter, shows that there is an inflection point at a pH of about 6 corresponding to the buffering action of the phosphate in the solution. This property of the solution may prove useful if it appears feasible to neutralize waste solutions to this pH in order to obtain more complete removal of the activities. Data given below show the amount of activity and bismuth remaining in the supernatant after neutralization of a second cycle unneutralized waste solution (from 15.9) to various pH values. The solutions were allowed to stand for only 4 hours before removal of the precipitate by centrifugation. It is possible that true equilibrium was not reached but the data nevertheless show that much better removal of activity is obtained by neutralization to a pH of about 6 than when neutralized to a pH of about 10, which is present practice.

pH	mg. Bi/l	alpha activity c/m/cc (52% Geom.)	Beta activity c/m/cc (2nd shelf)
9.8	40	778	8.3×10^4
6.7	15	139	6.9×10^3
5.6	16	47	2.5×10^3

M. F. Acken

M.F.Acken, Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.Dept.

MFA:jd

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#2	R.M.Evans
#3	W.O.Simon for The Area Engineer
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#5	W.O.Simon for The Area Engineer
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#7	N. Hilberry
#8	N. Hilberry
#9	N. Hilberry
#10	W.O.Simon-B.H.Mackey-700 Area
#11	M.H.Smith
#12	L.Squires
#13	300 Area File
#14	Pink Copy
#15	Yellow Copy
#16	M.F.Acken
#17	W.C.Kay - J.E.Cole
#18	R.S.Apple
#19	R.H.Beaton
#20	W.H.Sullivan
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200 AREAS
August 17 Through August 23

Canyon Buildings (221)

Sampling (B and T)

Further comparison of the analytical results on the product waste solution samples obtained on completion of centrifugation (-3-W) and on completion of cake removal (-3-WS) indicates only minor differences between the two samples. As a result, the -3-WS samples have been eliminated in T Area and the -3-W samples substituted for the -3-WS samples.

Extraction (B)

During heating following the nitrite pre-treatment on Run B-5-08-B-13, the precipitator (8-1) agitator again failed (for previous agitator failure see the weekly report dated August 8). Operation was resumed after approximately 8 hours delay during which the agitator and gear reducer were given a thorough lubrication. On the assumption that the higher cell temperature and humidity resulting from the 85°C pre-treatment and digestion temperature might be a contributing factor to the failure, the digestion on this run was carried out at 75°C. The waste loss on this extraction was slightly higher than usual (1.06%).

The lower pre-treatment and digestion temperature (75°C) will be used on subsequent runs to check the effect of lowered temperature on both agitator performance and waste loss.

Metal Solution Measurement and Pre-treatment (T)

Tests are being planned in T Area on the use of Section 6 for the adjustment of metal solution charge size and nitrite pre-treatment. A special precipitator (6-1) to (6-3) catch tank jet line is being fabricated and the necessary operating log book changes are being made. The initial test is scheduled to start on run T-5-08-D-25.

Decontamination Cycles (T)

Occasional turbid decontamination product cake solution samples continue to appear in T Area. A change in the ferrous ammonium sulfate make-up procedure entailing the use of an extra tank to permit settling and decantation of the solution prior to use is being held up pending the results of tests in B Area using the Metalloy ferrous ammonium sulfate which contains a higher percentage of acid insoluble material.

Production Test SE-221-T-PA-9 has been approved for a plant test to decrease the process time in the decontamination cycles by eliminating the reduction period prior to product precipitations. The present schedule calls for starting this test on Run T-5-09-D-2.

Ferrous Ammonium Sulfate (B and T)

Comparative spectrographic analyses of the acid insoluble material from the two lots of iron salt indicate that it is primarily iron, with a small amount of lead and tin as impurities. The approximate range of the impurities found in samples of the acid insoluble material from the two lots of salt are given:

Vendor	% Lead	% Tin
Baker	0.02	0.03
Metalloy	0.2 - 2.0	Trace

Wastes (B and T)

A summary of the temperature and reserve capacity data for the two areas is presented below:

Summary of Waste Solution Data

		Temperatures (°F)			
Area	Date	101 Metal Waste	104 Empty	107 Coating Removal and 1st Cycle	110 2nd Cycle
T	4-18-45	77	61	80	80
T	5-21-45	93	62	83	83
T	6-19-45	108	67	90	88
T	7-17-45	118	70	94	88
T	8-21-45	132	78	102	90
B	4-23-45	62	51	57	60
B	5-15-45	75	52	68	58
B	6-19-45	97	55	75	71
B	7-17-45	110	57	80	78
B	8-21-45	136	65	90	84

Reserve Capacity as of 8-21-45

Tanks	Type Waste	Gallons per run*	B Area Gallons	B Area Runs	T Area Gallons	T Area Runs
X-101, 102, 103	Metal	5260	1,254,000	237	1,078,300	205
X-107, 108, 109	Coating & 1st	4670	1,267,000	271	1,100,800	236
X-110, 111, 112	2nd cycle	3360	1,361,000	405	1,186,000	353

* Based on the average of the measured volumes of the wastes from the last 15 runs in both areas; approximately 1.2 tons/run, as per the 7-25-45 procedure.

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Concentration Buildings (224)

LaF$_3$ By-Product Losses

All T Area runs since T-5-08-D-9 have had a slurry wash of the LaF$_3$ by-product cake, while all B Area runs have continued to use a displacement wash. At T Area, the recent runs involving 8.0 lb. La in the LaF$_3$ by-product cake have waste losses (D-4-BP) averaging 0.76%, corrected to a performance basis. Also, at T Area, the 5.5 lb. La runs (no recycling) have waste losses averaging 0.32%. Two of the three runs involved in this average, however, ran 0.16 and 0.17% respectively, believed to be erroneously low because of sampling or analytical errors. At B Area, the recent runs average 0.57%.

LaF$_3$ Product Waste Losses

The LaF$_3$ product waste losses (E-3-WS) resulting from the use of 3.75 lbs. of La in the Cell E precipitations continue to average 1.20% at B Area and 0.84% at T Area. At T Area, the E-3-WS final waste is being discarded on the basis of the first strike effluent assay (E-3-WS-1) which averages about 5.5%. At B Area, the final waste sample is being moved up to be taken just prior to the acid wash of the centrifuge cake. This sample is expected to show about 0.1% lower loss than the former sample taken after acid wash and cake slurry removal. Both of these methods are designed to shorten the Cell E time cycle by eliminating the waiting for a final waste analysis after cake removal.

LaF$_3$ Product Cake Removal

It has been previously reported that the acid wash of the LaF$_3$ product cake centrifuge (E-2) at B Area reduced the Beckman meter reading to zero in cleaning out 23% of an 8-1-MR product weight from the bowl. Neither the Canyon Building acid wash run (B-5-08-AW-1) nor the Np^{237} run (B-5-08-IP) caused any additional cake hold-up in the bowl following this Cell E acid wash. The first subsequent production run caused the E-2 Beckman reading to rise nearly as high as before the clean-out, however, and the F-1-PS assay on this run (B-5-08-B-8) indicated a hold-up of approximately 13% in E-2.

The bowl sprays at B Area were taken out and inspected after this run and were found to be set at a 26° angle upstream (against the bowl rotation). They were replaced with a new spray set at a 15° upstream angle. The next run through (B-9) showed a slight decrease in the E-2 Beckman reading. The efficiency of this new spray for cake removal will be studied closely during the current series of runs.

At T Area, the E-2 Beckman readings have been building up slowly to a level of 20 x 10^{-14} amps. recently. This level is similar to the B Area readings of 22, which indicated a 23% hold-up. Accordingly, an acid wash of Cell E at T Area is to be carried out following Run T-5-08-D-24 and preceding the Canyon Building acid wash (T-5-08-AW-1).

The bowl sprays at T Area are to be removed on the completion of these runs and the angle of incidence measured. If the 15° angle now being used at B Area proves satisfactory, a new spray will be installed at T-Area with this angle. It is also being recommended that the present high pressure spray pump now being used on F-2 centrifuge be transferred to E-2 for trial tests when the new pumps now on order arrive.

Recent Beckman data for E-2 are given in the following tabulation:

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Centrifuge E-2 Beckman Readings (x10^{-14} amps.)

Run No.	Start (Empty)	Full	(1) After 700 # Spray	(2) After 250 # Slurry	(3) After 200 # Slurry	(4) After 150 # Spray	Cake Removal Procedure (See Report of 8-10-45)
T-5-08-D-10	9	92	21.5	--	12	16	Method B
T-5-08-D-11	15	100	40.5	--	28	27	Method B
T-5-08-D-12	10.5	123	29	--	6.5	4	Method B
T-5-08-D-13	4	45	22	--	9	19	Method B
T-5-08-D-14	18	110	27.5	--	24	18.5	Method B
T-5-08-D-15	18	55	20	--	15	16	Method B
T-5-08-D-16	17	130	43	--	23	30	Method B(20 after (3) & (4) repeated)
B-5-08-EAW	0	5	--	--	--	0	Method A
B-5-08-AW-1	1	6	--	--	--	3	Method A
B-5-08-IP	3	400	--	--	--	2	Method A
B-5-08-B-8	0	142	--	--	--	16.5	Method A
B-5-08-B-9	16.5	113	--	--	--	14.5	Method A (Note)

Note: 15° angle spray used on Run B-5-08-B-9

F-1-PS Assays

It was reported on August 17 that increased agitation of metathesis tank F-1, before sampling the LaF_3 product slurry (F-1-PS) in Run T-5-08-D-8 appeared to produce more representative and reproducible sampling. A second run, T-5-08-D-10, has been sampled at a half-hour and one hour additional agitation. While not very conclusive, the data for this run also show that increased agitation time is favorable.

Lanthanum Analyses

Recently, analyses have been made of the isolation recycle solution samples (E-4-RC) for lanthanum content preparatory to the setting up of a lanthanum accounting system based on analysis rather than the involved estimation method now being used. The analytical method involves precipitation of $La_2(C_2O_4)_3$ and an oxidation-reduction titration with a Ce^{+3}-Fe^{+2} system. The analytical data have not agreed very closely with those obtained by charge-by-charge inventory until the method of analysis was changed recently to include a more complete washing of oxalate out of the $La_2(C_2O_4)_3$ precipitated in the laboratory. The method now appears to be approaching 2 - 5% accuracy, which is to be demonstrated by further sampling and analysis.

Acid Wash Runs

Prior to the Np^{237} recovery run at B Area, an acid wash run was carried through the Canyon and Concentration Buildings, in order to flush out as much product as possible. In addition, an acid wash of Cell E was carried out, primarily to clean out the centrifuge E-2. This latter run was described in the report of August 8. The yield, loss, and product pick-up data are given below:

B Area Acid Wash Runs (Concentration Bldg.)

Process Vessel or Step	Sample Code	Cell E (B-5-08-EAW) Cum.%	Cell E (B-5-08-EAW) % Pick-up	Canyon (B-5-08-AW-1) Cum.%	Canyon (B-5-08-AW-1) % Pick-up
Basis	8-1-MR(B-5-08-B-7)	100	--	100	--
Canyon Bldg. (221)	17-4-P	--		4.4	
C-4	C-4-O	--		4.6	+0.2
A-1	A-1-OS	--		5.1	+0.5
$BiPO_4$ B.P.Loss	A-4-BP	--		0.46	
A-3	A-3-OS	--		5.4	+0.8
D-1	D-1-O	--		5.2	-0.2
LaF_3 B.P.Loss	D-4-BP	--		0.05	
D-3	D-3-OS	--		5.4	+0.2
E-2	E-8-AW-1	23.1	+23.1	--	
E-1	E-1-R	21.7	- 1.4	5.6	+0.2
LaF_3 Prod. Waste Loss	E-3-WS	0.8		0.08	
F-1	F-1-PS	27.7	+ 6.8	6.8	+1.2
15% KOH Metathesis Loss	F-7-WS	0.17		0.04	
2% KOH Metathesis Loss	F-9-WS	0.1		0.03	
Final Yield	P-1(or E-4-RC)	34	+ 6.6	9.3	+2.6
Total		34	+35.1	9.3	+5.5

Note: % Product pick-up corrected for waste losses.

Np^{237} Recovery Run

This recovery run was processed through Concentration as a regular production run with the exception that the old standard amount of lanthanum (2.5 lbs.) was used in the Cell E LaF_3 product precipitation, in order to keep the carrier/product ratio in the final solution as low as possible. Considering the low level of product being processed, waste losses as shown in the tabulation following were not considered abnormally high.

The use of high concentrations of ammonium fluosilicate in the $BiPO_4$ product strikes as well as the extra reduction in the second extraction step caused much greater amounts of Np^{239} to reach the Concentration Building than is normally the case. In addition, ruthenium appeared to be less well decontaminated.

These conclusions are based on the following evidences: (1) the 17-4-P solution arriving in C-4 gave an off-scale Beckman reading of greater than 100×10^{-12} amps; (2) a gross gamma decontamination factor of only 2 was obtained in the Cell A $BiPO_4$ by-product precipitation (Zr and Cb removal); (3) a gross decontamination factor of only 10 was obtained in the LaF_3 by-product precipitation, indicating that the increased activity was not due to larger amounts of La or rare earths; (4) a sample of the E-1-R, when evarporated with perchloric acid lost approximately one-half of its activity, roughly indicating the presence of a corresponding amount of Ru; and (5) the shipping container produced the highest radiation measurements thus far obtained.

The shipping container was a 15-gal. stainless steel drum. It was placed inside a regular PR-can outer jacket and shielded with 3/4 inch of lead on the sides and 1-inch of lead on the top and bottom, the annular space being filled with sand. The whole was enclosed in a wooden crate.

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The product loss, yield data, and shipping container Beckman readings are as follows:

Np^{237} Recovery Run Data (B-5-08-IP)

% Product Yield*				% Product Loss *				
17-4-P	E-1-R	F-1-PS	F-10-P	A-4-BP	D-4-BP	E-3-WS	F-7-WS	F-9-WS
91.5	101.4	466	752	0.71	1.28	3.11	4.8	4.3

* Based on 8-3-WS T.C. of B-5-08-B-7 (1.41×10^{11} c/m)

Np^{237} Shipping Container Beckman Readings (mr/hr)

Position	Bare Container	Through Pb Shield	Through Pb Shield + Sand
Top	83	--	--
Side	265	10	8
Bottom	370	4	--

Isolation Building (231)

Oxalate Method of Treating Recycles

Under Production Test 231-6-SE the oxalate method of treating recycles has been used on two plant runs, B-5-08-B-5 and T-5-08-D-9. Since the large catch tank CT-1 has no decant line, it was necessary to use precipitator P-1. This required splitting each charge in three portions to avoid exceeding the capacity of P-1. Three test "killings" were thus made on each run and the final cake solutions combined in one recycle container per run. Total losses on B-5 were greater than 6% of the material available for processing; the loss on T-9 was 10.6% on the same basis. Data are as follows:

Run	Fraction	"Solubility" in 1st Supernate	"Solubility" in 2nd Supernate	Total % Sewered via F-2 Tank	Total % Recycled to 224
B-5	1	ca. 7%	0.5%	15%	85%
B-5	2	4.7	0.05	5*	95
B-5	3	0.1	10	3.5*	96.5
T-9	1	4	1.4	4	96
T-9	2	0.2	1.6	11	89
T-9	3	0.2	9.2	15	85

* Estimated value; no sample was obtained.

The above data should be considered with some reservations, since some of the numbers are obviously in error. In the first place the high oxalate concentration of the waste solutions resulted in the appearance of abnormally large amounts of solids in the plates which checked poorly when counted. This was overcome in the later samples by solvent extraction. The second, and more serious, difficulty comes from the sampling equipment which is satisfactory for sampling homogeneous solutions but not for solutions containing suspended solids. To aid in overcoming the second difficulty, arrangements have been made to take 100-ml. bulk samples from the three wastes of the next run (T-5-08-D-13) after they are sent to F-2. Small and representative aliquots can then be taken from the 100 ml. in the control laboratory.

One operational difficulty appeared in these runs which had not been found in the dummy runs. Visual observation of the two important "end-points" was sometimes impossible due to fumes and fog on the sight glass and in the air over the solution. The first end-point, destruction of all H_2O_2 with $NaNO_2$, usually has been indicated by a clearing up of the solution. This was not noted on the production test runs and a large excess (that specified as maximum) of 40% $NaNO_2$ was added. This resulted in heavy red NO_2 fumes in the tank and rather vigorous effervescence of the solution. Apparently the plant waste solutions were considerably lower in H_2O_2 than the dummy run solutions; this could be due to the time interval between the first peroxide precipitation and the "killing" of the combined wastes. These fumes lingered in the tank and obscured the second end-point, conversion of the white color of the precipitated $La_2(C_2O_4)_3$ to tan as $Fe(OH)_3$ precipitated with excess KOH. It was later found that the latter end-point could be seen if a current of air were allowed to pass in the sight glass spray line and out the tank vent. The end-point was not as obvious as on the dummy runs, however, because the iron content of the plant solutions was apparently considerably less than that in the dummy runs.

Another difference between the production test runs and the dummy runs was the amount of $KMnO_4$ required to destroy the oxalate in the final $La_2(C_2O_4)_3$ cake solution. The dummy runs required about 400 grams of $KMnO_4$ per 1/3 charge, an amount very close to the theoretical requirement. The production runs have required less than 200 grams of $KMnO_4$. This resulted in a large excess of $KMnO_4$ in the first few production runs since the addition of 310 grams of solid $KMnO_4$ was specified to precede the final titration with 4% $KMnO_4$. Part of this difference may be accounted for by the loss of lanthanum occurring in Cells E and F of the Concentration Building and in the waste decanting during the oxalate precipitation at the Isolation (231) Building. This could be as much as 12 to 15%, but could hardly be 50% or more. A second theory to account for the low $KMnO_4$ requirement assumes that a large fraction of the $La_2(C_2O_4)_3$ is metathesized to $La(OH)_3$ by the excess KOH in the solution. This seems unlikely in view of the fact that laboratory tests with active wastes, as well as the plant scale dummy runs, showed no evidence of such metathesis. The fact remains, however, that the solid $KMnO_4$ addition, cut from 310 to 250 and then to 200 grams with successive runs, has always resulted in overrunning the end-point.

Two changes are being made in the procedure for Run T-5-08-D-13 now in progress. First, the entire charge will be treated with 40% $NaNO_2$ to destroy H_2O_2 in CT-1 before splitting to three portions for oxalate precipitation. This makes possible the use of the potentiometric titration equipment to locate the end-point and avoid a large excess of $NaNO_2$. The second change is to add no solid $KMnO_4$ to the final oxalate cake solution but titrate all the way with 4% $KMnO_4$. This may result in a final volume a bit too large to go into one 224-231 recycle can, but will give definite evidence of the amount of oxalate present.

Following completion of Run T-13, it is planned to equip CT-1 in one cell with an adjustable decant line leading directly to F-2 by steam jet. Additional production test runs can then be made under conditions more favorable to the successful demonstration of the method. Since the new decant line will be installed in a spare 1-inch opening in the top of the tank instead of in the 2-inch opening now occupied by the potentiometric titration electrode assembly, the latter equipment will not have to be removed. It can therefore be used for the H_2O_2 destruction with $NaNO_2$, either in the new oxalate method if adopted or in the present routine nitrite method if the other gives losses that are too high.

Cleanout No. 25 in Cell 4

A "disappearance" of about 77 units of Run B-5-08-B-4 between P-1 and the sample

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cans, followed by a similar "loss" of about 80 units of run B-5-08-B-5 between AT and the cans, led to a HNO_3 flush of ST-2, P-2, PR-3, and S-1, with evaporation in the latter. The solution when sent to AT and sample cans was found to contain about 171 units. This restored the Cell 4 material balance to essentially 100% on the P-1 basis.

Process Chemistry

Assay of Films Deposited on Slugs in the 105 Building

Decay curves are still being followed on the activities in film obtained from slugs discharged from 100 D Pile. Final data are not yet available. Spectrographic analyses have been obtained on the oil emulsion (with solids removed), the solids in the oil emulsion, and the aqueous phase (the solids dissolved). The data are tabulated below. There is no apparent explanation for the comparative high values of lead, barium and zinc in the aqueous phase.

Element	Oil Emulsion	Solids	Aqueous Phase
Al	less than 50	less than 50	1000
Ba	less than 5	less than 5	1000
Ca	100	10	greater than 1000
Cr	10	20	500
Fe	less than 10	50	1000
K	100	Less than 100	greater than 1000
Mg	20	less than 5	1000
Mn	less than 10	less than 10	10
Na	100	less than 10	greater than 1000
Pb	less than 50	less than 50	greater than 1000
Si	100	less than 100	100
Zn	less than 200	less than 200	greater than 1000

Waste Disposal Studies

Previously reported studies indicated that the alpha activity remaining in the supernatant solutions from decontamination cycle waste storage tanks could be decreased by lowering the pH of the solution. To determine whether the alpha activity could be precipitated from these solutions without lowering the pH, lanthanum equivalent to 200 mg/l was added to a sample of supernatant solution from the first decontamination cycle waste storage tank (107-West). At a pH of 10 a lanthanum compound precipitated but the alpha and beta activity was not decreased appreciably. At a pH of 9.4 (acid added along with the lanthanum) the amount of activity remaining in the supernatant was decreased somewhat but no more than would have been caused by lowering the pH without addition of lanthanum.

M. F. Acken per JWP
M.F.Acken, Chief Supervisor
Sep.Eng.Div. - 200 Areas Tech.

MFA:jd

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#1 R.M.Evans
#2 R.M.Evans
#3 W.O.Simon for The Area Engineer
#4 W.O.Simon for The Area Engineer
#5 W.O.Simon for The Area Engineer
#6 W.O.Simon for The Area Engineer
Attention: Patent Group
#7 N. Hilberry
#8 N. Hilberry
#9 N. Hilberry
#10 W.O.Simon-B.H.Mackey-700 Area File
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#15 Yellow Copy
#16 M.F.Acken
#17 W.C.Kay - J.E.Cole
#18 R.S.Apple
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August 24 Through August 30

Canyon Buildings (221)

Acid Wash (T)

An acid wash (T-5-08-AW-1) has been made in T Canyon, following the same procedure used in the recent acid wash in B Canyon and the June acid wash in both Canyons, with the inclusion of a greater number of samples to permit better localization of the point of product pickup. The major portion of the small amount of product recovered was found in the crude product storage tank (12-6), and represents approximately 1% of the preceding 8-4-P product solution, or approximately the normal amount of heel left below the jet suction leg in the large storage tank. The sampling points and product found, based on normal run, are tabulated below:

T-5-08-AW-1	Sample Description	% of Normal Run
8-1-P	Wash acid in extraction (8-1) precipitator	0.54
8-4-P-1	Wash acid after transfer through 8-1 and 8-2 centrifuge	0.88
8-4-P-2	Wash acid after agitation in solution (8-4) tank	0.86*
12-6-P	Wash acid after agitation in 12-6 intermediate crude product storage tank	3.48*
12-7-P	Wash acid after agitation in oxidation tank (12-7)	3.37*
13-1-P	Dissolving acid after circulation in 1st by-product precipitator (13-1)	0.40
13-4-BP	Regular waste on AW run	0.55
13-3-P	Product sample on AW run	3.44*
14-1-P	Wash acid in 1st product precipitator (14-1)	0.53
14-4-P-1	Wash acid after transfer through 14-1 precipitator and 14-2 centrifuge	0.44
14-4-P-2	Wash acid after agitation in solution tank (14-4)	0.41
14-4-P-3	Regular sample on AW	3.72*
14-3-WS	Regular sample on AW	0.048
16-1-P	Dissolving acid after circulation in 16-1 precipitator	0.015
16-4-BP	Regular sample on AW	0.15
16-3-P	Product sample on AW	3.85*
17-1-P-1	Wash acid in 17-1 precipitator	0.28
17-4-P-1	Wash acid after transfer through 17-1 precipitator and 17-2 centrifuge	0.44

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T-5-08-AW-1	Sample Description	% of Normal Run
17-4-P-2	Wash acid after agitation in solution tank (17-4)	0.43
17-4-P-3	Regular sample on AW	4.80*
17-3-W	Regular sample on AW	0.054

* Cumulative values

Special Product Precipitator Tank Cleanout (T)

Production Test SE-221-T-PA-8 provided for a special acid wash of the equipment in Sections 14 and 17 (1st and 2nd cycle product steps). The sampling points and product found based on preceding runs, are tabulated below:

Sampling Point	Sample Description	% of Preceding Runs Run D-23	Run D-24
14-1-A-1	Dilute dissolving acid after agitation in the precipitator preceding the run	8.1*	1.07*
14-4-A	Dilute dissolving acid after transfer to 14-4 via 14-2 and agitated in 14-4.	1.57	0.48
14-1-A-2	Dissolving acid after recirculation in precipitator to dissolve slurry heel left after transfer to centrifuge	11.41	6.4
14-4-P	Regular product Sample	92.0	95.0
17-1-A-1	Same as the 14 samples	2.72*	0.28
17-4-A	Same as the 14 samples	0.65	0.32
17-1-A-2	Same as the 14 samples	6.50	5.50
17-4-P	Same as the 14 samples	95.0	96.0

* The high results obtained on the dissolving acid samples after the initial agitation and circulation in the precipitator are attributable to failure to circulate the acid through the sampler (which is not normally used) for long enough to effect complete dissolution of product carrying precipitate deposited on the interior of the sample riser pipe during previous precipitations. Resamples of the acid in the solution tank before agitation support this theory.

The most significant data in the above tabulation are the 14-4-A and 17-4-A product analyses. These results show that in the first cycle product step (Section 14) the accumulated product picked up by the wash acid in the precipitator, centrifuge, and solution tank totaled less than 2% of the starting material in a standard size batch. In the second cycle product step (Section 17), the same acid washing treatment in the corresponding equipment resulted in a pick-up of accumulated product of less than 1% of a standard batch. These data constitute good evidence that current measures designed to minimize product hold-up in Sections 14 and 17 are proving very effective.

The product content of samples 14-1-A-2 and 17-1-A-2 shows that the normal slurry heel left in the precipitators in Sections 14 and 17 after jetting the charge to the centrifuge is probably somewhat less than the 15% which had been anticipated on the basis of Semi-Works results.

Equipment Performance (B)

The extraction precipitator (8-1) agitator continues to be a source of diffi-

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culty, with two more failures during the past week. After the failure on run B-5-08-B-13, the digestion temperature was lowered from 85°C to 75°C on runs B-13, 14, and 15 to determine if temperature was contributing to the agitator failures. As a result of the increase in extraction waste losses (8-3-W) from 0.8% (avg. of 5 previous runs) to 1.13% the digestion temperature was again raised to 85°C. On Run B-5-08-B-19 the agitator again failed. After greasing the lower bearing operation was resumed and the temperature was again dropped to 75°C. On Run B-5-08-B-21 another failure occurred and the agitator was removed and replaced by a new agitator. The use of 75°C maximum temperature during pre-treatment and digestion will be continued in an attempt to establish definitely the effect of temperature on the agitator performance and waste losses.

The agitator failure on Run B-5-08-B-21 occurred during the precipitation digestion, and the digestion was completed with sparger agitation. The charge was cooled and the cell blocks removed preparatory to replacing the agitator. This resulted in a moderate increase in the contamination of the canyon walls and crane cab. (A reference point which gave a reading of 20 mrep/hr after the 08-B-3 contamination, and which had decayed to 1 mrep/hr just prior to removing the cell 8-L cover blocks was increased to 8 mrep/hr.).

Miscellaneous

Technical Manual - Work has been continued on the preparation of drawings and final proofing and assembly of the first draft of Chapters 4, 5, and 6.

Concentration Buildings (224)

LaF$_3$ By-Product

LaF$_3$ by-product waste losses (D-4-BP) are in line with the previously reported averages except for Run T-5-08-D-21. The D-4-BP loss on this run was 1.44% (no recycle added). The high loss was believed to be caused by the accidental addition of a greater than normal amount of HF(estimated at 75 lbs. instead of the usual 53 lbs.) to D-1 as the oxidized charge was being cooled from 75°C to 35°C. The inadvertent addition was caused by incomplete closing of the D-1 HF inlet valve as HF was being added to E-1 for another charge.

LaF$_3$ Product Waste Losses

Beginning with Run B-5-08-B-10 and T-5-08-D-22, the LaF$_3$ product waste (E-3-WS) is being discarded on the basis of the assay of a sample taken at the end of the second centrifugation skimming, prior to cake wash and removal. No significant differences from the regular sample losses previously obtained have been observed. Sampling at this point is intended to save approximately 1-1/2 hours on the Cell E time cycle, as compared to the previous practice of waiting for a final waste sample assay after cake removal.

Cell E Time Cycle

Although recent Cell F jet stoppages and F-1-PS assay checks have caused considerable abnormal process delays in Cell E at T Area, "corrected" time cycles for the LaF$_3$ product precipitation are averaging 17 hrs. 30 minutes. A summary of recent time cycles is shown below:

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Cell E Time Cycles

Run No.	Actual Over-all Time		Abnormal Process Delays			"Corrected" Time	
	Hours	Minutes	Hrs.	Min.	Reason	Hours	Minutes
T-5-08-D-10	18	25	--	--	--	18	25
T-5-08-D-11	23	40	4	30	Waiting for E-3-WS-2 Assay	19	10
T-5-08-D-12	18	30	0	30	1-hr. spent in taking E-3-WS-2 sample	18	0
T-5-08-D-13	18	15	1	0	Waiting for E-3-WS-2 Assay	17	15
T-5-08-D-14	20	10	0	50	Delay in jetting E-3 to E-1		
			0	20	Delay in adding oxalic acid	19	0
T-5-08-D-15	18	10	--	--	--	18	10
T-5-08-D-16	22	15	4	30	Waiting for F-1-PS Assay	17	45
T-5-08-D-17	24	50	7	10	Plugged jet in Cell F	17	40
T-5-08-D-18	18	20	0	40	Waiting for F-1-PS Assay	17	40
T-5-08-D-19	17	40	0	30	Delay in starting cake removal	17	10
T-5-08-D-20	18	40	0	50	Extra E-2 Cake Removal Step	17	50
T-5-08-D-21	31	10	14	10	Plugged F-2 to F-1 jet in Cell F	17	0
T-5-08-D-22	18	55	2	0	Waiting for F-1-PS Assay	16	55
T-5-08-D-23	17	30	0	20	Waiting for E-3-WS Assay	17	10

LaF_3 Product Cake Removal

Considerable difficulty is still being experienced at T Area in effecting complete LaF_3 product cake removal from E-2 centrifuge, as indicated by the inability to reduce the E-2 Beckman meter readings below 20×10^{-14} amps. At B Area, since the new bowl sprays were installed at a 15° angle upstream (against the direction of the bowl rotation), as compared to the former installation at approximately 30°, the E-2 Beckman readings have been reduced to and are being maintained at $3\text{-}6 \times 10^{-14}$ amps. Following the acid wash run (-EAW) at T Area, the E-2 bowl sprays are to be replaced with a set (taken from B-2) installed at a 15° angle.

Recycle Runs

During the present shutdown of Building 224-T, scheduled for maintenance duties, the E-4 recycle inventory is going to exceed storage facilities, since 16 charges will be returned from Building 231 during shutdown. This is caused by (1) occasional falling behind on recycle disposal at T Area during the past month because of certain of the production runs arriving at Bldg. 224 with too high a product assay to permit recycling, and (2) the staggering of the T and B Area shutdowns in such a way that not enough recycle was available during the B Area shutdown to permit recycling into every T Area run, and the steady stream of recycles originating from B-Area while T Area is shutdown.

In order to gain control over the present recycle pile-up, a master recycle run is to be made (T-5-08-R-1) at 60% start-up volume. This run will contain 29 lbs. of lanthanum and will eliminate 3000 lbs. of recycle solution. It will include the E-4 inventory remaining from the partial recycles just completed plus (a) Run B-5-08-B-13, (b) T-5-08-D-21, and (c) B-5-08-B-14-special E-4-RC's, as well as (d) the T-5-08-EAW F-10-P solution. It may contain as much as 250 product units.

The E-4 storage tank will be cleared by jetting the above mixture to D-1 and storing it there until the next start-up, thereby making E-4 available for further storage.

Equipment Notes

The F-2 to F-1 jet at T Area (10 gal./min.) became severely plugged recently and could not be cleared. After its replacement with a new 20 gal./min. jet, it was found to be plugged with a small piece of the GX plow wiper blades from the F-2 centrifuge.

The entire Cell F floor at T Area has been repainted with Amercoat after extensive scrubbing to remove product contamination hazards created by recent maintenance work in the cell.

Isolation Building (231)

Oxalate Method of Treating Recycles

In addition to the two plant runs reported last week, one further plant test of the proposed oxalate method has been completed. In this run, T-5-08-D-13, the entire charge was treated with 40% $NaNO_2$ in the large catch tank (CT-1) to destroy H_2O_2, using the potentiometric titration equipment to locate the end-point. The solution was then treated in three portions in the precipitator (P-1) as with the two previous runs, except all $KMnO_4$ was added as a 4% solution instead of partially as a solid. The former change avoided the heavy fuming that resulted in the two previous runs from adding a large excess of $NaNO_2$. The latter change was made to avoid over-running the $KMnO_4$ end-point with the initial addition of solid as was previously done. Data for the run follow:

Fraction	"Solubility" in 1st Supernate	"Solubility" In 2nd Supernate	Total % Sewered	Total % Recycled
1	7%	0.3%	10.5%	90%
2	2	0.2	24	76
3	No Sample	7	15	85

The results of all three runs are summarized in the following table:

Run	Fraction	Sample	% of Re-Cycle
B-5-08-B-5	1	Waste	15
		Recycle	85
	2	Waste	?
		Recycle	?
	3	Waste	?
		Recycle	?
T-5-08-D-9	1	Waste	4.3
		Recycle	96
	2	Waste	11
		Recycle	89
	3	Waste	15
		Recycle	85
T-5-08-D-13	1	Waste	10.5
		Recycle	90
	2	Waste	24
		Recycle	76
	3	Waste	15
		Recycle	85
Total*		Waste	14
Total*		Recycle	86

* Excluding Fractions 2 and 3 of B-5-08-B-5

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The above total waste loss; 14%, is higher than expected or desirable. The "solubility" measurements on samples of the supernate before decanting would predict considerably lower losses. It, therefore, seems possible that a considerable fraction of the loss has resulted from drawing part of the settled cake off with the supernate. If this is the source of the trouble, losses should only be about one-third as large in a full scale run in CT-1 as in these 1/3 scale runs in P-1, since two instead of six decantings would be made per run. On this basis plans have been drawn up and a project submitted to equip CT-1 in one cell with a decant line leading to F-2. Full scale test runs will then be made to provide better data on which to base a decision regarding the possibility of adopting the oxalate method for routine use.

Acidity of $La(NO_3)_3$ Solution from Concentration (224) Building

In spite of repeated upward adjustment of the weight of HNO_3 used in the Concentration Building for preparing the $La(NO_3)_3$ solution, the acidity is currently running low. Although an acid normality of 2.0 is desired and adjustment is made if the normality is outside the range of 1.8 to 2.2N, the last 9 runs from T Plant averaged 1.75N with 6 requiring adjustment while the last 6 runs from B Plant averaged 1.64N with all 6 runs requiring adjustment. The previous 11 runs from T had averaged 1.99N with only 1 below 1.8 while the previous 7 runs from B had averaged 1.81N with 3 runs below 1.8. Another increase in Cell F HNO_3 seems indicated.

Process Chemistry

Film Formation and 100 Area Water Activity

A new sample of the aqueous phase of the oil emulsion, after the solids had been removed, was submitted for spectrographic analysis with the following results:

Element	Concentration (mg/l)
Na	30
K	10
Ca	4
Zn	1
Mg	1
Al	0.5
Si	0.5
Fe	0.3
Cr	0.3
Mn	0.05
Ba	0.05
Sr	0.03

All other elements were reported as less than a certain value or not reported at all. Further studies of activity in the film will be made.

Waste Disposal

Tests were made to determine the feasibility of precipitating bismuth as a sulfide from the 107 and 110 Tank solutions with the hope that the bismuth sulfide precipitate would also remove the alpha activity. In one experiment hydrogen sulfide was passed into the 107-W solution, then sodium hydroxide was added to correct for the change in pH caused by the hydrogen sulfide. The original pH of the waste solution was 10 and after treatment with hydrogen sulfide and sodium hydroxide 9.5. In the second experiment, hydrogen sulfide was first passed into 6N sodium hydroxide and this mixture was then added to the waste solution giving a final pH of 10. In both cases bismuth sulfide precipitated and settled satisfactorily leaving less then 2mg of bismuth per liter solution. However, in neither experiment did the precipitate carry the alpha activity.

Comparative Evaluation of the Standard, Fluomolybdate, and Fluosilicate Processes

Comparative decontamination runs were carried through two $BiPO_4$ cycles. A small amount of extraction precipitate solution (8-4-T) furnished the activity which was added to a dummy solution. Since extraction decontamination is no longer measured in the plant, calculations for the laboratory runs are based on a probable figure of 10, although an extraction decontamination factor of 12 would bring the results on the standard process run in line with plant data. There was not enough activity present to carry the runs through the cross-over. The product loss in the product precipitation steps in the fluomolybdate runs was slightly higher than in the other two cases (approximately 1% as compared to 0.4%). Since this does not agree with previous laboratory and Semi Works data, runs will be made at full product level to establish this point more firmly. The fluomolybdate process showed a 5 to 6-fold improvement over the fluosilicate process in one cycle and was one-half as effective as the Ce-Zr scavenger process. At the end of two cycles, however, all three processes showed better than 10^5 decontamination.

	Standard Process	Fluosilicate Proc.	Fluomolybdate Proc.
Extraction + one cycle	3800	323	1890
Extraction + two cycles	$>2x10^5$	$>2x10^5$	$>2x10^5$

High Activity in the E-1-R Solution of Run IP-2

The half-life of the activity in solution E-1-R, as measured over a period of 7 days was 60 to 75 days. The half-life of the activity remaining after evaporation of perchloric acid, as measured over the same period, was approximately 40 days. Some support was thereby given to the contention that the major offending activity was Ru.

Identification of Fission Isotopes via Mass Spectrograph

The recently constructed scanner for locating radio-isotopes on mass spectrograph plates was tested. Two of the lines of activity (consisting of the 275-day Ce isotope) separated by a distance of 1.9 mm were located accurately. The reported counts/minute decreased at the mid-point between the two lines of activity by a factor of 3 to 4 with respect to the maximum counting rate recorded. Radio-Isotopes having less energetic beta particles than the 3.1 Mev radiation associated with the 17.5-minute praseodymium daughter activity should be more readily located because the ratio (maximum activity/background activity) should be even greater. The operation of this apparatus should be entirely satisfactory for all measurements being considered since there will be a distance of 2 to 4 mm. between each mass number depending upon the mass number.

Separation of the 2-3 Year Element 61 Isotope

The first elution of rare earth activity from the Amberlite IR-100 adsorption column with citric acid-ammonium citrate buffer solution (pH 2.75-2.83) was completed. A total of 50 fractions with a total volume of 2400 ml. was collected. The activity-clution curve showed two peaks; the first due to 57 day Y^{91} isotope and the second, due to the 300 day + 28 day Ce isotope. In the valley between, the soft beta-ray emitting element 61 isotope appeared to be concentrated. Fractions 25-40 will be treated further to concentrate the element 61 activity.

M F Acken
M.F.Acken, Chief Supervisor
Sep.Eng.Div. - 200 Areas Tech. Dept.

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#1 R.M.Evans
#2 R.M.Evans
#3 W.O.Simon for The Area Engineer
#4 W.O.Simon for The Area Engineer
#5 W.O.Simon for The Area Engineer
#6 W.O.Simon for The Area Engineer
Attention: Patent Group
#7 N. Hilberry
#8 N. Hilberry
#9 N. Hilberry
#10 W.O.Simon-B.H.Mackey-700 Area
#11 M.H.Smith
#12 L.Squires
#13 300 Area File
#14 Pink Copy
#15 Yellow Copy
#16 M.F.Acken
#17 W.C.Kay - J.E.Cole
#18 R.S.Apple
#19 R.H.Beaton
#20 W.H.Sullivan
#21 G.W.Struthers
#22 J.D.Ellett
#23 J.J.Urban
#24 F.B.Vaughan

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Canyon Buildings (221)

Extraction (T)

The use of Section 6 for the pre-extraction treatment and assay of metal solution to control batch size and reduce the Section 8 cycle time was initiated on Run T-5-08-D-25. The average extraction waste loss (1.5%) on the five runs using this procedure has increased somewhat over those of the preceding five runs (1.2%). There is no definite evidence that this increase is due to the change in the point of pre-treatment, and this variation is within the allowable limit of analytical accuracy. The time required to perform the pre-treatment in Section 6 is approximately 12 hours, of which 4-6 hours is due to analysis time. The effect of this change on the Section 8 time cycle is to reduce it by about 1 hour giving an operable Section 8 time of 16-1/2 hours.

Use of the Section 6 assay for determining batch size and as the run basis has been rendered questionable as a result of an unexplained discrepancy between the 6-1-MR and 8-1-MR analyses. A comparison of the two analyses is given:

Run No.	8-1-MR Assay (Using 6-1-MR Assay as 100%)	Variation %	% Overall Jet Dilution 6-1 to 6-3 to 8-1
08-D-25	95.0	5.0	1.04
08-D-26	96.4	3.6	3.4
09-D-1	94.8	5.7	5.7
09-D-2	92.5	7.5	4.0
09-D-3	96.3	3.7	7.6
09-D-4	99.5	0.5	5.7
09-D-5	97.2	2.8	3.2

No leaks were found in Section 6; cell drains showed no increase in activity. The variation is attributed to error in the measured volumes as indicated by the weight factor readings of the various tanks and to some extent is substantiated by the low and erratic jetting increases obtained. The normal jetting increase is approximately 3% per transfer in transferring water and approximately 5.5% per transfer for metal solution. On the basis of subsequent product assays, the 8-1 assay gives the most reasonable material balances and has been used on the above

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runs. Both samples will be taken until the sources of the variation are determined.

Sampling (T and B)

Effective with Run T-5-08-D-25 the metal waste (8-3-W) is sampled after the precipitator wash, following shutdown of the centrifuge. This eliminates the possible contamination hazard of active material escaped via the sampler return line due to windage from the centrifuge. The latter sample point has been used in B Area since the adoption of the 8-3-W sample.

Concentration Buildings (224)

$BiPO_4$ By-Product

Recently, the $BiPO_4$ by-product waste losses in Concentration (A-4-BP) have increased at B Area. The increase was abrupt and began with Run B-5-08-B-17. Careful examinations of the Concentration Building procedure records and inspections of oxidation reagent analyses have revealed no likely cause in the Concentration Building. Both the 17-4-P and A-4-BP solution samples have been clear. A further check at the Canyon (221) Building, however, revealed that a shift from the use of Baker's C.P. ferrous ammonium sulfate to the less pure Metalloy salt was made in the reduction steps at approximately the same time. Reagent make-up records are not sufficiently detailed to provide a correlation for this period, but a close check will be made to determine whether the change in source of material is significant.

The previous A-4-BP average at B Area was 0.32%. The T Area average is being maintained at 0.31%. The higher waste loss runs at B Area are listed below:

$BiPO_4$ By-Product Waste Losses (A-4-BP)

Run No.	% A-4-BP
B-5-08-B-17	2.1
B-5-08-B-18	0.46
B-5-08-B-19	0.43
B-5-08-B-20	0.29
B-5-08-B-21	0.40
B-5-08-B-22	0.49

LaF_3 By-Product

Exluding the "master" recycle run at T Area, the LaF_3 by-product waste losses (D-4-BP) have averaged 0.56% at T Area and 0.63% at B Area. A double-batch slurry wash is still being used in all runs at T Area, and a displacement wash is used at B Area.

LaF_3 Product Waste Losses

LaF_3 product waste (E-3-WS) sampling at the end of the second centrifugation skimming has been used for final waste assay on Runs T-5-08-D-22 to D-24 and B-5-08-B-10 to B-22 inclusive thus far. The average waste loss at B Area has been 1.16% and at T Area 0.68% (corrected to a "performance" basis for recycling). At B Area, this loss remains unchanged from a final waste previously sampled after cake removal, averaging 1.15%. At T Area, the previous average was 0.80%, showing a difference of approximately 0.1% for the new sample.

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LaF$_3$ Product Cake Removal

Since the installation at B Area, of the new E-2 centrifuge bowl spray at a 15° angle (against the rotation) no difficulties in LaF$_3$ product cake removal have been encountered. The involved method of cake removal, described in the report of August 10, 1945 as "Method A", was abandoned on Run B-5-08-B-19 and the simpler T Area procedure, "Method B", is now being used. Thus far, no increase in E-2 Beckman meter readings has been observed.

Following an acid wash clean-out of E-2 at T Area (after Run T-5-08-D-24), the bowl spray angle was measured to be 26° "upstream". The spray was replaced with another set 15° upstream, as in the B Area. The flange bolt holes on this new spray were slotted to permit future adjustment over a 22° range, if necessary. Thus far, there have been no runs processed through Cell E since the spray change.

Recent E-2 Beckman meter readings are shown below:

Centrifuge E-2 Beckman Meter Readings ($\times 10^{-14}$ amps)

Run No.	Start (Empty)	Full	During Cake Removal Cycle (1) After 700# Spray	(2) After 250# Slurry	(3) After 200# Slurry	(4) After 150# Spray	Cake Removal Procedure (See Report of 8-10-45)
B-5-08-B-17	3	70	9	3	7	7	Method A
B-5-08-B-18	2	100	7	7	9	9	Method A
B-5-08-B-19	4	80	7	7	9	9	Method B
B-5-08-B-20	4	73	14	13	8	8	Method B - Note (a)
B-5-08-B-21	1	51	5	4	4	4	Method B
T-5-08-D-24	22	75	22	--	20	18[b]	Method B - Note (c)
T-5-08-EAW Rework	5	12	6	--	5	5[b]	Method B
T-5-08-AW-1	5	10	6	--	5	5[b]	Method B

Notes - (a) Acid wash of E-2 with 1000 lbs. of 1N HNO$_3$ (0.5% 8-1-M product) carried out after Run B-5-08-B-20 to replace broken skimmer. 1500 lbs. of water used in calibrating new E-2 skimmer picked up 1.8% 8-1-M product.

(b) Beckman readings taken with E-2 rotating of 30 rev./min.

(c) Acid wash of E-2 carried out (Run T-5-08-EAW) after run D-24 to replace bowl spray.

T Area Acid Wash Runs

Following Run T-5-08-D-24, an acid wash run (T-5-08-AW-1) was carried through both the Canyon and Concentration Buildings. This run was processed in the Concentration Building as a full volume (100% start-up volume) run, but otherwise was carried through the normal process. The final product solution (F-10-P), containing 7.6% of a normal 8-1-M product content, was recycled to the E-4 tank.

Prior to the passage of this run through Cell E, a separate acid wash run (T-5-08-EAW) was started in E-2 centrifuge and carried through Cells E and F recovery. The wash of E-2 was carried out by slurrying 300-lb. portions of 60% HNO$_3$ through E-2 from the gallery until a total of 1550 lbs. of acid had been used. Following this, approximately 12,000 lbs. of water were sprayed through the centrifuge bowl, to overflow into the catch-tank containing the acid. The resultant

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normal volume of 1N HNO_3 solution was reworked by throwing down 2.5 lbs. of lanthanum in a single strike LaF_3 product precipitation.

The lowering of the E-2 Beckman meter readings and product pick-ups during the acid wash runs are shown in the tables below:

Centrifuge E-2 Beckman Meter Readings (Cell E Acid Wash)

Washing Steps	Start (Empty)	300# acid	600# acid	900# acid	1550# acid	12,000# water
Beckman($x10^{-14}$amps)	16.5	17	15.5	12.5	13	5 *

Note:* E-2 rotating at 30 RPM

T Area Acid Wash Runs (Concentration Building)

Process Vessel or Step	Sample Code	Cell E (T-5-08-EAW) Total %	Cell E (T-5-08-EAW) % Pick-Up	221-224(T-5-08-AW-1 Total %	221-224(T-5-08-AW-1 % Pick-Up
Basis		$2.05x10^{13}$		$2.05x10^{13}$	
Canyon (221) Building	17-4-P	--		4.8	--
C-4	C-4-0	--		4.4	-0.4
A-1	A-1-0	--		4.5	+0.1
$BiPO_4$ B.P. Loss	A-4-BP	--		0.03	--
A-3	A-3-OS	--		5.3	+0.8
D-1	D-1-0	--		5.1	-0.2
LaF_3 B.P. Loss	D-4-BP	--		0.09	--
D-3	D-3-OS	--		5.2	0
E-2	E-3-AW	9.1	+9.1	--	--
E-1	E-1-R	10.5	+1.4	5.1	-0.1
LaF_3 Prod. Waste	E-3-WS	0.28	--	0.06	--
F-1 (plus E-2 to F-1 line)	F-1-PS	20.7	+10.5	6.8	+1.8
15% KOH Metathesis loss	F-7-WS	0.17	--	0.04	--
2% KOH Metathesis loss	F-9-WS	0.07	--	0.04	--
Final Yield	F-10-P	12.7	-7.7	7.6	+0.9
Total			+13.3		+2.9

Note: % Product pick-up corrected for waste losses

Equipment

During Run B-5-08-B-20 at B Area, it was found impossible to skim E-2 centrifuge at the end of the first Cell E (LaF_3 product) centrifugation. The run was completed by using two displacement washes with 500 lbs. of 1N HNO_3, one from the precipitator and one from the gallery. The E-3-WS waste loss was increased from 1.23% to 1.37% by the double wash. An acid wash of E-2 with 1000 lbs. of 1N HNO_3 was carried out (jetting to E-4) and the centrifuge casing was opened up. The skimmer was found to be hanging by approximately one-half of the pipe wall at a jagged break in the bend of the vertical to horizontal curve of the pipe. It appeared that the seam of the welded pipe had split, thereby weakening the pipe sufficiently to cause it to bend around about 90° and tear. There was some evidence of corrosion pitting along the seam weld.

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A new skimmer has been installed and is now in use. The broken skimmer is being decontaminated and is to be given a metallographical study as to cause of failure.

Isolation Building (231)

Oxalate Method of Treating Recycles

No laboratory or plant tests have been made since last week. After the installation of the adjustable decant line from the large catch tank (CT-1) of Cell 4 to waste receiver F-2, it is planned to make three to five full scale production test runs.

Concentration of "30%" H_2O_2 Reagent

On several recent occasions it was found that the "minimum" quantity of $NaNO_2$ (calculated empirically for each run) for peroxide destruction in wastes to be recycled to the Concentration (224) Building was more than enough to destroy all peroxide. In such cases, a large excess of nitrite, and hence a large volume of $KMnO_4$ solution, would have been necessary on all these "killings"; and extra large volumes would have been recycled. This difficulty might have been due to a low peroxide concentration which in turn could have been caused by a low H_2O_2 concentration in the "Becco" reagent. Analysis of several carboys showed concentrations varying from 22 to 28 percent instead of the nominal 30%. Since the H_2O_2 concentrations used in both precipitations are at least twice that necessary for reasonable yields, it has not been thought necessary to increase the weight of "30%" H_2O_2 used above the present flowsheet amounts. Instead, carboys are being spot checked to prevent the use of material more dilute than 20%, and the minimum 20% $NaNO_2$ addition has been cut from 42% to 35% of the weight of solution to be treated.

Chemical Method of Analysis

Both P-1 (initial solution from 224 Building) and AT (final solution loaded to sample cans) solutions will be analyzed by the new chemical methods for all Runs after T-5-08-D-24 and B-5-08-B-20. This change will result in assays several percent lower than would be obtained by the radio-chemical methods formerly used. To compare 231 Building data with that from 221 and 224, a conversion factor is required. The new methods of analysis are believed to be inherently better.

Sample Cans

Sample can 145 was given a part load and intermiediate evaporation in Cell 6A. When preparing for final loading, it was found that the adapter plug had seized in the sample can threads. After much effort, the plug was finally loosened with a 4-foot lever, stripping the threads of both plug and can. It was necessary to dissolve the semi-solid product nitrate. This was done by adding 500 ml. of 1N HNO_3 and stirring the solution with a stainless steel wire loop agitator at room temperature for 4-1/2 hours. This treatment apparently sufficed to dissolve the material (80% of a full load); the can was essentially clean when the solution was drawn to the Still Receiver SR-1 in Cell 3 and two washes of 1N HNO_3 (500 ml. and 800 ml.) were used. The product was combined with that present in the holding tank ST-2 (from PR-3 overflows) and charged back to the still to be concentrated again and loaded out as Cleanout 27.

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Process Chemistry

Decontamination

Three fluomolybdate acids may possibly be used as a decontaminating aid in the product precipitation step. All of these acid solutions are quite stable and 0.8M solutions of all have remained crystal clear after 2-weeks storage in Lusteroid tubes. However, the acids are not stable in glass containers.

The corrosion of 25/12 Cb stainless steel in these three fluomolybdate acids was determined at room temperature. As can be seem from the data presented below, the MoF_3 and MoF_4 derivitives have low corrosion rates whereas the MoF_5 is corrosive above the tolerable limit.

Derivitive	Inches/Month Penetration
MoF_3	0.000041
MoF_4	0.00021
MoF_5	0.00056

The MoF_2 derivitive is soluble, but does not appear to be stable under process conditions, whereas the other three derivitives are stable. Studies are now being conducted to obtain decontamination and product carrying data with the MoF_3, MoF_4, and MoF_5 derivitives to determine which of them is most satisfactory for optimum process conditions.

Waste Disposal

Further use of sodium sulfide solution in treating the 110 Tank waste solution has not yielded improved results over those reported last week. Bismith was precipitated, but the alpha activity was not removed.

The effect of reducing pH of waste solutions with weak acids was studied further. Tests with carbon dioxide showed that it was quite effective in lowering the pH to about 7 and under these conditions most of the bismuth and alpha activity was precipitated from solution. The same result was also obtained with phosphoric acid. In both cases, the alpha activity remaining in the solution corresponded to less than one microgram/liter.

The buffering action of the solutions due to the sodium phosphate content would allow considerable latitude if it were desired to lower the pH of the solutions already in the tanks by the addition of acid. If, for example, phosphoric acid in twice the amount needed to neutralize the 107-E waste solution to a pH of 7 were added, the excess acid would further lower the pH only to 6.3. A similar effect was observed with other acids including nitric acid.

Soil Retention Studies

Eight gallons of the 110-W Waste Tank solution were obtained from the 241 Tank Farm. This solution will be passed through a column to study the retention of product, the total holdup, the first appearance of break-through, and the tenacity with which the product is held.

M F Acken per GEB

M.F.Acken, Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.Dept.

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#2 R.M.Evans
#3 W.O.Simon for The Area Engineer
#4 W.O.Simon for The Area Engineer
#5 W.O.Simon for The Area Engineer
#6 W.O.Simon for The Area Engineer
Attention: Patent Group
#7 N. Hilberry
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#9 N. Hilberry
#10 W.O.Simon - B.H.Mackey - 700 Area
#11 M.H.Smith
#12 L.Squires
#13 300 Area
#14 Pink Copy
#15 Yellow Copy
#16 M.F.Acken
#17 W.C.Kay - J.E.Cole
#18 R.S.Apple
#19 R.H.Beaton
#20 W.H.Sullivan
#21 G.W.Struthers
#22 J.D.Ellett - 700 File
#23 J.J.Urban
#24 F.B.Vaughan

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September 15, 1945

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September 7 Through September 14

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Canyon Buildings (221)

Dissolver Performance

The dissolving time cycle on Dissolver 3-5R in the T Canyon has increased appreciably over that on 4-5L indicating depletion of the metal heel. Accordingly, the weight of 60% nitric acid used per charge has been reduced from 5500 lbs. to 5300 lbs. In the B Canyon, the acid weights are 5200 lbs. and 5300 lbs. respectively for 3-5R and 4-5L.

Extraction Waste Losses

Over a period of several months operation using 20, 22, and 24% UNH processes the average extraction loss has approximated 0.65%. However, with the processing of more active metal (240 g/T) the reported extraction waste losses increased to 1.0% in the B Canyon and 1.3% in the T Canyon (10 and 14 runs respectively). With the return to lower activity metal the reported losses dropped to 0.61% in the B Canyon and 0.94% in the T Canyon (10 and 5 runs respectively). While excessively high beta backgrounds can influence the accuracy of alpha counting it is not believed, on the basis of present evidence, that difficulties of this nature are responsible for the waste loss variations noted above. Investigations of this possibility are being made, however. The higher extraction wastes reported for the T runs may be due to making the pre-extraction nitrite treatment in Section 6 several hours before the strike is made in Section 8. This possibility can be checked in the near future, as similar use of Section 6 in the B Canyon is being inaugurated with Run B-5-09-D-11, using sorted metal of lower activity.

Decontamination Cycles

The use of 24% bismuth solution instead of 14% solution for making the product strikes has been in effect in B Canyon for approximately 1-month without adverse effect. In the T Canyon this change has been in effect since Run T-5-09-D-4, with no increase in waste losses or cake handling difficulties.

The 1-hour reduction time following the addition of ferrous iron was eliminated in the T Canyon with Run T-5-09-D-1. The average product loss in the first and

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second cycle wastes has not been altered significantly by this change: first cycle - 0.45% before and after (10 runs); second cycle - 0.31% before, and 0.41% after (8 runs).

Concentration Buildings (224)

$BiPO_4$ By-Product

The $BiPO_4$ by-product waste losses (A-4-BP) have continued to average 0.43% at B Area (Runs -09-F-1 to F-7) as compared to 0.27% at T Area (Runs -08-D-25 to -09-F-7). No differences in area operation can be detected. The T Area is using Metalloy ferrous ammonium sulphate in the second cycle reduction step while B Area is using Baker's C.P. grade salt for the same operation. The high losses are being obtained in the area where the more pure salt is being used, so there does not appear to be any correlation between reagent "cruds" and Concentration Building waste losses at present.

Recycle Runs

Recently at T Area, it was necessary to make a separate or "master" recycle run starting in the Cell D LaF_3 by-product step, in order to bring the recycle solution inventory in Tank E-4 down during the T Area shutdown. This run (T-5-08-R1) was carried out on the normal 60% start-up volume scale and involved the precipitation of approximately 31 lbs. of lanthanum in the LaF_3 by-product step. A single displacement wash of the centrifuge (D-2) cake was carried out, with a skimming before and after the wash to a 40-gal. instead of the usual 10-gal. heel. The LaF_3 by-product waste loss (D-4-BP) for this large cake was no higher at 2.34% than could be predicted from past performances.

Lanthanum Purity

It has been discovered recently that the purity assay of the lanthanum ammonium nitrate salt used in the LaF_3 cycle increased several months ago from 23.0 to an average of 24.7% lanthanum (92 to 99% pure $La(NO_3)_3 \cdot 2N\ H_4NO_3 \cdot 4H_2O$ salt). For the weight of lanthanum salt currently used in the Cell E LaF_3 -product precipitations (16.5 lbs.), 4.05 lbs. of La have been used instead of the 3.75 lbs. formerly believed used. This means that actually 9.1 lbs. of La is being recycled to the Cell D LaF_3 by-product at T Area instead of the 8.5 lbs. intended.

Beginning with Run T-5-09-B-8, a new system of recycling calculations is being put into effect, based on the use of an actual La assay of the solution in the E-4 tank. This replaces the cumbersome charge-counting method that has been used to date. This method will call for the recycling of 9.0 lbs. of La to Cell D, in keeping with what has been found to be past practice, as explained above.

LaF_3 Product Cake Removal

After the change in the setting of the T Area centrifuge E-2 bowl spray to an angle 15° "upstream" from the perpendicular, the first run through the centrifuge was the low activity recycle run (T-5-08-R-1). No cake removal difficulties were experienced on this run. The next run through the bowl (T-5-08-D-25), however, produced final (empty) E-2 Beckman meter readings ranging from 5 to 49×10^{-14} amps. as the bowl was inched around to various positions and 23×10^{-14} amps at 30 rev./min. A second complete cake removal cycle was carried out (as Run D-25-A) and it was found that close control of the bowl speed at 10 rev./min. during the initial

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700-lb. water spray permitted the Beckman to be easily reduced to 4×10^{-14} amps. Since this run, a stripe has been painted on the centrifuge drive shaft and the 10 rev./min. speed is controlled by the operator counting the bowl revolutions through the window on the operating gallery. No difficulties have been observed in cake removal since then, up through Run T-5-09-D-5.

At B Area, considerable cake removal difficulties have developed recently. After approximately 10 runs had been successfully carried out with the new 15° angle spray and the extended slurrying method ("Method A" as described in the report of August 10), a shift to the simpler T Area method ("Method B") was made. After 3 equally successful runs, cake retention began to appear on the fourth and fifth runs. A return to "Method A" for a series of 3 more runs did not stop the continued building up of cake hold-up. Inspection of the Beckman records during the removal cycle, however, indicates that it is the failure of the initial 700-lb. water spray, which is the same in both procedures, that is the cause of incomplete cake removal, rather than the effects of more or less water slurrying in the two procedures.

An acid wash of E-2 was carried out after Run B-5-09-F-4, since both the E-2 Beckman readings and the F-1-PS product assays had indicated product hold-up in Runs F-1 to F-4 inclusive. This wash was carried out by successively slurrying two 250-lb. batches of 60% HNO_3 through E-2 to E-4 and following with a 250-lb. water spray. The initial Beckman reading of 25×10^{-14} amps. (at 30 rev./min.) was successively reduced to 18 to 14 to 12×10^{-14} in the three steps quoted. The product pick-up was 11.8%. At the end of Run B-5-08-F-5, approximately 4,000 lbs. of water was sprayed through E-2 to E-4 to dilute the high acid solution in E-4 to permit recycling and the E-2 Beckman was reduced from 13 to 5×10^{-14} amps. Product pick-up in E-4 was increased to 27.7%.

At the present time, Production Test No. SE-224-B-PA-2 is being carried out, after being started on Run B-5-09-F-5. This test substitutes a slurrying with 250 lbs. of 24% HNO_3 for the usual two slurryings with water between the spraying operations. This is being done in an effort to dissolve or loosen any densely plastered cake from the bowl wall. The KOH added to metathesis is being corrected to neutralize the extra acid jetted to F-1 by this operation. It is also necessary to carry out metathesis on a slightly larger volume scale, 260 instead of 250 gal.

Thus far, this test procedure has not effected any visible improvements in cake removal.

F-10-P (P-1) Acidities

Although the desired range of the final product solution acidities (F-10-P or P-1 solutions) is 1.8 to 2.2N, the use of 23 lbs. of 60% HNO_3 at B Area and 25 lbs. of HNO_3 at T Area for final cake solution in F-2 centrifuge has been producing acidities averaging less than 1.8N. The weight of acid used at both areas has been increased recently to 26 lbs., producing average acidities of 2.23N at T Area and 2.17N at B Area.

Run T-5-08-D-25-A

As outlined in the discussion of the E-2 cake removal problem, a second cake removal cycle was carried out on Run D-25 to form Run D-25-A. The small amount of lanthanum removed from E-2 in effecting the very nearly complete flushing was carried through a standard volume metathesis, with no additional lanthanum being added. Metathesis losses were normal, however, and the F-10-P solution was recycled to Tank E-4. Product losses and yields, based on the 8-1-MR product assay of the original charge (T-5-08-D-25) are shown below:

Recovery Run T-5-08-D-25-A Yield and Loss Data

Basis	8-1-MR of Run D-25	100
Pick-up from E-2	F-1-PS	16
F-7-WS	Loss	0.55
F-9-WS	Loss	0.28
Final Yield	16	22.4

Skimmer Failure

At B Area it was recently found impossible to skim the LaF_3 by-product centrifuge, D-2, during Run B-5-09-F-3. The run was completed by the use of a displacement wash of the cake in the bowl. Upon dismantling the centrifuge, the skimmer was found to be broken in much the same manner as was the E-2 skimmer, which broke the preceding week. The welded tubing had split for a distance of 2 inches along the weld, beginning at the base of the shaft, causing the pipe to fold back and tear in two places.

Isolation Building (231)

Oxalate Method of Treating Recycles

Three full scale production runs in Catch Tank CT-1 of Cell 4 (newly equipped with an adjustable decant line leading via a steam jet to Waste Receiver F-2) have been completed. Of the total product available for recycling, 4% was sent to waste and 96.4% recycled with a material balance of 100.4%. These results are sufficiently encouraging to warrant two additional test runs; if these are also satisfactory, routine test operation in one cell will be proposed in order to determine such factors as time cycles and process reproducibility before adopting the new method completely.

One difficulty encountered in the test runs has been longer time cycles than was expected. Some of the time has been lost while waiting for results of laboratory analyses. It is expected that this delay will be greatly reduced when suitable laboratory methods of analysis have become available. Other operational short cuts are expected to reduce the waste handling time cycle to 8-12 hours.

In the last of the three full scale test runs, TD-26, the final solution filled two sample cans. This resulted from the large $KMnO_4$ requirement to destroy oxalate which in turn was present in excess due to the large heel left after decanting. This heel, about 25 liters instead of the usual 10 to 13 liters, was large because the precipitate was more bulky than usual or settled poorly. The cause of this is not known, but may have resulted from too low an acidity after the initial KOH addition. The latter quantity had been arbitrarily set to reduce the acidity to about 0.5N in the dummy runs. It has recently been found that the initial acidity of plant supernates combined for treatment is about 1.1N, whereas the dummy run waste solutions were made up to be about 1.4N. Therefore, the quantity of KOH used in the dummy runs has been sufficient to reduce the acidity to 0.0 - 0.2N in the production test runs. This allows the possibility of poor-settling $La(OH)_3$ precipitation before the addition of oxalic acid and could account

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for the large cake volume found on Run TD-26. On future runs, the initial KOH addition will be reduced from 10.5 to 7.7 lbs. of 50% KOH.

The low $KMnO_4$ requirement of the final lanthanum oxalate solution from plant runs has been discussed in previous reports. This had been expressed as due to either loss of lanthanum or partial conversion of lanthanum oxalate to the hydroxide. The former possibility has been shown unlikely by obtaining lanthanum analyses on the initial and final solutions handled in the three production test runs.

For a more detailed picture of the distribution of product in the three production test runs, the following table is given:

Sample Description →	CT-1-W Initial waste		F-2S 1st Supernate		F-2-SW 2nd Supernate		CT-1-R Final Recycle		Mat. Bal.
↓ Run	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W
T-5-8-R-1*	100	3.1	4.1	.13	0.5	.016	87.5	2.7	92.1
T-5-8-D-25	100	2.6	4.5	.12	1.9	.05	98.8	2.56	105.2
T-5-8-D-26	100	3.2	0.7	.02	0.9	.03	103	3.3	104.6
Average	100	3.0	3.0	.09	1.0	.03	96.4	2.9	100.4

* 8-1-MR of T-5-8-D-25 used as basis

Process Chemistry

Waste Disposal

The possibility of using Amberlite IR-100 resin to remove activity from the 110 Waste Tank solutions was studied. The resin was used as the air dried sodium form and was packed in a 9-mm diameter, 11-cm. high column. The passage of the 110-E waste solution at pH 9.6 through the column at about 1 ml./min. resulted in very little decrease in either alpha or beta activity of the solution. The same result was obtained in a batch test with 1 g. of resin in contact with 10 ml. of the 110-E waste solution for 48 hours.

On the basis of these results, it would not appear feasible to attempt to decontaminate the waste solutions by using Amberlite IR-100. No advantage would be gained by using the resin in the hydrogen form since the waste solutions are approximately 2N in sodium ion. In addition, the change of pH by replacement of hydrogen with sodium might result in blinding of the adsorbent because of precipitation of bismuth hydroxide.

Fluomolybdate Process-Carrying of Product

The carrying of tracer product in the presence of $-MoF_3$, $-MoF_4$, and $-MoF_5$ derivitives was determined. The losses in all three cases were of the order of 1% indicating that one derivitive is no better than either of the others. At Hanford product concentration (25-mg. Pu/l) the $-MoF_4$ derivitive gave a loss also of approximately 1%. These results are less favorable than earlier laboratory and semi-works tracer experiments, thus casting considerable doubt on the advisability of carrying out plant scale trials.

Specific Activity of 265g/T Pu

The first series of measurements to compare the specific activity of 265g/T

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Pu with 2g/T Pu (Clinton Laboratories product) was completed. Eight discs of each were prepared in the usual manner and ignited at 750° for one-half hour. One Clinton disc was discarded because of mechanical difficulties with the platinum plate. The samples were weighed and counted for 1 hour each in random order in the vacuum counter. The results are tabulated below:

No.	Site	Spec. Activity x10^{-5} d/ug	No.	Site	Spec. Activity x10^{-5} d/ug
1	X	1.3748	9	W	1.4149
2	X	1.3835	10	W	1.4323
3	X	1.3805	11	W	1.3915
4	X	1.3791	12	W	1.4839
5	X	1.3583	13	W	1.4320
6	X	--	14	W	1.4543
7	X	1.3586	15	W	1.4505
8	X	1.3937	16	W	--
Average X		1.3755	Average W		1.4371

$$\frac{\text{Site W}}{\text{Site X}} = 1.045$$

The ratio 1.045 is lower than expected. The spread of the Site W results makes the ratio quite uncertain. Possible difficulties are (1) self absorption, (2) change of counting factor due to a bent disc, and (3) some impurity in the W solution.

Two more peroxide precipitations are being carried out on both the W and X solutions after which eight more discs will be prepared of each solution for weighing and counting.

Effect of the Presence of H_3PO_4 in UNH solutions

The amount of H_3PO_4 which could be tolerated in 60-70% UNH solution was determined. The 60-70% concentration value corresponds to that at the end of the dissolver operation. When H_3PO_4 was added in small increments to 68.6% UNH solution at 105°C no precipitation was detected until 0.5% H_3PO_4 by weight was added. The comparable material containing 0.3% H_3PO_4 was stable while hot, but crystallized on standing a short while at room temperature. When the H_3PO_4 concentration was 0.1%, no precipitation was evident after standing several hours.

M.F.A.

M.F.Acken, Chief Supervisor
Sep.Eng.Div.-200 Areas Tech.Dept.

MFA:jd

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#1	R.M.Evans	#13	300 Area File
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#6	W.O.Simon for The Area Engineer Attention: Patent Group	#18	R.S.Apple
#7	N. Hilberry	#19	R.H.Beaton
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#11	M.H.Smith	#23	J.J.Urban
#12	L.Squires	#24	F.B.Vaughan

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Canyon Buildings (221)

Extraction Waste Losses

The processing of lower activity metal in the B Canyon has made possible an increase in the weight of metal processed per charge. Beginning with run B-5-09-D-11, the extraction conditions were those previously employed for 1.5 ton charges with the exception that the nitrite treatment was performed in Section 6 and the strike was made in Section 8 at 75°C. The waste losses were 3.2% and 2.0% on runs -D-11 and -D-12. On runs -D-13 and -D-14 the extraction temperature was increased to 85°C and the waste losses were 0.44% and 0.83% respectively. On the basis of these few results it does not appear probable that the use of Section 6 is responsible for the higher waste losses obtained in the T Canyon (approximately 1.5%). A survey of all the runs made during the past two months in both the B and T Canyons is being made in an attempt to obtain an explanation for these variable and high waste losses.

The use of Section 6 as an assay point and for performing the nitrite pre-extraction treatment is being continued in both the B and T Canyons.

Neutralized Waste Storage (B and T)

A summary of the temperatures and reserve capacities in each area are presented below:

		Temperature (F°)					
Area	Date	Metal Waste		Empty	1st cycle		2nd cycle
		101	102	104	107	108	110
T	5/21/45	93	--	62	83	--	83
	6/19/45	108	--	67	90	--	88
	7/17/45	118	--	70	94	--	88
	8/21/45	132	--	78	102	--	90
	9/20/45	138	102	--	94	86	89
B	5/15/45	75	--	52	68	--	58
	6/19/45	97	--	55	75	--	71
	7/17/45	110	--	57	80	--	78
	8/21/45	136	--	65	90	--	84
	9/20/45	142	--		96	--	86

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Tanks	Type Waste	Gallons per run*	Reserve Capacity as of 9/20/45 B Area Gallons	Runs	T Area Gallons	Runs
X-101, -2, -3	Metal	5900	1,096,000	185	953,000	161
X-107, -8, -9	Ctng., 1st cycle	4700	1,137,000	242	985,000	210
X-110,-11,-12	2nd cycle	3500	1,264,000	362	1,103,000	315

* Based on 1.5 ton charges, the most probable future charge size

Of particular interest is the fact that the first of the cascading series of three tanks for metal solution and first cycle wastes have been filled in the West Area and that the corresponding tanks in the East Area will be filled during the next month. The spare metal solution tanks (X-104, -5, -6) have not been included in the above calculations of reserve storage capacity.

Concentration Buildings (224)

LaF_3 Product Cake Removal

Both T and B Areas are continuing to have difficulty in removing the LaF_3 product cakes from the E-2 centrifuge bowl. During the past week in both areas product has built up in E-2 equal to 50% of a single run. An acid wash in the East Area between runs B-5-09-F-10 and D-11 removed product equivalent to 50% of run F-10. A KOH clean out of E-2 in West Area between runs T-5-09-F-12 and F-13 netted product equivalent to 47% of run F-12.

In view of the success obtained in eliminating the LaF_3 product cake hold-up by treatment with hot 50% KOH in the T Area cleanup (T-5-09-ECW-1), it has been proposed that the KOH normally added directly to the F Cell precipitator (F-1) during metathesis be added via the E-2 centrifuge as part of the regular LaF_3 product cake removal procedure. Production test SE-224-T-PA-7 has been written to cover a test of this procedure which will begin with run T-5-09-F-13.

In B Area the D-2-D and the E-2-D pumps have been connected in series to give a high pressure spray (150 lbs./sq.in.) to assist in the E-2 cake removal. Further modifications of the sprays are planned but in the first two runs the higher pressure alone was not sufficient to give good cake removal.

The use of 24% nitric acid to assist in slurrying the product cake from the bowl (Production Test SE-224-PA-2) was tried in the East Area with erratic results. Further work along this line has therefore been abandoned.

Recycle Runs

The recycle solution inventory in tank E-4 has reached a point where it is again desirable to make a "master" recycle run starting in Cell D. This run will come between the regular scheduled runs T-5-09-D-14 and D-15 and will be made following the same procedure as the last recycle run T-5-08-R-1.

Run T-5-09-F-9-A

In conjunction with the E-2 cake removal problem, a second cake removal cycle was carried out in run F-9. The cake removed was processed as a separate batch (F-9-D). This second cake removal only partially reduced the E-2 Beckman background. The lanthanum removed was carried through a standard volume metathesis with no additional La being added.

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Isolation Building (231)

Oxalate Method of Handling 231 Building Recycle Solutions

Two more plant runs have been made by the oxalate method with quite satisfactory results. The total cycle time has been reduced from 25 hours to 11 hours by discarding the supernates before a control analysis is obtained. Losses have been tolerable and usually small. In all but one run (T-5-08-D-26), the total recycle has gone into one can. It is believed the cause of this exception was precipitation of the oxalate in too basic a solution, with some basic oxalate being formed. Below is a summary of all of the plant runs made using the adjustable decant line in tank CT-1 of Cell 4.

Run Number	Initial Volume in CT-1	% Prod. in Supn't.	% Prod. in Wash	Final Recycle Volume	% Prod. in Fin. Soln.	Mat. Bal. %	Actual Loss %	Total Operating Cycle Time
T-5-08-R-1	144 l.	1.4	2.7	36.0 l.	87.5	92.1	4.6	18
T-5-08-D-25	123	4.5	1.9	29.0	98.8	105.2	6.4	23
T-5-08-D-26	115	0.68	0.92	65.7	103	104.6	1.6	25½
T-5-09-D-5	117	0.6	0.5	43.1	99.9	101	1.1	25½
T-5-09-D-[illegible]	119	2.2	1.2	32.4	87.4	90.8	3.4	11

The total product losses given above average about 0.1% based on the amount of product in the starting solution (8-1-MR)

Process Chemistry

Reduction in Caustic Usage for Waste Solution Neutralization

Recent laboratory work has shown that it would be desirable to neutralize the second cycle waste solutions to a pH of 6-7, thus minimizing product solubility. This in turn would permit ditching of the supernatant solution from the settled neutralized waste, thereby increasing the waste storage tank capacity.

The standard plant procedure calls for the use of excess alkali in carrying out the waste neutralization. This is considered necessary, particularly under start-up conditions, to insure the absence of any corrosion of the steel liners in the waste storage tanks from under-neutralized waste. As a result there was, on September 15, about 315,000 gla. of second cycle waste in the 110-E tanks at a pH of 9.6. In the West Area the corresponding figures were 480,000 gal. at a pH of 9.8. In view of this situation the following program has been recommended.

a) To avoid the further introduction of excess caustic in the plant wastes, neutralization of the second cycle wastes in both areas should be carried out with reduced caustic ratios to give a pH of 7.

b) Laboratory experiments will be made to determine the extent of corrosion on the type of steel used for tank liners by waste solutions neutralized to pH values in the range from 3-10.

c) If it should prove feasible to neutralize second cycle wastes to pH values of less than 7, a plant test will be recommended in an effort to reduce the high pH of waste solutions already stored in the plant tanks by the addition of under-neutralized wastes from current operation.

d) Further consideration will be given in the laboratory and plant as to the desirability of reducing the amount of caustic used for neutralizing other plant waste solutions.

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Pertinent data (based on laboratory experiments concerning caustic requirements for pH adjustment of the second cycle waste solutions are given below:

Final pH	Gallons of Current Waste at Indicated pH Needed to Bring Contents of Tank to pH-7 110-E(1)	110-W(2)	Gallons of 50% NaOH Needed to Neutralize 2nd Cycle Waste Solutions to Various pH's 1st part of 2nd cycle waste (assume 1550 gal./batch)	2nd part of 2nd cycle wast (assume 1410 gal./batch)
2	54,000	102,500	163	97
3	65,700	128,000	174	111
4	74,500	142,000	178	116
5	85,000	162,000	185	120
6	169,000	322,000	205	137
7			218	159
8			226	169
9.0			236	172
(3) 9.6			(3) 250	(3) 175
10			268	187

(1) Based on 315,000 gal. of stored waste in East Area at pH - 9.6
(2) Based on 480,000 gal. of stored waste in West Area at pH - 9.8
(3) Based on plant experience in East Area

It is of interest to note that titration data indicate that all of the free nitric acid and the primary hydrogen ion of the phosphoric acid present in the waste are neutralized at a pH of approximately 3.

The Fluomolybdate Process

Product Losses - The reasons for the 1% product loss in the product precipitation step with fluomolybdates present are being investigated. The data given below indicates that the fluomolybdate derivitives complex bismuth and increase its solubility under normal process flow sheet conditions:

Added Agent	mg. Bi/liter in effluent from $BiPO_4$ Product Precipitation
None (control)	100
.05M fluosilicate	160
.05M - MoF_3	550
.05M - MoF_4	500
.05M - MoF_5	495

Tests are being made to determine whether adjustments in the phosphoric acid concentration will improve the product yield.

Specific Activity of 265 gt Pu

A second series of measurements to compare the specific activity of 265 gt Pu with 2 gt Pu (Clinton product) was completed. As in the previous measurements, 8 discs of each were prepared. The procedure consisted of pipetting 5 microliter samples from a solution containing approximately 10mg Pu/ml onto 8 mm. diameter, 0.1 mil. thick Pt plates, diluting this solution to 20 microliters with 2N nitric acid, drying the solution with a heat lamp, then igniting for one-half hour at 750°C. Care was taken to spread the sample over as large an area as possible and as uniform as possible since the lack of precision in previous experimental results suggested

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that perhaps some self absorption was taking place. The discs were then weighed on the quartz microbalance and counted for one hour each in random order on the vacuum counter. The results are shown below:

Experimental Data for the Comparison of 265gt Pu with 2gt Pu

Sample No.	Site	Specific Activity	Sample No.	Site	Specific Activity
1	X	discarded	9	W	1.4939
2	X	1.3712	10	W	1.4932
3	X	1.4084	11	W	1.4832
4	X	1.4007	12	W	1.4724
5	X	1.4030	13	W	1.4913
6	X	1.4108	14	W	1.4768
7	X	1.4196	15	W	1.4711
8	X	1.4160	16	W	1.4834
Average	X	1.4042	Average	W	1.4830
	-#2	1.4098			

Increase in specific activity = 5.6% if #2 is included
5.2% if #2 is omitted

* These results are tentative-depending on results obtained by the Statistics Group

Soil Retention Studies

One aspect of the proposal to ditch the supernatant solution from settled 2nd cycle neutralized wastes involves the question of product retention in the soil in contact with the discarded waste.

Approximately 3.5 gal. of 110 tank (second cycle) waste solution have now been passed into the column containing 43½ lbs. of soil from the 361 tank excavation. About 2.8 gal. were collected from the outlet during the 24 hour period that the solution was run through. The starting solution had approximately 950 alpha counts/ml. and the last fraction of the effluent solution failed to show more than 2 alpha counts/ml. In the major portion of the solution which was collected over night at a flow rate of approximately 7 ml./min. there was a heavy flocculent precipitate. This material will be submitted for spectrographic analysis. The pH of this solution was about 7, whereas the pH of the starting solution was 10.5. The final fraction of the effluent solution had a pH of approximately 9. The effluent solution had a much darker brown color and appeared to contain more suspended solids than did the starting solution.

After passing 6.5 gal. of 110 waste tank solution through the soil column, it was observed that approximately 25% of the Pu was coming through. The initial breakthrough of Pu in the effluent occurred after passing approximately 4 gal. of solution through the column. Work will be continued to determine the saturation value for the product in the soil and attempts will be made to observe the breakthrough of fission products in the current effluents which contain paractically no solids.

M. F. Acken

M.F.Acken, Chief Supervisor
200 Areas Tech. - Sep.Eng.Div.

MFA:jd

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#1	R.M.Evans
#2	R.M.Evans
#3	W.O.Simon for The Area Engineer
#4	W.O.Simon For The Area Engineer
#5	W.O.Simon for The Area Engineer
#6	W.O.Simon for The Area Engineer Attention: Patent Group
#7	N. Hilberry
#8	N. Hilberry
#9	N. Hilberry
#10	W.O.Simon-B.H.Mackey-700 Area
#11	M.H.Smith
#12	L.Squires
#13	300 Area File
#14	Pink Copy
#15	Yellow Copy
#16	M.F.Acken
#17	W.C.Kay - J.E.Cole
#18	R.S.Apple
#19	R.H.Beaton
#20	W.H.Sullivan
#21	G.W.Struthers
→ #22	J.D.Ellett-700 File
#23	J.J.Urban
#24	F.B.Vaughan

INV. 9-47

September 28, 1945

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200 AREAS
September 21 through September 27

Canyon Buildings (221)

Dissolver Performance (B and T)

The time cycles for the 3-5-R dissolver in each area have been consistently longer than for the 4-5-L dissolver, as indicated below. This is due to a slower reaction rate, in turn due to a smaller weight of metal heel. In order to shorten the time cycles so that the second and third charges can be completed within 15 hours elapsed time, the heels in all dissolvers are being increased.

Dissolving Time (Hours)

	Dissolver					
	3-5-R (5300 lbs. acid)			4-5-L (5300 lbs. acid)		
	Batch No.			Batch No.		
	1	2	3	1	2	3
B-08 Series	7	6	8	8-1/4	6-1/2	8
	6-1/2	6-1/4	10	6	6-1/2	8-1/4
	7	6-1/2	11-3/4	5-3/4	6-1/2	7-1/4
	5-3/4	6	12	6-1/4	6	7-3/4
B-09 Series	(5200 lbs. acid)			(5300 lbs. acid)		
	6	5-3/4	11-1/2	5	5-3/4	7
	(5000 lbs. acid)			5-1/2	6-1/4	7-1/4
	6-1/4*	6-1/4	11	6-3/4	6-3/4	8
	6	6-3/4	12	5-3/4	8-1/2	7-1/2
T-08 Series	(5500 lbs. acid)			(5500 lbs. acid)		
	7-1/2	8-1/2	10-1/2	7	6-1/2	8-3/4
	7	8-1/2	11	6	5-3/4	8
	6-3/4	7	11	5	6	8
	7-1/4	10	13-3/4	7	7	8-3/4

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	1	2	3	1	2	3
T-09 Series	(5500 lbs. acid)			(5500 lbs. acid)		
	8-1/4	10-1/2	16-1/4	6-1/2	7-1/2	8-3/4
	(5300 lbs. acid)			6-1/2	7	9.
	7-1/4	8-1/2	18	6	7	9-1/4
	(5000 lbs. acid)					
	7	8-1/2	13-3/4			

* 5200 lbs. acid

Extraction - Skimmer Failure

The skimmer on the extraction centrifuge (8-2) in the B Canyon failed on run B-5-09-D-16. Starting with run B-5-09-D-17, the extraction is being performed in Section 7. The cause of this failure is not definitely known, but at the start of the B Canyon, the skimmer was bent (B-5-04-I.P.) and it is presumed that subsequent strains have resulted in its ultimate failure.

Waste Disposal (B and T)

The Separations Engineering Laboratory has found that the most feasible method of reducing the product in the effluent of the second cycle neutralized waste is by reducing the final alkalinity from a pH of approximately 9.5 to a pH of 6.5-7.5. Accordingly, beginning with run B-5-09-D-15 in the B Canyon and run T-5-09-D-17 in the T Canyon, the caustic addition to the second decontamination cycle wastes has been reduced approximately 10%. The pH of the subsequent wastes have been:

pH (Neutralized Second Cycle Waste

Run	1st half	2nd half	Run	1st half	2nd half
D-15	6.4	9.1	D-17	6.8	8.7
D-16	7.0	9.2	D-18	6.5	7.2
D-17	7.9	8.4	D-19	7.2	6.5

Concentration Buildings (224)

LaF_3 Product Cake Removal

T - Area - After the regular LaF_3 product cake removal had been carried out on Run T-5-09-F-9, a second cake removal cycle was carried out using approximately 2000 lbs. of water with extensive spraying and slurrying, in an attempt to bring the E-2 Beckman meter reading down. The reading was reduced from 31 to 18×10^{-14} amps., giving rise to Run T-5-09-F-9-A.

The regular cake removal procedure was used on Run T-5-09-F-10, with a rise in cake hold-up again, as indicated by the E-2 Beckman. Run F-11 contained a slurrying with 250 lbs. of 24% HNO_3 with another gain in cake hold-up resulting.

As pointed out last week, a hot caustic flush of E-2 was carried out (Run T-5-09-ECW-1) by the following procedure: (a) slurrying with two consecutive 400 lb.

batches of hot (85°C) 50% KOH; (b) slurrying with 250 lbs. of 24% HNO_3; and (c) spraying with 1000 lbs. of water. This procedure succeeded in reducing the E-2 Beckman from 40 to $1x10^{-14}$ amps. and 47.1% of a regular product charge was picked up.

Since this clean-out run, a caustic cake removal procedure has been used on E-2 centrifuge in accordance with production test SE-224-T-PA-7. This procedure consists of (a) spraying 700 lbs. of water through the bowl at 10 rev./min.; (b) slurrying 5 times with 361 lbs. of hot (85°C) 50% KOH; (c) repeating (b); (d) flushing with 200 lbs. of water from the scale tank; and (e) spraying enough water (approximately 800 lbs.) through E-2 to bring the F-1 total weight to 2400 lbs. On the six runs thus far carried out (Runs F-13, R-1, D-14-15-16-17) the final E-2 Beckman readings have ranged from 0 to $2x10^{-14}$ amps., indicating excellent cake removal.

B - Area - After five runs had been carried out at B-Area using the 2 slurryings with 250 lb. batches of 24% HNO_3 (Production Test No. SE-224-B-PA-2 on Runs B-5-09-F-5 through F-9), no appreciable change in the E-2 Beckman readings could be noticed, either run by run or for the over-all series. F-1-PS assays indicated an increase in cake hold-up, however. A return to the regular East Area cake removal procedure (extensive water slurryings) on Run B-5-09-F-10 caused an appreciable rise in cake hold-up.

An acid wash (B-5-09-EAW-3) was carried out on E-2 after run F-10 by slurrying enough 60% HNO_3 and water through E-2 to give 5000 lbs. of 1N HNO_3 in E-4. This reduced the E-2 Beckman from 24 to $0x10^{-14}$ amps. and picked up 50.8% of the F-10 product content. On Run D-11, two of the Taber spray pumps were connected in series to give 150 lbs./sq. in. pressure and the regular West-Area cake removal procedure used. A large cake hold-up resulted. Run D-12 gave similar results by the same procedure.

Following run D-12, a second acid wash (Run B-5-09-EAW-4) was carried out on E-2, reducing the E-2 Beckman from 24 to $0x10^{-14}$ amps. and increasing the total E-4 pick-up to 63.4% of a normal product charge. Run D-13 was carried through Cell E with another large product product hold-up in E-2. The acid wash solution was jetted from E-4 to D-1 to D-3 (via D-2) to E-1, where 2 strikes were made with 500 lbs. of 1% lanthanum salt solution.

After the LaF_3 product cake from the acid wash recovery run (B-5-09-EAW-4) had been carried on to the metathesis wash centrifugation, the residual high E-2 Beckman reading warranted a second cake removal cycle. This was carried out by using two slurryings with the E-2 plows inserted first three-fourths in and then all the way in. The E-2 Beckman was reduced from 29 to $2x10^{-14}$ amps. and an additional 22.4% product was sent on to be combined with the 55% product in metathesis. The two-pump high-pressure spray was and is still being used on all cake removals.

On Run D-14, the plowing procedure was again used, with an increase in final cake hold-up. Complete cake removal was effected by slurrying all of the 50% KOH (at 85°C) for metathesis through E-2, however. Since this time, the West-Area hot KOH cake removal procedure has been used on two subsequent runs with excellent results and is to be continued.

Installation of new high-pressure spray headers is being planned at both areas for early trials. The West Area tests will be carried out with the 300 lbs./sq.in. pump and the East Area tests with the series pump arrangement.

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Isolation Building (231)

Oxalate Method of Treating Recycles

The method has been used on a total of nine full scale plant runs in Cell 4. Losses have in general been satisfactorily low while yields and material balances wer good. Data for the six runs not previously discussed follow:

Sample Description	CT-1-W Initial Waste		F-2-S 1st Supernate		F-2-SW 2nd Supernate		CT-1-R Final Recycle		Mat.Bal.
Run	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W
T-5-09-D-5	100	3.14	0.6	0.018	0.5	0.016	99.9	3.14	101
T-5-09-D-8	100	1.95	2.2	0.043	1.2	0.023	87.4	1.70	90.8
T-5-09-F-10	100	2.26	0.67	0.015	0.66	0.015	99.1	2.24	100.4
T-5-09-D-14	100	3.14	9.2	0.29	0.06	0.0019	99.2	3.12	108.4
T-5-09-R-1	100	5.93*	14.2	0.84	4.2	0.25	136	8.07	154
T-5-09-D-15	100	2.94	2.61	0.077	0.41	0.012	97.0	2.85	100
Ave. of 9 runs	100	2.94	3.9	0.115	1.37	0.040	100.5	2.96	105.8

* Based on E-4-RC rather than 8-1-MR

It will be noted that F-2-S was high on runs T-D-14 and T-R-1 while F-2-SW was high on T-R-1. No reason is known for the high T-D-14 result and it is probably in error since the material balance is 108% for the run. The high losses on T-R-1, however, are probably real since it is known that KOH was inadvertently added in place of part of the $NaNO_2$ during peroxide destruction.

Time cycles for the entire oxalate procedure have varied from 11 to 25-1/2 hours. A routine time cycle of 8 to 12 hours still seems probable, however. Considerable time has been lost due to repeated failure of the steam jet in the line from CT-1 to F-2. This failure seems to be due to occasional plugging, overheating, and loosening of flanges. Revision of the installation seems indicated.

The volumes recycled varied from 30 to 43 liters in the six runs, thus requiring the use of only one transfer can per batch. Acidity of this recycle has varied from 1.36N to 2.64N in the same runs. In order that 224 Building may dilute with water and stay above 0.7N, it is preferable that the acidity of the recycle be about 3.0N.

An additional aid has been found for locating the two end-points about which some concern has been expressed in previous reports. When adding KOH to make the solution alkaline after lanthanum oxalate precipitation the end-point has been indicated by precipitation of $Fe(OH)_3$ which changes the white color of the oxalate precipitate to a brownish-orange. It has been found that the G.E. potentiometric titration equipment used in locating the end-point when titrating H_2O_2 with $NaNO_2$ is also applicable in the KOH titration. A very strong deflection of the recorder pen occurs at the end-point if the instrument is simply turned on; no polarization, such as is required for the peroxide-nitrite end-point, is necessary. The final end-point also has been found well delineated by the titrometer, a very marked deflection being obtained when all oxalate has reacted with the $KMnO_4$ and MnO_2 precipitation begins; again no polarization is needed.

Acidity of P-1 Solution

Repeated upward adjustments of the quantity of nitric acid used in preparing the F-10-P solution, have finally resulted in solutions of too high an acidity. After 18 of 25 runs required KOH addition to P-1 because the acid normality was above 2.2, it was requested that a decreased amount of HNO_3 be added. This has been done and lower P-1 acidities are expected; the change was made at runs B-5-09-D-11 and T-5-09-D-17.

Sample Can Modifications

Due to reports from our consumer that thread seizure occurs between sample cans and filter caps or adapter plugs, a program has been started to eliminate some threaded surfaces. The filter cap threads (both inside and out) on new sample cans are being removed by turning down on a lathe. The filter medium and screens are held in place by a snap ring. The cap is firmly seated in the can by means of two eye bolts hooked over the sample can lugs and bolted to the lid. A high pressure lubricant is being applied to the lower sample can threads and to the threads of the adapter plug.

Cell 1 Revisions

The project for modifying the equipment to make Cell 1 conform with Cells 2, 3, and 4 has been approved.

P-1 Analyses

The chemical assay value of the product solution received from the Concentration Building (P-1) is currently used as basis for accounting within the Isolation Building since shipments must be based on chemical assays of AT solution.

Process Chemistry

Fluomolybdate Process

In an experiment to determine the loss of product in the $BiPO_4$ product precipitation in the presence of .05M fluomolybdate acid, it was found that bismuth was complexed to the extent of 500mg Bi/l with a 1% loss of product. Further experiments have shown that this high product loss can be avoided by making the process solution .3M in H_3PO_4 prior to the bismuth strike, then eventually building up the H_3PO_4 concentration to 0.6M H_3PO_4 as is ordinarily done in plant practice. Losses of a magnitude of 0.5% or less were obtained. Such losses are essentially equal to those in the control runs.

In one set of runs to determine the effect of fluomolybdic acid on ruthenium contamination, it was found that the decontamination factor was higher using fluomolybdic acid than with fluosilicic acid under normal process conditions, (no added agent). This result is somewhat surprising and should be verified. The results obtained were:

Conditions	Decontamination Factor for One Cycle	
Control	No Added Agent	103
.05M	H_2SiF_6	88
.05M	H_2MoOF_4	158

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Preparation of Samples for Half-Life Determination of Isotope 95^{241}

Because of the frequent handling of the standard platinum discs during decay measurements, with consequent accidents and damage to plates, it was deemed advisable to use special mounting methods so that measurements can be made over long periods of time without damage to the samples. As a result, a new type of sample mount similar to that developed by the Analytical Division, is to be employed for samples now in preparation. The sample mounts consist of stainless steel "cases" having nylon windows of 1.5mg/cm^2 thickness. Two of a total of 6 discs will be made up as standards, using highly purified 2gt Pu. The other 4 plates will contain isotope 95^{241} isolated from Pu at 2 different gt levels. Two plates will be prepared from each batch with different chemical operations being employed in the preparation of each of the pair of discs.

Storage of AT Solutions at Various gt Values

It is desirable for future reference and research that a series of samples of t final plant product solution be obtained from metal irradiated at various power levels.

Consequently, samples of AT solutions from 25, 55, 110, 155, 185, 235, and 265 gt runs have been prepared for storage. Each of these solutions was run through one additional peroxide precipitation following the plant operations. The solutions, varying in volume from 0.5 to 1-ml. in 8 to 10N nitric acid, have been placed in glass-stoppered volumetric flasks.

M. F. Acken

M. F. Acken, Chief Supervisor
200 Area Technical

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#1 R.M.Evans
#2 R.M.Evans
#3 B.H.Mackey for The Area Engineer
#4 B.H.Mackey for The Area Engineer
#5 B.H.Mackey for The Area Engineer
#6 B.H.Mackey for The Area Engineer
Attention: Patent Group
#7 N.Hilberry
#8 N.Hilberry
#9 N.Hilberry
#10 B.H.Mackey -D.A.Miller -700 Area
#11 M.H.Smith
#12 L.Squires
#13 300 Area File
#14 Pink Copy
#15 Yellow Copy
#16 M.F.Acken
#17 W.C.Kay - J.E.Cole
#18 R.S.Apple
#19 R.H.Beaton
#20 W.H.Sullivan
#21 G.W.Struthers
→#22 J.D.Ellett – 700 File
#23 J.J.Urban
#24 F.B.Vaughan

October 5, 1945

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200 AREAS
September 27 Through October 3

Canyon Buildings (221)

Displacement Wash of Extraction Cake (B)

As reported previously, the skimmer on the extraction centrifuge (8-2) failed in the B Canyon on Run B-5-09-D-16. The usual slurry washing of this cake was replaced by a displacement wash, with uncertain results on the decontamination efficiency. The observed loss in decontamination on the run in question is believed due in part to the shorter cooling period of the metal processed:

Run Number B-5-09-	Wash	Cooling Time to Extraction Days	Decontamination G dF 17-4-P	 P-1	 C-4 Beckman
D-15	Slurry	42 (8090)	5.34	7.58	44
D-16	Displacement	35 (8160)	5.05	6.96	74
D-17	Slurry	36 (8160)	5.22	7.11	48
D-21	Slurry	40 (8160)	5.57	7.30	36
F-22	Slurry	40 (817F)	5.06		77
F-23	Slurry	41 (817F)	5.13		56

High Basis Assays (B)

Due to the failure of the Section 8 centrifuge in the B Canyon, the extraction step has been performed in Section 7, beginning with Run B-5-09-D-17. However, on the majority of runs made in this section, the 7-1-MR basis assay has been 3-5% higher than (1) the quantity transferred from 6-1 and (2) the product found in 7-4. One assay of the circulated precipitate dissolving acid, sampled from the precipitator (7-1) showed approximately 3.5% product; previous data obtained on the extraction step indicate approximately 7% product remains after the precipitator is emptied. These high assays could be explained, therefore, by postulating unsatisfactory functioning of the precipitator spray, leaving 3-5% $BiPO_4$ in the precipitator which is dissolved by the UNH solution and is assayed in the 7-1-MR sample. On Runs -09-D-17 through -10-F-2 (excluding -D-21 and -D-22 on which the basis was taken as 7-4-P plus 7-3-W) the average product yield (7-4-P) was 96% and the average waste (7-3-W) was 1.0%.

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Sept. 27-Oct. 3 '45

High Waste Loss Second Cycle Product (T)

The product waste (17-[illegible]-W) from the second decontamination cycle of Run T-5-09-F-20 contained approximately 22% product. The cause of this high loss is not apparent from the recorded run data. The other half of the iron and silico fluoride solutions which were added to this section went into the product precipitation of the first decontamination cycle on Run -F-22 with a resultant normal waste loss (0.38%). An acid analysis of the waste revealed that at the time of the strike, the nitric acid acidity was slightly over 2.0N. On the basis of previous data obtained at Clinton Laboratories, an acidity of 2N would probably result in a waste loss in excess of 10%, under present process conditions. The manner in which this additional acid was added to the charge is not known. The waste has been moved into Section 18, from which it will be moved into Section 19, one half at a time, diluted to the proper acidity, and recovered by a regular productstrike. Each product cake will be metathesized, using KOH, and recombined before sending it to the Concentration Building.

Run T-5-09-F-21 was advanced from Section 16 before the above waste was cleared from Section 17, thus necessitating advancing the -F-21 Run to Section 19 for the second cycle product precipitation. No difficulty was encountered in this procedure, although an insoluble brown mud appeared with the product solution which caused difficulty in the Concentration Building. (The precipitators and catch tanks in Sections 18 and 19 had been left filled with water, which may have deposited a slime on the tank surfaces. The equipment was not acid cleaned after jetting out the water.)

Water Leaks in the Canyon (B and T)

Liquid is accumulating in the sump tank (5-7) at the rate of approximately 170 lbs./hr. in the B Canyon and 1200 lbs./hr. in the T Canyon. This liquid contains very little product or by-product and is thought to be leaking cooling water. In the T Canyon, the leak has been traced to the out-flow pipe between the jacket wall and the connector head on the precipitator in Section 13. Elimination of this leak would mean replacing the precipitator, so nothing further is being done about it at this time.

Activity on Ventilating Fans (B)

The ventilating fans (291 Building) in both the B and T Areas continue to build up activity at a rate which shows no tapering off. The present level is 5-10 r/hr. at the fan housing. One approach to the problem of decontaminating the fans and prolonging their life is that of running only one fan. The feasibility of this is being investigated in B Area. In order to determine the ventilation obtained in the canyon with one fan, it is planned to run in this manner for short periods of time. Before this can be done, however, the automatic controls on the steam fan have to be adjusted to permit shutting down one of the electric fans without having the steam fan start up. These adjustments are now being made.

Concentration Buildings (224)

LaF_3 Product Cake Removal

Both T and B Areas are continuing to use hot 50% KOH slurry removal of the LaF_3 product cake [illegible] centrifuge E-2. The method being used was described in the report of September 2[illegible]s far, twelve runs have been carried out successfully at T Area and nine runs a[illegible] Area. The average final Beckman meter reading is less than

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$1x10^{-14}$ amps. and the highest observed reading has been $3x10^{-14}$ amps. The F-1-PS product assays support the Beckman readings in indicating no cake hold-ups in E-2.

Thus far, all runs have been carried out with hot (85°C) KOH solution. Tests with cold (35°C) KOH solution are to be requested in the near future, since the use of cold KOH would permit the make-up of the solution in a more convenient section.

Both T and B Areas are proceeding with preparations for the installation of a new high pressure spray nozzle system in the E-2 bowl.

A-4-BP Rework on Run T-5-09-F-21

Due to the hold-up in the Canyon (221) Building caused by the high 17-3-WS loss on Run T-5-09-F-20, it was necessary to send Run F-21 on down through Sections 18 and 19 for the second-cycle $BiPO_4$ product strike. The 19-4-P solution of Run F-21, on an 80% start-up volume basis, was found to contain a large amount of yellowish, insoluble "crud", picked up from the long-unused process vessels. This insoluble material was found to carry essentially no product when centrifuged in the laboratory, however, and the charge was received into the Concentration (224) Building.

The cross-over $BiPO_4$ by-product waste (A-4-BP) contained approximately 40% of the 8-1-MR starting product. Laboratory examination of the sample showed large amounts of the yellow crud previously observed in the 19-4-P solution to be present. This insoluble material centrifuged out easily and carried only about 0.7% of the total product present. The C-4-0 sample was also extremely cloudy and contained 45% reduced product.

Run T-5-09-F-22 was extremely close behind F-21 in the process, so Run F-22 was carried through the Cell A $BiPO_4$ by-product precipitation with the F-21/A-4-BP held in A-4. The precipitator, A-1, and catch tank, A-3, were carefully flushed with water after Run F-22 to lower the phosphoric acid heel and the F-21 A-4-BP removed from A-4 to A-1. It was impossible to flush the A-4 heel to A-1 because of process piping limitations. The F-22 by-product cake was then removed to A-4 by direct addition of HNO_3 to A-2 centrifuge from the gallery, to give a total A-4-BP of 1.4% (F-21 heel in A-4 plus A-2 cake loss on F-22).

Since it was felt that the presence of the insoluble cruds had inhibited the original C-4 oxidation, these cruds were removed before re-oxidation of the F-21 A-4-BP by centrifugation. The filtrate was diluted to an 80% start-up volume by 60% HNO_3 flushes from A-1 to A-2 to A-3 to A-1. After re-oxidation in the precipitator A-1, the rework by-product precipitation was made, producing a loss of only 0.54% this time.

Samples of the original A-4-BP cruds are being examined at present for identity and nature of interference with oxidation.

Recycling Control Methods

At T Area, calucation of production run recycling of isolation supernatants from Storage Tank E-4 to the LaF_3 by-product precipitation is now being based on a lanthanum as well as product assay of the storage tank solution. The equivalent of 9.0 lbs. of lanthanum is jetted into each run, a 1.0 lb. excess over the 2-charge total of 8.0 lbs. returned from the Isolation Building each day. In the event that the product contribution of this recycle addition would cause the limit of 315 product units to be exceeded in D-1, tailoring of the weight of recycle added, between 6.0 and 9.0 lbs. of lanthanum, is permitted. Control of aliquot recycling by analytical methods appears to be performing satisfactorily.

B Area A-4-BP Waste Losses

Recently the cross-over $BiPO_4$ by-product waste losses (A-4-BP) at B Area have been erratically high, ranging upward from the previous average of 0.35% as high as 0.75%. Inspection of the Cell A operating records has revealed that the A-1 to A-2 full-steam jet pressure has fallen to approximately 80 lbs./sq.in. from the usual 100+ lbs./sq.in.. Previous experience in emptying precipitator heels at anything less than 100 lbs./sq.in. makes it seem likely that variable product heels have been left behind, to be picked up in the cake-dissolving acid and sent to the waste tank, A-4. The steam valve on the A-1 to A-2 gang valve was replaced afterRun B-5-09-D-21 and higher steam jet pressures are now obtainable. On the few runs carried out since this change, the A-4-BP losses are ranging from 0.36 to 0.41%.

P-1 Acidities

Due to variations in lanthanum delivery to the Cell F metathesis, caused by run-to-run LaF_3 cake hold-ups in centrifuge E-2, the F-10-P (or P-1) final product solution acidities were exceeding the upper limit of the desired 1.8 to 2.2N HNO_3 concentration. The weight of 60% HNO_3 added to F-2 centrifuge for $La(OH)_3$ cake solution has been reduced from 26 to 25 lbs. at both T and B Areas, starting with Runs T-5-09-D-17 and B-5-09-D-11. A 12-run average for the P-1 acidities since this change is 2.19N at B Area, and a 5-run average is 2.26N at T Area. The acid addition is to be dropped to 24 lbs. at T Area, beginning with Run T-5-09-D-23.

Electronic Inch Control

The Electrical Department has recently constructed and installed a new type of electronic inch control on F-2 centrifuge at T Area. The device is called a "Revatrol". The control instrument is based on the use of a Wheatstone bridge containing two 6SH7 rectifier tubes on the input legs. The 7.5 millivolts from the tachometer generator (at 10 rev./min.) is balanced against a 2.0 volt circuit containing a Thyrotron tube, which fires in the range of 1.9-2.1 volts to open or close the motor circuit. Close control of the bowl speed from 8-10.5 RPM during the low-pressure spraying is effected by this inch control. During the high-pressure spraying, the motor circuit remains closed throughout, but the bowl speed falls and levels off at about 3 rev./min. This is considered to be satisfactory, however.

The 2.0 volt supply to the Thyrotron circuit makes possible the use of a voltmeter in the ordinary range of sensitivity as an excellent measure of bowl speed at low speeds.

Isolation Building (231)

Oxalate Method of Treating Recycles

Test use of this method has continued in Cell 4. Losses remain low, but low apparent yields and material balances have been reported on two runs.

The probable cause of the occasional erratic loss or yield figures reported on several runs is believed to be sampling difficulties. Visual examination of typical samples of the starting solution, wastes, and final recycle solution has shown the presence of varying quantities of precipitate after the sample had been stored. The CT-1-W samples contained a white crystalline solid which may have precipitated after sampling but during storage. The waste samples contained a very fine brownish-white solid which probably consisted of "fines" in the supernatant solutions and which settled out during storage. The CT-1-R samples contained the expected manganese dioxide precipitate. All the precipitates except the latter probably carry product. Laboratory aliquots removed from the main sample will obviously not be representative of the latter if the precipitate has settled out or if the pipette serves to "filter" out larger particles. This problem is not new, but was formerly encountered in the Isolation Building P-1-S and P-2-S samples and is currently met in the Concentration Building F-1-PS samples. One method of taking representative laboratory aliquots is to treat the entire large sample with reagents to dissolve the precipitate. This requires a fore-knowledge of total volume or total weight of the large sample. This presents practical difficulties, but various proposals are being considered.

Time cycles remain in the vicinity of 12 hours. Little of the time currently lost can be charged to steam jet difficulties discussed last week. The taking of four samples per run, however, requires from 2 to 5 hours since a total of 12 "peanuts" of 0.3 ml. each are required. One sample, CT-1-W, would presumably be dropped for routine operation.

The use of the G.E. potentiometric titration equipment for locating all three end-points, nitrite vs. H_2O_2, KOH vs. HNO_3, and $KMnO_4$ vs. $H_2C_2O_4$ has continued to be successful.

Acidity of P-1 Solution

The recent decrease in amount of HNO_3 used at Concentration Buildings to prepare the F-10-P solution has resulted in P-1 acidities much nearer the desired 2.0N. The ten September B Plant runs before the adjustment averaged 2.35N with nine values above 2.2N, while the thirteen B Plant runs after adjustment (after B-10) averaged 2.21N with only five values above 2.2N. The seventeen September T Plant runs before adjustment averaged 2.34N with thirteen above 2.2, while the six T-Plant runs after the change (after T-16) averaged 2.19 with three above 2.2. If this range of values continues, another slight decrease in amount of HNO_3 used in Concentration Building will be desirable.

Modified Sample Can

The modified sample can described in the September 26 report is being used exclusively in Cell 3. Consumer evaluation is awaited before converting all production.

Leak at Valve

The valve in the SR-1 to AT line developed a bad leak which was noticed while processing Run T-5-09-F-21. Dismantling the valve revealed a crumbled gasket which had obviously not been GX but probably an asbestos material. The repair was completed without incident, a Koroseal gasket being installed.

Increased Product Content of Recycles

At about Runs T-16 and B-14 in the 5-09 series there was a definite increase in the weight of product recycled to the Concentration Building. This recycle averaged 11 units for the 12 runs following B-EAW-4 whereas it had averaged 8.3 grams for the preceding 31 runs. No process difficulties that might be responsible have been found.

[redacted]

Flush of WR-1 to F-1 Line

It has been pointed out that the present procedure for transferring the second cycle supernate from WR-2 to CT-1 traps a small volume (approximately 500 ml.) of the solution in the line from WR-1 to F-1 station. This trapped peroxide material is then loaded out to the RC cans ahead of the recycle solution itself which has been treated to destroy H_2O_2. Effervescence has been observed in some RC cans as a result. A procedure to return this trapped solution to CT-1 tank before treatment has been tested in a Cell 2 dummy run. It will be incorporated in the operating procedure for regular runs as soon as possible.

Process Chemistry

100 Area Investigation of Film Formation

Preparations were completed for the analysis of film to be obtained from the October 2 push in the 100 B Area. The procedure for chemical separations will be essentially the same as that used for analyzing the film obtained in the 100 D Area, except for a few modifications. Calcium oxalate will be precipitated from the calcium fraction and also from the manganese fraction in order to establish the existence of calcium in the samples. The decay of gamma activity in the calcium and chromium fractions will be followed also.

Waste Disposal Problems

A sample of un-neutralized waste from the first cycle was obtained for titrimetric studies with standardized NaOH, so that data could be obtained to permit calculations of the amount of alkali required to neutralize the wastes to different pH values. The results of these studies, based on titration of 5 ml. of 1st cycle product waste solution are as follows:

	Gallons of 50% NaOH to Reach Indicated pH for:	
pH	1st half (1650 gal. Prod. Waste + B.P. Solution in 2520 lbs. 60% HNO_3)	2nd half 1650 gal. Prod. Waste
2	268	117
3	283	132
4	286	135
5	298	147
6	323	172
7	346	195
8	354	203
9	361	210
10	387	236
Present Practice*	397	226

* The neutralized 1st cycle wastes now in the 107 tanks have a pH of 10.2.

Calculations were made also of the amount of NaOH that should theoretically be used to neutralize the plant wastes, based on the amounts of acid used. The calculated and experimental values will be compared.

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M. F. Acken

M.F.Acken, Chief Supervisor
200 Areas Technical Division

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#1	R.M.Evans	#13	300 Area File
#2	R.M.Evans	#14	Pink Copy
#3	B.H.Mackey for The Area Engineer	#15	Yellow Copy
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#6	B.H.Mackey for The Area Engineer Attention: Patent Group	#18	R.S.Apple
#7	N. Hilberry	#19	R.H.Beaton
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Classification Cancelled (Change to DECLASSIFIED)
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200 AREAS
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Canyon Buildings (221)

Basis Assays (B and T)

As reported last week, the basis assay (7-1-MR) in the B Canyon was running 3-5% higher, since the use of Section 7 was started, than the indicated value obtained from Section 6 or subsequent product assays. (In the T Canyon, the basis assay (8-1-MR) for the last 9 runs has averaged 99.7% of the Section 6 value. The precipitator spray in 7-1 was suspected as being the cause of these high assays and was replaced after B-5-10-B-6. The comparison of 7-1-MR to 6-1-MR since B-5-10-B-8, the first run with a "clean" 7-1 Tank, are as follows:

B-5-10-B-8	-	100.5%
B-5-10-B-9	-	99.5%
B-5-10-B-10	-	102.0%
B-5-10-B-11	-	103.0%

These data, although encouraging are insufficient to determine whether or not the high basis assay difficulty has been corrected.

Activity in the Extraction Section (B)

Since discontinuing the use of Section 8 in the B Canyon on September 22 (due to centrifuge skimmer failure), the gross activity on the precipitator and centrifuge has decayed with a half-life approximating 1-1/2 to 2 years, while on the catch tank and precipitator dissolver it has decayed with a 1-3 month half-life.

Equipment Piece	Beckman Readings, Amps. 9/22/45	Beckman Readings, Amps. 10/9/45	Indicated Half-Life, Days
Precipitator (8-1)	37×10^{-10}	30.5×10^{-10}	600
Centrifuge (8-2)	67.5×10^{-10}	66×10^{-10}	500
Catch Tank (8-3)	16.5×10^{-9}	9.5×10^{-9}	21
Precipitate Dissolver (8-4)	17×10^{-9}	15×10^{-9}	95

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Elimination of Reduction Time in Decontamination Cycles (B)

The 1-hour digestion time at 75°C following the addition of ferrous ion in the product precipitation step of each decontamination cycle was eliminated in the T Canyon with Run T-5-09-D-1; it is being eliminated in the B Canyon beginning with Run B-5-10-B-13.

Neutralization of Second Decontamination Cycle Wastes (B and T)

Lowering the pH of the neutralized second cycle wastes to values between 6.5 and 7.5, for the purpose of more complete product precipitation from the effluent, has been attempted by reducing the 50% NaOH added to the first and second halves of the waste to 2860 lbs. and 1930 lbs. respectively. The average of the pH's obtained on the last 12 runs in the T Canyon are 7.7 and 7.3 respectively. On the basis of limited experience in the B Canyon using 2760 lbs. and 1930 lbs. respectively, these values will be adopted in the T Canyon, starting with Run T-5-10-F-14.

Run	1st Half (2760 lbs.)	2nd Half (1930 lbs.)
B-5-10-B-5	7.3	7.3
B-5-10-B-6	7.0	6.6
B-5-10-B-7	6.5	7.0
B-5-10-B-8	6.8	6.6

Water Leak in the Canyon (B)

The liquid previously reported collecting in the sump tank in the B Canyon has been traced to a leaking seam in the jacket of the Section 16 precipitator (second decontamination cycle by-product). The leak is not serious enough to require immediate maintenance work which would interfere with production schedules.

Slow Speed (650 rev./min.) Skimming (B)

Consideration is being given to skimming at a centrifuge speed lower than normal (870 rev./min.), with the resultant lessened strain on the skimmer. Preliminary to making such tests, a gauge is being installed on the 18-2 hydraulic line to permit determining the pressure required to skim at various centrifuge speeds.

Ventilating Fans (B)

Tests made in the B Canyon have indicated that satisfactory airflow conditions can be maintained in the Canyon building with only one of the 291 Building fans operating, providing the number of 4500 cu.ft./min. fans discharging into the Canyon (crane cab way) is reduced from 7 to 5. A production test covering operation in this manner over an extended period of time is to be prepared.

Concentration Buildings (224)

B Area A-4-BP Waste Losses

Since the replacement of the steam valve on the A-1 to A-2 jet gang valve at B Area, higher jet pressures (approximately 95 lbs./sq.in.) are now obtainable at full throttle. This provides greater assurance of complete emptying of the precipitator. Smaller precipitator heels will result in lower $BiPO_4$ by-product waste (A-4-BP) losses, since this heel is picked up by the acid used for cake solution.

The 11-run average for the B Area A-4-BP losses since this change (after Run B-5-09-D-21) is 0.35%, which checks closely with the T Area average of 0.31% for the same period.

F-10-P (or P-1) Acidities

Beginning with Run T-5-09-D-23 at T Area, the weight of 60% HNO_3 used to dissolve the $La(OH)_3$ cake from centrifuge F-2 was decreased from 25 to 24 lbs. An 8-run average of the F-10-P (or P-1) acidities since this change is 1.96N HNO_3, with only one run above the desired range of 1.8 to 2.2N. At B Area, a 20-run average obtained with the use of 25 lbs. of 60% HNO_3 is 2.20N, with 9 runs above the desired range of 1.8 to 2.2N. Beginning with Run B-5-10-B-5, the amount of acid used at B Area will be decreased to 24 lbs.

LaF_3 Product Cake Removal

Both T and B Areas are continuing to use the hot 50% KOH slurry removal of the LaF_3 product cake from Centrifuge E-2. The method being used was described in the report of September 26. Thus far 21 runs have been successfully carried out at T Area and 18 at B Area. The empty-bowl Beckman meter readings at the end of cake removal range from 0 to $2x10^{-14}$ amps., indicating excellent cake removal.

The high-pressure pump (300 lbs./sq.in.) and the new E-2 Spray Header (4 nozzles with 0.062-inch orifices) is to be tested at T Area on Run T-5-10-D-3.

F-1 Hold-Up

Recently at B Area, the empty F-1 (metathesis tank) Beckman meter reading has climbed up from an average of $10x10^{-14}$ amps. to $24x10^{-14}$ amps. over a period of about 3 runs. In order to determine whether or not this signifies an increasing hold-up of product, an acid wash of F-1 is to be carried out after Run B-5-10-B-5. Product pick-up will be recovered by neutralization and centrifugation of the acid wash solution.

Recycle Solution Neutralization

Analysis of the isolation supernatant recycle solution stored in Tank E-4 at T Area for lanthanum at the time of recycling to Run T-5-09-F-27 showed an unexpectedly high lanthanum assay. Further inspection of the storage tank solution revealed that it contained a large amount of precipitate, other than the usual traces of MnO_2. In addition the solution was basic, approximately 0.4N in KOH, instead of being 0.8N in HNO_3. Apparently the 50% KOH solution used for E-2 cake removal had leaked from the E-2-C-4C Scale Tank down into Tank E-4. The storage solution was acidified and the precipitate dissolved. No significant effects on the LaF_3 by-product waste (D-4-BP) losses were observed during this period. Gallery feed line changes are to be made shortly to prevent a recurrence.

LaF_3 Product Centrifugation Rates

In an effort to shorten the Cell E LaF_3 product precipitation cycle time by approximately 1 hour, the centrifugation rate has been increased in test at B Area from 70 to 80 lbs./min. Close observation of the effect on the waste (E-3-WS) losses is being made. Thus far, the 5-run average (Runs B-5-10-F-1 to B-5) loss is 1.36%. The previous monthly average (September) loss was 1.22%. Preliminary indications are, therefore, that the waste loss is slightly but significantly increased. The test is being continued to affirm or disprove this preliminary conclusion.

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Isolation Building (231)

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Oxalate Method of Treating Recycles

Twenty-three runs in Cell 4 have been processed through the oxalate treatment. Losses remain low, but occasional low yields and material balances are encountered. Data for the eight runs completed since last week follow:

Sample Description	CT-1-W Initial Waste		F-2-S 1st Supernate		F-2-SW 2nd Supernate		CT-1-R Final Recycle		Mat. Bal.
Run	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W	% of 8-1-MR	% of CT-1-W
T-5-09-F-22	100	4.0	0.67	0.027	0.09	0.004	89.8	3.6	90.6
T-5-09-F-21A	100	4.8	0.59	0.028	0.28	0.013	93.3	4.5	94.2
T-5-09-D-23	100	4.55	1.1	0.05	0.62	0.028	98.3	4.5	100.0
T-5-09-D-24	100*	22.8*	0.05	0.012	0.06	0.014	24.2	5.5	24.3
T-5-09-D-25	100	4.47	10.4	0.47	0.43	0.019	97.2	4.34	108
T-5-09-F-26	100	6.33	1.34	0.085	0.26	0.016	66.8**	4.2	68.4
T-5-09-F-27	100	3.64	1.1	0.04	1.26	0.046	93.8	3.4	96.2
T-5-09-F-28	100	4.14	1.45	0.06	0.69	0.029	91.0	3.77	93.1
Ave. of 23 runs	100	4.82	2.51	0.12	0.97	0.047	92.0	4.43	95.5

* CT-1-W assay obviously high by factor of about 4

** Sample contained white precipitate

It is evident from the above values that evaluation of the oxalate method is handicapped by poor material balances. As was discussed in the report of October 2, this is probably due to non-homogeneous samples delivered to the laboratory. A continued effort will be made to improve sampling technique.

Increased Product Content of Recycle Solutions

As pointed out in the report of October 2, there has been a recent increase in the weight of product recycled to the Concentration Building. For 13 runs starting with B-5-09-D-14 the average recycle in Cell 3 was 11.1 units, while for the same number of runs in Cell 4, starting with T-5-09-D-16, the average was 11.3 units. This eliminates the iron content of the P-1 solutions as a basic cause, since 50% more iron is currently present in P-1 solutions from T than from B Plant. (See below).

Spot checks of the concentration of the "Becco" hydrogen peroxide used for product peroxide precipitation have revealed that this reagent currently runs from 24 to 25% H_2O_2. Since the amounts added in both cycles have been calculated on the basis of 30% H_2O_2 (to make first cycle solution 9% and second cycle solution 10% in H_2O_2), precipitation is now being made at 7.5% in the first and 8.3% H_2O_2 in the second cycle. In the present high acidity (2.0N) process, these values may be low enough to cause the higher supernate losses mentioned above. This would be especially true of the first cycle. It is, therefore, proposed to increase the amount of H_2O_2 used. If such a change does reduce recycle losses, it will be very desirable to obtain stronger H_2O_2 reagent in order to minimize process volumes.

Iron Content of P-1 and AT Solutions

It has been noted that the iron content of P-1 and AT solutions increased about the middle of the 5-09 series. This is shown in the following table:

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Runs	Average Fe Molarity in P-1	Average mols Fe in AT
B-1 through B-13	0.0062	0.013
B-14 through B-26	0.0073	0.018
T-1 through T-15	0.0068	0.013
T-16 through T-27	0.0102	0.020

This change occurred at about the time of the increase in weight of product recycled, and may be a contributing factor. That it is not mainly responsible is evident because both T and B runs furnish large recycles whereas the change in iron content of P-1 solutions largely affected T runs in Cell 4. The latter variation is probably associated with recycle handling at T plant and may be caused by the addition of ferrous ammonium sulfate as an indicator in the Isolation Building oxalate method of waste handling, though only 20 grams of iron are in the latter procedure. It is, therefore, planned to eliminate the ferrous indicator and depend if possible on the G.E. potentiometric titrometer to show the end-point.

It is also of interest to note that the increase of Fe content of P-1 solutions seems to be followed by a similar increase in Fe content of AT solutions. The latter change has affected purity measurements very little..

Process Chemistry

100 Area Problems

As scheduled, the films obtained in the 100-B area during the push of October 2, were subjected to chemical treatment. The decay of the radioactivity associated with the various fractions resulting from the chemical treatment is being followed.

Specific Gravities of 15% KOH Solution at Various Temperatures

Since samples of F-9 metathesis waste effluent are now taken at elevated temperature, the specific gravities of 15% KOH at various temperatures were determined so that the necessary volume corrections could be made. The values (determined with hydrometers) are as follows:

Temperature °C	Specific Gravity
15	1.140
25	1.129
35	1.121
40	1.119
45	1.117
50	1.114
55	1.112
60	1.109
65	1.106
70	1.103
75	1.101
80	1.100
85	1.099

Assay on A-4 Sample From Run T-5-09-F-21

In Run T-5-09-F-21 a 40% product loss was encountered in the $BiPO_4$ by-product in the cross-over. An A-4 sample, submitted to the laboratory and known to contain

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an insoluble crud, was tested to determine whether the presence of the crud was responsible for the loss experienced in the plant.

The following tests were conducted on the sample:

1) A sample of the washed crud was submitted for spectrographic analysis. The following elements were reported (relative portions).

Al	200	Fe	G1000	Si	G1000
Ba	150	La	L50	Sn	L100
Bi	G-1000	Mg	80	Sr	L1
Ca	150	Mn	L10	Th	L200
Cb	L20	Ni	80	Zn	L200
Ce	L100	P	L1000	Zr	L50
Cr	1000	Pb	L50	Y	L1000

The high percentage of iron, chromium, and silicon suggests that the precipitate is tank scale or some related substance. The material is soluble in HF but is insoluble in HCl or HNO_3.

2) A portion of the crud was dissolved in HF, and titrated with standard ceric sulfate solution. It was found to possess no reducing properties.

3) A representative sample of A-4 was oxidized in the laboratory following plant flow-sheet conditions. A 19% product loss in the by-product resulted. This confirmed suspicions that the crud was responsible for the loss.

4) A second sample of A-4 was oxidized using five times the flowsheet quantity of sodium bismuthate. This time a 29% loss occurred in the by-product, showing that bismuthate concentration is not a controlling factor when the crud is present.

5) A centrifuged sample of A-4 was oxidized under flowsheet conditions. A normal by-product loss resulted in this case.

All these tests lead to the conclusion that it is necessary to remove the crud prior to oxidation. Fortunately, this foreign material is not encountered in normal operation.

Corrosion of Mild Steel in Waste Solutions Neutralized to Specified pH Values

Since it was found that plant second cycle wastes neutralized to a pH of 7 retain less product in solution than when they are neutralized to a pH of 10, it has been proposed that the pH of the contents of the 110 Waste Storage Tanks be lowered by adding wastes that are slightly acidic. Since the feasibility of operating at lower pH values is dependent on the extent of corrosion of the tank liners, a series of laboratory tests have been made in regard to this point. As can be seen from the data below, solutions of pH 3 appears too corrosive, a pH of 4 appears permissable but is close to the border line, and pH's of 5, 6, and 7 are relatively non-corrosive.

Simulated waste solutions were used for the tests.

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Corrosion of Mild Steel Coupons(1) in Partially Neutralized Simulated Waste Solutions

Portion of Coupon Immersed	pH of Test Solution	First Exposure inches/month	Second Exposure inches/month	Third Exposure inches/month
1) Total	3	0.00023	0.0011	0.0012
2) One-half	3	--	0.0012	0.0016
3) Total	4	0.00035	0.00038	0.00075
4) One-half	4	0.00042	0.00048	0.0008
5) Total	5	0.00018	0.00003	0.00018
6) One-half	5	0.00002	0.00004	0.00002
7) Total	6	None	None	None
8) One-half	6	0.00012	0.00012	0.00009
9) Total	7	None	None	None
10) One-half	7	None	None	None
11) Total	10	None	None	None
12) One-half	10	None	None	None
13) Total	3*	0.00015	0.00048	0.0014
14) One-half	3*	0.00005	0.0010	0.0019
** One-half	3		0.0012	0.0010
One-half	10		None	None

* Solution contains iron. Other solutions do not.

** Galvanic corrosion. Coupons connected by wire and solutions by liquid bridge.

(1) Same specifications as still used for tank liners. Each test piece contained a welded section.

M. F. Acken

M.F.Acken, Chief Supervisor
200 Areas Technical

MFA:jd

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October 18, 1945

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200 AREAS
October 9 Through October 15

Canyon Buildings

Extraction Waste Losses (B and T)

The extraction waste losses (8-3-W, 7-3-W) continue to vary over a range of 0.80% to 2.4%. Approximately 100 runs made in the B and T Canyons have been analyzed and the results summarized in Table I. From these data the following conclusions may be drawn:

1. The waste loss on individual runs varies by a factor of two independently of UNH concentration, bismuth to plutonium ratio, or strike and digestion temperature.
2. In some cases the waste loss decreases with increasing carrier to product ratio; in other cases there appears to be no such relationship.
3. When conditions become more critical (higher UNH concentration, lower temperature) the effect of the bismuth to plutonium ratio becomes more apparent.
4. At low UNH concentrations (18%), the concentration of phosphoric acid is less critical.

A variable not included in Table I (data not obtained) is the acidity of the extraction solution before the start of the strike. These data have been obtained on recent runs in each canyon; they fail to explain the variations in waste loss:

Run	Acidity (H^+, g/l)	Waste Loss (%)
B-5-10-B-16	0.96	0.87
B-5-10-B-17	0.87	0.82
B-5-10-B-18	0.90	2.0
B-5-10-B-19	0.94	1.7
B-5-10-B-20	0.89	1.0
T-5-10-F-14	0.67	1.3
T-5-10-F-15	0.80	1.1
T-5-10-F-16	0.73	1.4
T-5-10-F-17	0.81	1.3

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Table I

Extraction Waste Loss (8-3-W) vs Various Process Variables

Conditions*	UNH (%)	H_3PO_4 (M)	Bi (g/l)	Bi/Pu Avg.	Bi/Pu Range	8-3-W(%) Avg.	8-3-W(%) Range	No. of Runs
A	18.3	0.59	2.6	81	75-86	1.3	0.73-1.7	4
B	18.2	0.74	2.5	84	81-87	1.3	0.81-1.8	3
	19.0	0.77	2.5	98	94-102	1.2	0.76-1.6	8
C	20.0	0.60	2.6	75	73-78	1.2	0.88-1.7	3
D	20.0	0.72	2.4	83	81-85	1.6	1.4 -1.7	3
	19.9	0.77	2.5	93	89-95	1.4	0.61-2.1	9
	19.9	0.76	2.5	99	96-101	1.2	0.68-1.7	9
E	20.9	0.75	2.5	78	78	1.1	1.0 -1.1	2
	21.1	0.76	2.5	94	89-98	1.4	1.1 -1.9	5
	21.0	0.75	2.5	103	100-111	1.3	0.87-2.4	7
F	21.9	0.74	2.5	79	76-81	1.0	0.69-1.4	7
	21.9	0.76	2.5	97	87-103	1.2	0.40-2.0	7
G	23.0	0.74	2.5	81	76-85	0.80	0.67-1.0	5
	23.4	0.76	2.5	94	89-97	0.64	0.44-0.96	8
H	21.9	0.71	2.5	85	84-86	1.1	0.81-1.3	5
	21.7	0.71	2.5	103	99-109	0.61	0.38-0.88	10

* Conditions:

A - UNH under 19.5%; H_3PO_4 = 0.59 ± 0.03M; Bi = 2.5 ± 0.1 g/l; Temp. = 85°C.

B - UNH under 19.5%; H_3PO_4 = 0.75 ± 0.03M; Bi = 2.5 ± 0.1 g/l; Temp. = 85°C.

C - UNH = 20.0 ± 0.5%; H_3PO_4 = 0.59 ± 0.03M; Bi = 2.5 ± 0.1 g/l;
Temp. = 85°C.

D - UNH = 20.0 ± 0.5%; H_3PO_4 = 0.75 ± 0.03M; Bi = 2.5 ± 0.1 g/l;
Temp. = 85°C.

E - UNH = 21.0 ± 0.5%; H_3PO_4 = 0.75 ± 0.03M; Bi = 2.5 ± 0.1 g/l;
Temp. = 85°C.

F - UNH = 22.0 ± 0.5%; H_3PO_4 = 0.75 ± 0.03M; Bi = 2.5 ± 0.1 g/l;
Temp. = 85°C.

G - UNH over 22.5%; H_3PO_4 = 0.75 ± 0.03M; Bi = 2.5 ± 0.1 g/l; Temp. = 85°C.

H - UNH ± 22.0 ± 0.5%; H_3PO_4 = 0.70 ± 0.02M; Bi = 2.5 ± 0.1 g/l;
Temp. = 75°C.

Equipment Contamination (B and T)

The contamination of process equipment, as indicated by the Beckman instruments, has not increased materially (except in a few instances) over the past several months. A summary of the activity change on the equipment pieces in each canyon is presented below:

T Canyon	Extraction	B Canyon
Essentially constant since 06 series.	Precipitate	Essentially constant for 05 - 08 series.
Slight steady increase since 06 series.	Centrifuge	Essentially constant for 06 - 07 series.
Marked increase; now above full tank.	Catch Tank	8-3 catch tank essentially constant for 05-08; up rapidly 7-3 catch tank building.
Constant since 05 series	Precipitate Dissolver	Essentially constant for 06 - 08 series.

1st Cycle By-Product

Definite increase since 05 series; approaching full tank value.	Precipitator	Slight increase since 06 series.
Slow increase since 05 series.	Centrifuge	Slow increase since 05 series.
Slight steady increase since 05 series.	Catch Tank	Slight increase since 06 series.
Slight steady increase since 05 series.	Precipitate Dissolver	Slight steady increase since 06 series.

1st Cycle Product

Essentially constant since 07 series.	Precipitator	Essentially constant since 04 series.
Slight steady increase since 05 series.	Centrifuge	Essentially constant since 07 series.
Slight steady decrease since 07 series.	Catch Tank	Slight steady increase since 07 series.
Slow increase since 06 series; above full tank.	Precipitate Dissolver	Slight steady increase since 05 series; above full tank.

2nd Cycle By-Product

Slight increase since 06 series	Precipitator	Essentially constant since 06 series
Slight steady increase since 06 series	Centrifuge	Slight steady increase since 06 series.
No change since 07 series	Catch Tank	No change since 07 series.
Slight increase since 07 series	Precipitate Dissolver	Slight increase since 06 series.

2nd Cycle Product

Essentially constant since 04 series.	Precipitator	Slight increase since 06 series.
Essentially constant 04 - 09; slight increase since.	Centrifuge	Essentially constant since 06 series.
Slow decrease since 07 series.	Catch tank	Essentially constant since 06 series.
Slow steady decrease since 05 series.	Precipitate Dissolver	Slight steady increase since 05 series.

Concentration Buildings

Beckman Meter Background

Recently there have been several slow but steady increases in the Beckman meter background (empty tank) readings for certain process vessels. At T Plant, Tank C-4 background readings have risen to approximately 25×10^{-12} amps. (2500×10^{-14}), which is more than one-half of the full-tank readings which range from 3500 to 6500×10^{-14} amps. A similar situation has existed for some time at B Plant. No carry-over of these activity-bearing cruds in a manner such as to affect subsequent decontamination has been observed. Since these high backgrounds do not permit the original intention of detecting product solution hold-up by Beckman meter monitoring, the meter checking system has been replaced in the operating procedures by a certification by supervision that a C-4 and A-1 weight factor check indicates complete transfer of solution.

Centrifuge A-2 Beckman background at B Plant has recently climbed slowly above the 20×10^{-14} amp. limit presently specified. Additional acid washes of the centrifuge (500 lbs.) of 60% HNO_3) have lowered the background but slightly on three runs thus far. If the background remains high, the Beckman limit is to be revised upward since it is not now set as high as the average background plus 10% of the difference between the full and empty readings.

Metathesis tank F-1 (at B Plant) Beckman meter backgrounds rose to 24×10^{-14} amps. after run B-5-10-B-5 over a period of three runs (previous average was 10 - 15×10^{-14} amps.). A wash of F-1 with 500 lbs. of 2% KOH solution after Run B-5 reduced the Beckman reading from 24 to 15×10^{-14} amps. The centrifugation of this wash solution raised the F-2 Beckman from 0 to 20×10^{-14} amps. The P-1 product yield on the subsequent run (B-6) showed no product pick-up, however, making it appear that the high activity cruds in F-1 did not carry any product. The Cell F centrifugation procedure is being revised to include the use of air sparging of the F-1 slurry heel when the level drops below the agitator (approximately 350 lbs.) and the raising of jet steam pressure to full at the end of centrifugation. These measures are designed to assure a more complete removal of both liquid and solid heel during the centrifugation operations.

Final Solution

(F-10-P or P-1) Acidities - Since the weight of 60% HNO_3 used for solution of the F-2 metathesis cake has been reduced to 24 lbs., the F-10-P (or P-1) acidities are staying more closely within the desired range of 1.8 to 2.2N HNO_3. At T Plant, a 16-run average (T-5-09-D-23 to T-5-10-D-8) is 1.96N, with 5 runs slightly below 1.8N and none above 2.2N. At B Area, a 10-run average (B-5-10-B-5 to B-15) is also 1.96N, with but one run below 1.8N and none above 2.2N.

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LaF$_3$ Product Centrifugation Rate

In an effort to determine if the LaF$_3$ product centrifugation rates could be speeded up without significantly increasing product waste (E-3-WS) losses, the through-put rate has been increased in test from 70 to 80 lbs./min. at both T and B Plants. At T Plant, an 8-run average waste loss (Runs T-5-10-D-3 to D-9) is 0.93% at 80 lbs./min., as compared to the September average of 0.83% at 70 lbs./min., an increase of 0.1% in loss. At B Plant, a 10-run average (Runs B-5-10-F-1 to B-10) is 1.40% at 80 lbs./min., as compared to the September average of 1.22% at 70 lbs./min., an increase of 0.18%. Recently, at B Area, the test procedure has been modified so that the first centrifugation is carried out at 80 lbs./min. and the second at 70 lbs./min. A 5-run average loss (B-5-10-B-11 to B-15) is 1.26%, essentially no different from the September average. If the waste loss continues to stay down with this procedure, another set of test runs will be made with a rate of 90 lbs./min. on the first centrifugation.

At T Plant, the present time cycle saving is approximately 1 hour. At B Plant, the time saved is approximately 30 minutes, while the 90-70 lbs./min. rate combination will save 1 hour.

LaF$_3$ Product Cake Removal

At T Plant, the use of the hot KOH slurry method for removing the LaF$_3$ product cake from centrifuge E-2 was modified on Run T-5-10-D-3 by the installation of a high-pressure spray (250-300 lbs./sq.in.) made up of four 0.062-inch orifices designed to deliver a total of 8 gal./min. Both the high-pressure spray and the hot KOH slurry were used on this run. On Run D-4, the high-pressure spray alone was used. Cake removal was incomplete, as indicated by a final Beckman meter reading of 17x10^{-14} amps., as compared to the usual 0 - 2x10^{-14} amps.

On Runs D-5, D-6, and D-7, the high-pressure spray-KOH combination was used, resulting in progressively poorer cake removal, until finally the sprays plugged completely on Run D-7. The cake had to be removed by extra KOH slurrying and water spraying after re-installation of the low pressure sprays. The low-pressure sprays and KOH slurrying are being used successfully since the return to the former procedure.

At B Plant, the use of the series pumps (150 lbs./sq.in.) and the KOH slurrying have continued to produce complete cake removal, as indicated by both Beckman meter readings and F-1-PS assays.

At present, gallery tests are being carried out at B Plant with the new high-pressure spray header (4 nozzles with 0.062-inch orifices) to determine if the spray characteristics are satisfactory. At T Plant, gallery tests of factory-made 0.062-inch and 0.093-inch orifices are to be made at 300 lbs./sq.in.

Recovery Run T-5-09-F-20-A

The high-product 17-3-WS waste solution from Run T-5-09-F-20 was reworked in the Canyon Building in Section 19. After the 19-4-P product solution had been obtained, it was found to contain much of the same yellowish, insoluble crud which had caused the high A-4-BP loss on the previous run sent through Section 19, Run T-5-09-F-21. The crud was removed by recentrifugation in Section 19 and sent to the Concentration Building as an 80% start-up volume run. It was carried through concentration on this volume scale without difficulty and recycled to the E-4 Storage Tank.

The process data are given below:

Run T-5-09-F-20-A

% Yield (8-1-M of T-5-09-F-20)					% Loss				
17-4-P	C-4-0	E-1-R*	F-1-PS*	F-10-P*	A-4-BP	D-4-BP	E-3-WS	F-7-WS	F-9-WS
14.6	15.4	25.3	26.3	--	0.17	0.29	0.72	0.42	0.13

* Recycle of 10.3% added to run at D-1.

F-2 High-Pressure Sprays (T)

The high-pressure sprays in F-2 Centrifuge at T Plant became nearly completely plugged on Run T-5-10-D-7 and the plows were used instead of the sprays for the cake removal procedure on this and the following two runs. No difficulties of cake removal were experienced. The sprays have since been replaced.

Centrifuge E-2 Skimmer (T)

At the time of the second skimming of Centrifuge E-2 on Run T-5-10-D-8 at T Plant, it was found impossible to skim. The bowl was given a second 6% HNO_3 wash to reduce the fluoride heel and the cake was taken out without incident and sent through an adjusted-volume (3000-lb.) metathesis. Dismantling of the centrifuge revealed the skimmer to be sheared off completely at the first bend below the butt weld on the shaft. No evidence of welded seam split of the tubing could be seen, as was the case of the previous East Area breaks. A section of the tube will be examined by photo-micrographic examination after it has been decontaminated.

Isolation Building

Oxalate Method of Treating Recycles

Nine additional runs have been processed by this method. Losses remain low but apparent yields and material balances fluctuate widely. As previously mentioned this difficulty is believed to result from sampling mixtures which are not homogeneous but contain solids carrying disproportionate amounts of product. Whether the problem is primarily related to plant sampling or to the taking of laboratory aliquots of the plant samples is currently being investigated.

Increased Product Content of Recycle Solutions

The larger weight of product currently recycled to the Concentration Building was discussed in the two preceding reports. It has recently been observed that this change corresponded exactly with the adoption of the new E-2 cake removal procedure at the Concentration Buildings in which method hot 50% KOH is used to slurry the product LaF_3 cake out of the Cell E Centrifuge. This is shown in the following table:

Plant	Run Numbers	KOH Addition Point	Average Units Recycled
T	5-9-D-1 through F-12	Cell F	7.5
T	5-9-ECW-1 through F-30	Cell E	10.8
B	5-9-F-1 through EAW-4	Cell F	8.2
B	5-9-D-14 through F-26	Cell E	11.1

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The adoption of the new E-2 cake removal procedure was also accompanied by an increase in the iron content of P-1 solutions. This has also been previously discussed; it is not considered responsible for the higher recycle, however, since the P-1 iron content has slowly drifted downward while weight of product recycled has remiained high.

Current theories connecting the new metathesis conditions with higher recycles are that the more vigorous treatment results in either (a) more extensive oxidation of product during metathesis, or (b) formation of the "abnormal" form of product. If more extensive oxidation occurs, the present pre-reduction treatment may be incapable of completely reducing the larger amount of oxidized product.

As a result of several analyses of the H_2O_2 reagent, all of which showed 24 to 25% H_2O_2 instead of the 30% assumed in the Operating Log and Procedure, it has been decided to increase the weight of the weaker solution used. This change (from 15.75 to 18.9kg) was put into effect with Runs T-5-10-D-9 and B-5-10-B-13 and should reduce the weight of product recycled.

Volume of Wash Solutions

The purity of AT solutions was increased at Runs T-5-05-B-11 and B-5-05-D-15 by increasing the size and number of washes given the P-1 and P-2 cakes. The specification of three washes of 6 liters each in both cycles was felt to leave some margin for reduction in size of wash if this later became necessary. In view of the desire to increase the weight of H_2O_2 solution added in the first cycle (see above paragraph) and of the fact that the large catch tank CT-1 might not safely hold the increased volumes resulting, it was decided to save a few liters of volume by reducing the washes to 5 liters each. This change was made with Runs T-5-10-D-4 and B-5-10-B-7. The average AT purity for 8 runs since this change was 98.6%, while that for the 9 preceding runs was also 98.6%. No harmful effect of reduced washing is evident.

Backwashing of Nutsch (N-1) in Cell 4

When Run T-5-10-D-8 was sent into N-1 and agitated, considerable foaming occurred. The solution was observed to be a very dark color and a thin black cake was left on the nutsch after filtering the charge. The origin of this black mud is unknown; from appearance it might have been oil-coated filter-aid. No difficulties appeared in the processing of Run T-8, although the peroxide supernates had a dark color when combined in CT-1. To remove the mud, N-1 was leached with 60% HNO_3. About 35 units of product were obtained; this was returned to the Concentration Building and does not enter into Isolation Building accounting.

Process Chemistry

Film and Water Activities in the 100 Areas

Attempts to resolve the decay curves of the activities associated with various chemical fractions from the 100-D Area film (August 9, 1945 push) will be discontinued because of complications due to the presence of fission products. The presence of fission products was suspected when it became evident that certain decay curves could not be resolved satisfactorily without obtaining half-lives which could be attributed only to some fission product. This suspicion was further substantiated by an analysis of the calcium fraction for plutonium by precipitation of LaF_3. Approximately 400 c/m of alpha activity were found. Since all of the original solution has been used, it will not be possible to determine more accurately the

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amount of plutonium present but on the basis of 400 c/m representing 10% yield, it is calculated that 0.2 mg. of uranium as dust on the outside of the can could account for this much plutonium under normal conditions of pile operation, that is, 250 grams product/ton metal.

Activity in Air Condensers

During the past several months, there has been noticed a slow increase in activity level at the base of the air condenser on top of the 101 Tank (metal storage) in the 241 Area. To determine the cause of this activity, swipe samples were taken by forcing a stiff wire with a wad of wet glass wool on its end down into the air condenser tube to a distance of 16 feet from the top. The sample gave a 100 mr/hour reading for betas and 7 mr for gammas with the Beckman meter. To remove the activities from the swipe sample, the glass wool was treated with approximately 50 ml. of boiling 1N HNO_3 and the extract was filtered through a sintered glass filter. The glass wool was then further washed with 30 ml. of 1N HNO_3. This treatment removed all but approximately 2 - 3% of the beta activity originally present and gave an extract containing 2.5×10^5 beta counts/minute/ml. and 380 gamma counts/min./ml. (2nd shelf). Fission product analyses showed that approximately 66% of the total beta activity was due to rare earth and about 20% to Ba and Sr. This result indicates that the activity in the condenser tube is due to mist rather than some volatile fission product since it is difficult to see how these particular activities could get into the condenser tube by any mechanism other than as a mist or spray. Air blow-through following the completion of the jetting operation (from waste neutralizers to storage tanks) could account for the presence of the mist.

M. F. Acken

M. F. Acken, Chief Supervisor
200 Areas Technical Division

MFA:jd

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#1 R.M.Evans
#2 R.M.Evans
#3 B.H.Mackey for The Area Engineer
#4 B.H.Mackey for The Area Engineer
#5 B.H.Mackey for The Area Engineer
#6 B.H.Mackey for The Area Engineer
Attention: Patent Group
#7 N. Hilberry
#8 N. Hilberry
#9 N. Hilberry
#10 B.H.Mackey-D.A.Miller-700 Area
#11 M.H.Smith
#12 L.Squires
#13 300 Area File
#14 Pink Copy
#15 Yellow Copy
#16 M. F. Acken
#17 W. C. Kay - F. B. Vaughan
#18 R. S. Apple
#19 R. H. Beaton
→ #20 W. H. Sullivan
#21 G. W. Struthers
#22 J. D. Ellett
#23 J. J. Urban
#24 W. K. McCready

October 26, 1945

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Canyon Buildings

Coating Removal (B)

Several coating removal operations performed in the 3-5-R Dissolver in the B Canyon have reacted more vigorously than those performed in the 4-5-L Dissolver. During one coating removal (B-5-10-Bd-28, 29, 30) the reaction became vigorous enough to cause a brief loss of dissolver vacuum and excessive pressure drop across the column (in excess of 30 inches H_2O). The caustic addition rate on this charge, estimated from the weight factor charts, was approximately 45 lbs./min. instead of the specified 10 lbs./min. An inspection of the orifice in the caustic line to 3-5-R revealed a 3/16 inch orifice instead of the 5/32 inch orifice in the line to 4-5-L. Both dissolvers in the B Canyon now have 5/32 inch orifices in the caustic addition lines.

End Point of Metal Dissolving (T)

As a possible aid in lowering the extraction waste losses (8-3-W) by reducing the free nitric acid in the metal solution, the dissolvers are being run to a final specific gravity of 1.82 instead of 1.81. This change became effective in the T Canyon with dissolver charge T-5-10-Fd-31; it will not become effective in the B Canyon until the beginning of the next series. On the basis of limited data, this change adds 1 hour to the dissolving time for the first and second charge and 2 hours to the third charge.

Increased Sulfuric Acid Concentrations in the Extraction Strike (B and T)

The present extraction conditions are based on processing 1.5 tons of metal/batch; actual operation has approximated 1.35 tons of metal/batch. Since the sulfuric acid is added to the metal solution storage, the sulfuric acid concentration in the strike solution is dependent upon the weight of metal processed per batch; charges containing less than 1.5 tons of metal have a sulfuric acid concentration less than 1N. Analyses obtained on recent runs in both the B and T Canyons have shown a total acidity in the extraction solution of 0.8 - 0.9N. Approximately concurrent with this method of operation the background activity readings on the extraction catch tanks increased to, or exceeded, the full tank readings. These data indicate that the extraction waste solution is meta-stable with respect to the precip-

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itation of uranyl phosphate at these lower sulfuric acid concentrations and that some uranyl phosphate is precipitating on the surfaces of the catch tanks. In order to correct this condition, the quantity of sulfuric acid added to metal solution storage has been increased, effective with T-5-10-F-27 and B-5-10-D-34, to make the solution 1.0N with 1.35 tons/batch. No other change has been made in the extraction step conditions.

High Extraction Waste Loss (T)

Through error, the phosphoric acid strike on Run T-5-10-F-20 was made in 9 minutes instead of 3 hours. Although the digestion time was increased to 6 hours in an attempt to compensate for this short addition time, the waste loss was 8.0%. Operating conditions were such that the waste was discarded without reworking.

Neutralization of First Decontamination Cycle Wastes (B and T)

Following the satisfactory reduction of excess alkalinity in the second cycle wastes, the decision was made to reduce the alkalinity of the first cycle wastes. Based on the few data obtained thus far, the initial caustic reduction appears slightly excessive. A further adjustment will be made in an attempt to obtain a final pH closer to 7.0.

Neutralized Waste Storage (B and T)

The first storage tank in each series of three for the respective wastes is full, with the exception of the second cycle waste tank in B Plant, which is 86% full. A summary of the temperatures and reserve capacities in each area are presented below:

		Temperature (°F)					
		Metal Waste		1st Cycle		2nd Cycle	
Area	Date	101	102	107	108	110	111
T	5-21-45	93	--	83	--	83	--
	6-19-45	108	--	90	--	88	--
	7-17-45	118	--	94	--	88	--
	8-21-45	132	--	102	--	88	--
	9-20-45	138(full)	102	94(full)	86	90	--
	10-23-45	146(full)	92(66% full)	95(full)	90(53% full)	89(full)	93(17% full)
B	5-15-45	75	--	68	--	58	--
	6-19-45	97	--	75	--	71	--
	7-17-45	110	--	80	--	78	--
	8-21-45	136	--	90	--	84	--
	9-20-45	142	--	96	--	86	--
	10-23-45	138(full)	106(36% full)	100(full)	78(19% full)	82(86% full)	--

			Reserve Capacity as of 10-23-45			
			B Area		T Area	
Tanks	Type Waste	Gals./Run	Gallons	Runs	Gallons	Runs
X-101, -2, -3	Metal	5900	871,000	147	710,000	120
X-107, -8, -9	Ctng. & 1st Cycle	4700	960,000	204	780,000	166
X-110, -11, -12	2nd Cycle	3500	1,134,000	324	970,000	277

The spare metal solution tanks (X-104, -5, -6) have not been included in the above calculations of reserve storage capacity.

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Special Transfer Jet (T)

The special transfer jet to be used for jetting the neutralized wastes from the 241-T Tanks to the 241-U Tanks has been assembled and tested in Section 2-R of T Canyon. In the test, against a back pressure calculated to be equal to that of the line from 241-T to 241-U, the jet moved 30 gal. of water/min.

Centrifuge Motor Overloads (B)

The thermal overload relay on Centrifuge 16-2 (second cycle by-product) opened and failed to close during Run -10-B-14. The overload relay circuit was by-passed. A similar occurrence on Centrifuge 14-2 (first cycle product) took place during Run -10-D-25; this relay was also by-passed. The electrical department will install an overload relay on the motor leads to replace the inoperative relay within the motor housing.

Concentration Buildings

LaF_3 Product Centrifugation Rates

Test runs at both T and B Plants on Cell E time-cycle savings by increasing the LaF_3 product centrifugation through-put rates have demonstrated that the product waste (E-3-WS) losses are increased 0.1 to 0.2% by using 80 lbs./min. through-put on both centrifugations and approximately 0.1% by using 80 lbs./min. on the first and 70 lbs./min. on the second centrifugation. Accordingly, a return to the 70 lbs./min. rate has been made.

A summary of the test data is given below:

Effect of Cell E Centrifugation Rates on LaF_3 Product (E-3-WS) Waste Losses

Run No.	Total No. of Runs	Cent. Through-Put Rate (lbs./min.) Cent. #1	Cent. #2	% Product Loss (Ave.) (E-3-WS)
Sept. Ave. (T Plant)	30	70	70	0.87
T-5-10-D-3 to D-10	9	80	80	0.94
T-5-10-D-11	1	80	70	0.96
T-5-10-D-12 to F-17	6	70	70	0.78
Sept. Ave. (B Plant)	26	70	70	1.20
B-5-10-F-1 to B-10	10	80	80	1.40
B-5-10-B-11 to B-22	12	80	70	1.30

LaF_3 Product Cake Removal

The use of the hot KOH slurry removal of the LaF_3 product cake from Centrifuge E-2 to Metathesis Tank F-1 has continued to provide complete cake removal, as indicated by both the "empty" E-2 Beckman meter readings and the F-1-PS product assays. A set of 3 test runs was carried out at T Plant, using cold (43°C) KOH solution, in order to determine if the KOH could be used at less than the initially recommended 85°C. The first run did not completely empty the bowl, but the following two were successful. Since the successful runs followed the incomplete removal run, it is felt that some other factor contributed to the partial failure of the first run. At present, the KOH is being made up during the second Cell E centrifugation, a more convenient place for make-up than the previous on-the-spot make-up, and ranges in temperature from 60 to 85°C at time of use. Cake removal appears to be highly satisfactory.

A summary of recent E-2 Beckman data is given below:

Centrifuge E-2 Beckman Meter Readings ($x10^{-14}$ amps.)

			During Cake Removal Cycle				
			700 #	361# 50% KOH Slurry		500 #	
Run No.	Start (Empty)	Full	Water Spray	#1	#2	Water Spray	Remarks
T-5-10-D-10	2	52	17.5	16	10.5	6.8	Low Press. spray - KOH temp. = 43°C.
T-5-10-D-11	6	46	14	11.5	6	3	" " " " " "
T-5-10-D-12	3.5	76	22	20	5.5	2.7	" " " " " "
T-5-10-D-13	2.7	56	16.5	14.5	5	3.0	" " " " " 58°C.
T-5-10-F-14	3	54	21	19	8.7	3.5	" " " " " 59°C.
T-5-10-F-15	4	70	20	10	6	3	" " " " " 71°C.
T-5-10-F-16	2.5	46	8	6	0	0	" " " " " 75°C.
T-5-10-F-17	2	49	15	9	7	2.8	" " " " " 87°C.
B-5-10-B-16	0.5	100	26	7.5	0	0	Low-press. spray + hot (85°C) KOH
B-5-10-B-17	0	85	31	14	5	0	" " " " " "
B-5-10-B-18	0	97	40	21	4	0	" " " " " "
B-6-10-B-19	0	98	20	7	2	1	" " " " " "
B-5-10-B-20	0	91	29	17	8	1	" " " " " "
B-5-10-B-21	3	102	29	12	4	1	" " " " " "
B-5-10-B-22	1	54	26	23	11	2	" " " " " "
B-5-10-B-23	1	87	17	6	1	1	" " " " " "

E-2 High-Pressure Spray

Various tests of different sized orifices for the proposed high-pressure spray for E-2 Centrifuge has been continued in the gallery at B Plant. A 5-nozzle spray with 0.062 inch orifices delivered 6 gal./min. at 200 lbs./sq.in. pressure, but the spray was in the form of a very fine mist-not considered to be effective for LaF_3 cake removal. The orifices in this test model header were drilled out to 0.085 inches. This header produced a markedly better spray of 10 gal./min. at 195 lbs./sq.in. pressure, but the spray angle was decreased to 45°. A further drilling of the orifices to 0.106 inches produced a spray of 15 gal./min. at 190 lbs./sq.in., but the spray jets were very narrow and uneven in pressure distribution. It is believed that the milled face slots are far too narrow for these larger orifice drills, so they are being widened before further tests are made.

HF Spargers

Recently, the HF spargers in D-1 and E-1 Precipitators at B Plant were removed for inspection and replaced (as a matter of routine). Both sparger pipes (1-inch 25-12 welded tubing) showed a loss of 2-3 inches at the bottom with the terminal edges corroded to sharp edges (similar to a laboratory cork-borer). When the pipe had not yet been dissolved away at the bottom, the seam weld had been eaten out, to leave only a thin wall closure.

F-1-PS Assay Safety Checks

Due to the fact that (1) the present E-2 cake removal method has demonstrated a reproducible efficiency for a long enough period to cause a gain in confidence in the E-2 Beckman meter readings, and (2) the F-1-PS assay time has been causing repeated delays in the start of centrifugation of the successive run in E-2 Centrifuge, the former safety practice of awaiting the results of the F-1-PS assay on charge No. 1 before starting to centrifuge charge No. 2 in E-2 has been abandoned. Present practice permits centrifugation if the "empty" E-2 Beckman meter reading is below 5×10^{-14} amps. and a delay for the F-1-PS assay is observed only if the Beckman reading is higher than this limit.

Cell E Time Cycle

The LaF_3 product precipitation (Cell E) procedure has been brought up to date and the new log procedures are now being used at both T and B Plants. Significant time-cycle savings should be obtained. The first run through T Plant on this process (T-5-10-D-17) had an over-all unit time (in-and-out of cell) of 16-1/2 hours. Overlapping of the following charge was such that the net operating time of Run D-17 in Cell E (successive jettings from D-3 to E-1) was 15-3/4 hours. Further monitoring will be carried out in both areas.

Isolation Building

Oxalate Method of Treating Recycles

A survey of the data for the first 34 runs processed in Cell 4 by the oxalate method of handling wastes shows satisfactorily low losses, but erratic material balance and yield figures. A summary follows:

Sample Code Description	CT-1-W Initial Waste	F-2-S 1st Supernate	F-2-SW 2nd Supernate	CT-1-R Final Recycle	Mat. Bal.
% of CT-1-W	100	1.87	1.18	93	96
% of 8-1-MR	4.09	0.077	0.048	3.81	3.94

To shorten time cycles and decrease losses, it was proposed to omit the water wash of the lanthanum oxalate cake. After three dummy runs in Cell 2 showed no unexpected difficulties, the change was made in active plant runs in Cell 4, starting with T-5-10-D-10. Four runs complete at this writing were carried out satisfactorily. Total losses were 2% of CT-1-W. The total time per run varied from 7 to 9 hours as compared with 10 to 13 hours for the former procedure. Runs will continue.

Increased Product Content of Recycle Solutions

No reason has been found for the rise in weight of product recycled to the Concentration Building recently observed. To combat the effect, the weight of H_2O_2 solution added in the first cycle at the Isolation Building was increased with Runs T-5-10-D-9 and B-5-10-B-13. This precipitation is now made at about 9% H_2O_2. The effect of increasing the H_2O_2 concentration has been slight in Cell 3 (B Plant runs) but definite in Cell 4 (T runs), as the following table shows:

Plant	Run Numbers	Average Units Recycled
T	5-10-F-1 thru D-8	13.5
T	5-10-D-9 thru F-14	9.8
B	5-10-F-1 thru B-12	9.9
B	5-10-B-13 thru B-20	9.7

In neither cell, has the amount recycled been reduced to its level for runs before the recent change in metathesis procedure in the Concentration Buildings.

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The theory that the new metathesis procedure results in more extensive oxidation of product so that the ammonium sulfite concentration in P-1 is not sufficient for complete reduction seems discredited by the report that only 18% of the product in P-1 of Run TD-3 was oxidized while 17.3% of that in B-B6 was oxidized. It may not be coincidental, however, that the weights recycled in these two runs were only 8.7 and 9.6 units, respectively.

Volume of Wash Solutions

The reduction in wash volume (from 6 to 5 liters) for all six washes has not decreased final purity of AT solutions. The latter have averaged 98.8% for 25 runs since the change discussed in the report dated October 16, 1945.

Process Chemistry

Film and Water Activities in the 100 Areas

Studies since the last report have not confirmed as yet the suspicion that the alpha activity present in the films was due to product. Although the alpha activity is carried by LaF_3, tests to determine its behavior in oxidized solutions have not shown satisfactorily that the activity is due to product. Further studies are in progress.

Soil Retention Studies

Because of the inconsistent results in day to day operation of the column, attention will be directed primarily to the determination of the total capacity of the soil rather than an accurate rate capacity for product adsorption. On this basis the 110 Tank waste solution is again being passed through the column continuously. When complete break-through of product is obtained, the amount adsorbed on the soil will be calculated and subsequently a series of elution studies will be made.

M. F. Acken, Chief Supervisor
200 Areas Technical Division

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#24 W.K.McCready

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200 AREAS
October 23 Through October 29

Canyon Buildings

High Basis Assay (B)

As has been mentioned previously, the basis assay (7-1-MR) in the B Canyon continues to average approximately 5% higher than the calculated quantity transferred from 6-1 or the sum of the extraction product (7-4-P) and extraction waste (7-3-W). The performance of the precipitator spray was checked, by means of temperature measurements, and found to be circulating the dissolving acid. On a series of five runs, the 20-minute spray period was increased to 30 minutes without decreasing this discrepancy:

Run No.	Transferred from 6-1	7-1 Assay	7-4-P + 7-3-W
-10-D-30	100%	108.5%	99.0
-31	100%	106.5	99.0
-32	100%	106.5	101.0
-33	100%	104.0	98.5
-34	100%	106.5	101.5

The solution transferred from 6-1 is diluted further in 7-1 before the 7-1-MR sample is taken. If the circulation rate of the sampler was low, it would be possible to obtain a non-representative, high concentration sample. Conversely, on this assumption, if the dilution water were added to the tank before the metal solution, a non-representative, low concentration sample would be obtained. In a series of five runs, this was not the case:

Run No.	Transferred from 6-1	7-1 Assay	7-4-P + 7-3-W
-10-D-33	100%	104.0	98.5
-34	100%	106.5	101.5
-35	100%	110.0	99.5
-36	100%	106.0	97.5
-37	100%	103.5	98.5

The 7-1 precipitator is being re-calibrated again in an attempt to locate the cause of this trouble.

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Extraction Waste Loss (T)

Ten extractions have been made in the T Canyon in which the sulfuric acid concentration was increased to 1.0N and the metal solution was dissolved to an end point of 1.82 sp.gr. The average waste loss on these runs was 0.93% as compared to 1.25% for a like number of previous runs. Similar data from the B Canyon are not available at this time. No discernible effect has been produced on the activity level of the extraction catch tanks.

Lower Decontamination (B)

The decontamination obtained on most of the runs in the B Canyon since B-5-10-B-20 has been lower than normal and on Runs -D-29 through -D-34 it was below standard. That this was real was reflected in higher Beckman readings on the tank in Sections 16, 17 and Cell C in the Concentration Building. A preliminary check of the operating procedures revealed nothing unusual. Decontamination factors are being determined at the end of the extraction step and first decontamination cycle in both the B and T Canyons in an attempt to determine where the loss of decontamination occurred.

Time Cycles (B and T)

During the month of October, 40 charges were started in the B Canyon and 38 in the T Canyon. In the T Canyon, two days were lost due to equipment limitations, making an average time per run of 18-1/2 hours; in the B Canyon one day was lost to equipment limitations, making an average time per run of 18 hours.

Slow Speed Skimming (B)

Tests performed on the centrifuge in Section 18 in the B Canyon indicate a maximum hydraulic pressure on the skimmer of approximately 150 lbs./sq.in. when skimming at 870 rev./min. and approximately 100 lbs./sq.in. when skimming at 650 rev./min. Slower speed skimming will be tested on the extraction and first cycle product centrifuges to determine the effect on product loss during skimming and washing operations.

Concentration Building

F-1-PS Assay

Analytical and/or sampling difficulties are still being encountered with F-1-PS samples. On a series of runs in the East Area, two special F-1-PS samples will be taken which are expected to give more reproducible results. These samples come later in the process but are feasible time-cycle-wise due to the fact that the F-1-PS assay is no longer required prior to starting centrifugation of the following run in Cell E if the E-2 empty Beckman reading is sufficiently low. The special samples are: F-1-PS-2 to be taken at the end of the 90 minute metathesis digestion; and F-1-PS-3 to be taken during the 2% KOH metathesis wash digestion. Reproducibility of results and ease of analysis of these samples will be compared.

Slow Speed Skimming

With the start of slow speed skimming in A-2 (Run B-5-10-D-35) all 40 inch centrifuges in the East Area Concentration Building are being skimmed at low speed (approx. 200 rev./min.). In the West Area slow speed skimming is standard procedure only for the LaF_3 by-product centrifuge (D-2). Slow speed skimming was tried

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in the LaF_3 product centrifuge (E-2) on three runs (T-5-10-F-24 through F-26). High E-3-WS waste losses resulted on two of these runs (3.3% and 2.84%) and high speed skimming was reinstated as a check on other parts of the process. Runs T-5-10-F-27, F-28 and F-29 skimmed at high speed returned normal E-3-WS waste losses. Beginning with T-5-10-F-30, three more runs will be made skimming at low speed. There is no obvious explanation for these high losses obtained with slow speed skimming as this process has been used successfully in the East Area.

Cell E Time Cycle

The time cycle for the LaF_3 product precipitation (Cell E) procedure has been followed in both areas since the adoption of the new log procedures. Excluding one West Area run (T-5-10-F-26) which required a third centrifuging to bring the E-3-WS product loss within standard limits, all times have been appreciably below the 21-hour limit set as satisfactory to meet future production schedules.

E-2 High-Pressure Sprays

The five-nozzle spray being tested in the gallery at B Plant has been judged satisfactory for trial and will be installed in E-2. These nozzles have orifices of 0.106 inches and will deliver 0.5 gal./min. at 190 lbs./sq.in. pressure. The performance of the spray was greatly improved by widening the milled face slots which were designed for an 0.062 orifice.

Concentration Building Waste (T)

A random sample of the neutralized wastes from the Concentration Building was obtained from the 361-T Dry Well for the purpose of comparing the alpha, beta and gamma activity therein with that expected in the second decontamination cycle wastes when the pH is adjusted to approximately 7. The analysis of the 361 Dry Well sample was:

A - 60 counts/(min.)(ml.)
B - 1800 counts/(min.)(ml.)
C - 24 counts/(min.)(ml.)

This is higher than the expected count in the second cycle waste when adjusted to neutrality.

Isolation Building

Oxalate Method of Handling Recycles

Evaluation of this procedure is still handicapped by erratic loss and material balance data for individual runs. This is believed to result mainly from sampling difficulties in dealing with solutions containing suspended solids which carry product. When the data for a large number of runs are averaged, the gross fluctuations disappear and a satisfactory picture is presented. A summary of the 49 runs completed to date follows:

Sample Code	CT-1-W	F-2-S-1	F-2-S-2	CT-1-R	
Description	Initial Wa*te	1st Supernate	2nd Supernate	Final Recycle	Mat.Bal.
% of CT-1-W	100	2.34	1.09	94.7	98.1
% of 8-1-MR	3.99	0.093	0.043	3.78	3.92

* Data for T-5-9-D-24 were omitted as obviously erroneous

In an effort to obtain better samples of the two wastes on a few runs, nitric acid will be added to the wastes in F-2 to dissolve the precipitates. Samples of these true solutions will be assayed for comparison with F-2-S-1 and F-2-S-2 as obtained by sampling the alkaline "solutions". If the assays on the special samples agree with or are lower than those on the standard samples, it will be recommended that the oxalate method be adopted for all production. If the special samples contain appreciably more product than the standard samples the test should be repeated with the expectation of abandoning the oxalate method if losses are above about 5% of the CT-1-W assay.

In the report of October 23, there were presented the preliminary results of omitting the water wash from the oxalate procedure. Fifteen such runs have now been completed with the expected results. Time cycles have averaged 8 hours per run as compared with the former 10. Losses in the second waste (now coded F-2-S-2 instead of F-2-S-W) were about 70% of their former magnitude. The amount of $KMnO_4$ required for final oxalate destruction has been practically unchanged as has the final recycle volume. The wash has been permanently discontinued.

Product Content of Recycle Solutions

The recent increase in weight of H_2O_2 added in the first cycle at the Isolation Building has reduced the weight of product recycled per run, though not as much as the recycle was increased by the previous change in metathesis procedure at the Concentration Building. This is shown by the following table, part of which represents a repetition of data previously reported:

Plant	Run Numbers	KOH Added in	H_2O_2	Av. Units Recycled
T	5-9-D-1 through F-12	Cell F	15.75kg.	7.5
T	5-9-ECW-1 through 5-10-D-8	Cell E	15.75	11.5
T	5-10-D-9 through D-23	Cell E	18.9	9.4
B	5-9-F-1 through EAW-4	Cell F	15.75	8.2
B	5-9-D-14 through 5-10-B-12	Cell E	15.75	10.5
B	5-10-B-13 through D-28	Cell E	18.9	9.5

Flush of Head Tanks

The procedure recently suggested for flushing of these tanks was instituted with Runs B-5-10-D-26 and T-5-10-D-20. This involves sending 1 liter of solution (either 1.2% HNO_3 or such PR-3 overflow as ST-2 contains) from ST-2 to PR-1 to N-2 to PR-2 to P-2; the flush is made just before time to move a charge from P-1 to P-2. Other slight modifications in procedure have also been made with the same objective, namely prevention of the compositing of two charges.

Process Chemistry

Soil Retention Studies

During a 5-day period of continuous operation about 3-1/2 gallons of effluent from the soil test column were collected. The flow rate, which had dropped to approximately 1.6 ml./minute prior to this run, was fairly constant during this period of collection. Samples have been prepared for alpha and gamma analyses.

Isolation of Europium

The new run using 10 ml. of 6-1-M solution has progressed to the point where

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europium is to be separated from the rest of the rare earths in a reduced valence state. This operation will be carried out twice. The sample will then be checked radiochemically for purity by absorption measurements. Dummy runs were made to develop techniques which will be used in the reduction procedure so as to minimize radiation hazards.

M F Acken per GED

M.F.Acken, Chief Supervisor
200 Areas Technical Division

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#2 R.M.Evans
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November 9, 1945

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200 AREAS
October 30 Through November 5

Canyon Buildings

High Basis Assay (B)

A re-calibration of the 7-1 Precipitator in the B Canyon checked the previous calibration, thereby eliminating the possibility of an erroneous tank calibration as the cause of the high 7-1-MR basis assays. Exploring further the possibility suggested last week, that the circulation rate of the 7-1 Sampler was abnormally low, resulting in concentration of the warm solution by evaporation under vacuum, a series of runs is being made on which the 7-1-MR Sample is taken at 35°C instead of 75°C or 85°C. Five runs have been made under these conditions with greatly decreased discrepancy between the 6-1-MR and 7-1-MR Assays:

Canyon	Temp.	Runs Avg.	Transferred from 6-1	7-1 Assay
B	85°	10	100%	106%
	35°	5	100%	101%
T	85°	10	100%	99.8 (8-1 assay)

The better assay checks obtained at 85°C in the T Plant than at B may possibly be due to a restriction in the 7-1 sampler circulating system in the B Plant which would result in increased evaporation (and therefore erroneously high product concentration) at the high temperature employed. By the same reasoning, the improved results obtained at 35°C might be explained on the basis of less evaporation of the solution being circulated.

Lower Decontamination (B)

Decontamination factors for the extraction step and the decontamination cycles have been obtained on a number of recent runs in each canyon in an effort to determine the point and cause of the lower decontamination in the B Canyon.

		Arithmetic Factors					
		Extraction		1st Cycle		2nd Cycle	
Area	Runs	Avg.	Range	Avg.	Range	Avg.	Range
B	7	5.9	4.8-6.9	300	257-380	55	42-69
T	4	5.7	5.0-6.8	345	270-535	88	65-98
B	06 Series	7.6		347		87	
T		8.5		380		85	

These data show that there has been some loss of decontamination in the extraction step in each canyon since June, that the first cycle factors have remained constant and that the greatest loss of decontamination has occurred in the second cycle in the B Canyon. The investigation of this problem is being continued.

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High Second Cycle Waste Loss (B)

The dissolving acid for the second cycle product cake of Run- -10-D-37 was prematurely jetted from the precipitator to the centrifuge, overflowing into the catch tank. This acid dissolved some precipitate and increase the waste loss to 5.7%. The waste was discarded without reworking.

Slow Speed Skimming (B)

Skimming at 650, instead of 870 rev./min., is being continued on the extraction cake (7-2) and first cycle cake (14-2). Waste analyses fail to show any significant change as a result of the reduced skimming speed.

Ventilating Fan (B)

The No. 1 30,000 cu.ft./min. Ventilating Fan (291 Bldg.) in the East Area developed noise and a slight vibration during the past week. As a result, it was shut down to check the bearings. Operations are being continued using only one fan.

Concentration Buildings

F-1-PS Assay

The special F-1-PS Samples, taken in the East Area in an attempt to obtain a sample which would be easier to analyze, have been discontinued. Few definite conclusions can be drawn because the control sample gave no difficulties during the test. Judging by ease of analysis and material balances, the F-1-PS-1 (regular sample taken prior to metathesis digestion) and F-1-PS-2 (taken at the end of metathesis digestion) are comparable. The F-1-PS-3 (taken during the metathesis wash digestion) was more difficult to analyze, slightly erratic in results and was discontinued after three runs. These difficulties may be due to inadequate laboratory procedure but no further work is planned because this sample has disadvantages from an operation viewpoint. If difficulty is again encountered with the regular F-1-PS, F-1-PS-2 will be taken for another series of runs.

Slow Speed Skimming

In the East Area all 40-inch centrifuges are still being skimmed at slow speed (approximately 700 rev./min.) with good results. In the West Area a second slow speed skimming trial has been made in the LaF_3 Product Centrifuge (E-2). The E-3W product waste losses have remained normal and slow speed skimming is being adopted as regular procedure.

E-2 High Pressure Sprays

The five nozzle spray scheduled for installation in E-2 (East Area) has been delayed until the fifth nozzle (a spur nozzle designed to spray the bottom of the centrifuge) can be modified so that the spray dip tube can be installed. The spray assembly was tailored to fit B-2 Centrifuge but lacks 1/16-inch clearance for installation in E-2.

Beckman Limits

The change in location of the GE tube at F-10 Tank has changed the geometry such that the full tank readings are approximately three times the old reading. The Beckman limit for the empty F-10 Tank has been raised from 20×10^{-14} to 30×10^{-14} amps.

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Isolation Building

Oxalate Method of Handling Recycles

There has been no change in the erratic loss and material balance picture previously discussed. Some data have been obtained which indicated that the waste assays reported from the laboratory were of the proper order of magnitude on two runs checked, but were in error by a factor varying from 1.3 to 5. This was done by adding HNO_3 to both the wastes, F-2-S-1 and F-2-S-2, after sampling and taking new samples, F-2-A-1 and F-2-A-2. The amount of HNO_3 was sufficient to furnish a clear solution one normal in nitric. Samples of this solution were considered more reliable than samples of the alkaline solution which contains suspended solids that carry product. Data follow:

Run	Sample	Assay	Volume	Units
T-5-10-F-27	F-2-S-1	0.000099	84	0.008
T-5-10-F-27	F-2-A-1	0.00028	94.6	0.0265
T-5-10-F-27	F-2-S-2	0.00016	55.8(?)	0.009
T-5-10-F-27	F-2-A-2	0.00056	78.4	0.044
T-5-10-B-31	F-2-S-1	0.00122	78.9	0.096
T-5-10-B-31	F-2-A-1	0.00145	91.5	0.133
T-5-10-B-31	F-2-S-2	0.00203	75	0.152
T-5-10-B-31	F-2-A-2	0.00226	87.2	0.197

It is significant that the acidified samples checked the alkaline samples more closely on the higher level waste of Run T-31. It is also of interest that the acidified sample assayed higher in each case than the alkaline sample. The results in general would seem to substantiate the approximate accuracy of the alkaline samples taken in the past, but warrant further such checks. It is hoped that this will be possible. Routine acidification before sampling is not operationally feasible with present equipment, however.

Product Content of Recycle Solutions

Recent checks of the concentration of the reagent H_2O_2 showed it to be up to 30% again, so it was decided to decrease the weight of the reagent added in the first cycle to the amount used before it was recently increased to combat lower H_2O_2 strength. This was done with Runs T-5-10-B-36 and B-5-10-D-41. Shortly thereafter another carboy of 26% H_2O_2 was found. It is planned to analyze each carboy for about a week and compare the weight of product recycled with the H_2O_2 strength, using the same weight in each run. If it develops that H_2O_2 strength has enough bearing on the weight recycled to warrant routine analysis of the reagent, a procedure will be instituted whereby the desired weight of H_2O_2 for addition will be determined by reference to a chart or graph which takes into account weight of solution (as at present) and concentration of H_2O_2.

P-1 Acidity

The average P-1 acidity for the first 74 runs in the 5-10 Series was 1.98N. This is very close to the desired 2.0N, but there was considerable spread of values, one-third being outside the range of 1.8 to 2.2N and therefore requiring adjustment.

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Oct. 30-Nov. 5 '45

Process Chemistry

Investigation of Film in Water Activities in 100 Area

An analysis for the 12.5-day Ba activity was made in the "calcium" fraction from the 100-B Area film. No Ba was detected in this fraction 24 days after the push. An analysis of the initial emulsion indicated that radioactive Zr might be present. However, if the activity is due to Zr it is not the only activity present as indicated by the complex decay curve. Over 10 days of decay the apparent half-life was approximately 55 days. The chemical procedure which was followed should eliminate the possibility of other fission products being present but it does not exclude the possibility that other activities, e.g. iron, are carried along. Further work will be carried on to identify this long-lived constituent.

Despite the rather elaborate chemical operations to isolate individual elements in the film from the 100-B Area on October 2, the decay curves for the activities appear to be too complex for accurate resolution. However, from the work conducted thus far, the following conclusions were reached:

1) There is strong evidence for the 42-day Fe^{59} activity in the ether-oil phase obtained by breaking the original oil emulsion and in the ether phase from the step designed to remove the iron.

2) There is strong evidence for the presence of a 67-hour activity (possibly Mo^{99}) in the two ether fractions.

3) There is also strong evidence for a 12.8-hour activity (possibly Cu^{64}.) This activity was equally divided between the aluminum fraction (obtained by the precipitation of $Al(OH)_3$) and the sodium fraction (the supernatant of the $Al(OH)_3$ precipitation).

4) The resolution of the complex gamma ray decay curve associated with the Mn fraction shows that a 2.6-hour activity, which is probably Mn^{56}, is present. This activity is not due to Ca^{49}, since the beta activity of the calcium oxalate precipitated from the Mn fraction is only 0.1 as intense as the 2.5-hour activity in the Mn fraction.

Soil Retention Studies

An additional approximately 4-1/2 gallons of the 110 Tank waste solution have been passed through the test column. Analyses of samples collected during the past two weeks are in progress.

Isolation of Europium

Operations to isolate europium have now progressed through two reduction and two lanthanum hydroxide by-product steps. These operations would ordinarily have been sufficient to strip the remaining rare earths from europium, but in transferring the first supernatant from the lanthanum hydroxide by decantation after centrifuging, some of the precipitate was transferred also. The second by-product precipitation reduced the gamma activity in the europium to less than 25 mr/h, but it will be necessary to conduct a third by-product precipitation to obtain complete decontamination.

Mass Spectrograph Studies of Isotope 95^{241}

A sample of isotope $95^{(241)}$ from the B-8 (5-07) AT Solution in the Isolation

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Building was obtained in the usual manner by successive peroxide precipitations with the element 95 being concentrated in each supernatant. Operations have progressed now to the micro scale and the element 95 will be further purified from the approximately 1 mg. of plutonium now associated with it. This material in its final form will be submitted to analysis in the mass spectrograph.

130-Day Tin Activity

In an experiment to isolate antimony from an old sample of tin activity, it was observed that only a very small percentage of the total tin activity was found in the antimony fraction. Thus, it would appear that the 130-day tin activity decays either to a stable antimony isotope or to an isotope of very long half-life and/or having very weak radiations.

M. F. Acken

M.F.Acken, Chief Supervisor
200 Areas Technical Division

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HW-3-2758

#1 R.M.Evans
#2 R.M.Evans
#3 B.H.Mackey for The Area Engineer
#4 B.H.Mackey for The Area Engineer
#5 B.H.Mackey for The Area Engineer
#6 B.H.Mackey for The Area Engineer
Attention: Patent Group
#7 N. Hilberry
#8 N. Hilberry
#9 N. Hilberry
#10 B.H.Mackey-D.A.Miller-700 Area
#11 M.H.Smith
#12 L.Squires
#13 300 Area File
#14 Pink Copy
#15 Yellow Copy
#16 M.F.Acken
#17 W.C.Kay - F.B.Vaughan
#18 R.S.Apple
#19 R.H.Beaton
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#21 G.W.Struthers
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November 16, 1945

200 AREAS

November 6 through November 12

This Document consists of [redacted] Pages No.

Canyon Buildings

End Point of Metal Dissolving (B)

To conform with the change made in the T Canyon with dissolver charge T-5-10-Fd-31, the end-point of metal dissolving in the B Canyon was changed from a final specific gravity of 1.81 to 1.82, effective with dissolver charge B-5-11-Dd-11. Data are being accumulated to determine whether this change has had any effect on extraction waste losses.

Basis Assay (B)

Sampling the 7-1 Precipitator at 35° instead of 85° has been continued for a total of 11 runs. Agreement between the quantity transferred from 6-1 and the assay in 7-1 has been better than before, ranging between 98.5% and 104.0%, averaging 102%. The fact that the assays check more closely at the lower temperature lends support to the theory that normal circulation rates are not being obtained with the 7-1 Sampler. This is being investigated further.

Lower Decontamination (B)

A series of seven runs was made in the B Canyon in which the extraction precipitation was made in 0.6M H_3PO_4 instead of 0.8M acid. No significant effect upon the decontamination factors was observed. The average factor for these runs was 5.09; for the seven previous runs made at 0.8M acid, the average factor was 4.96.

Acid Washes (B and T)

An acid wash, duplicating the ones made in August, has been made in each canyon. The results obtained, together with those obtained in the August washes, are presented below. The most significant data in the table are the small quantities of product picked up in any one Section or vessel. Before the August wash in the T Canyon, a special procedure (metathesis) was employed on the two runs immediately preceding the wash, to insure a minimum product heel in the first and second cycle product precipitators. No such precautions were made before the current wash. The acid wash produced only slight changes in the activity level of the empty equipment pieces (Beckman backgrounds) in the T Canyon.

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Acid Washes - B Canyon

Sample	Description	August Wash B-5-08-AW-1 Total	August Wash B-5-08-AW-1 % Sec.** Increase	November Wash B-5-11-AW-1 Total	November Wash B-5-11-AW-1 % Sec.** Increase
7-1-P	Wash acid in extraction precipitator (8-1, 7-1)	0.14		2.7	
7-4-P-1	Wash acid after transfer from precipitator through centrifuge.	0.20		4.1	
7-4-P-2	Wash acid after agitation in solution tank (8-4, 7-4)	0.40*	0.40	3.9*	3.9
12-6-P	Wash acid after agitation in crude product storage tank (12-6)	0.72*	0.32	No Sample	
12-7-P	Wash acid after agitation in oxidation tank (12-7)	0.72*	0.0	5.9*	2.0
13-1-P	Dissolving acid after circulation in 1st by-product precipitator (13-1)	0.01		0.73	
13-4-BP	Regular waste sample on AW	0.05		0.79	
13-3-P	Product Sample on acid wash	0.79*	0.07	5.9*	0.0
14-1-P	Wash acid in 1st product precipitator (14-1)	(3.9)		(3.8)	
14-4-P-1	Wash acid after transfer from precipitator through centrifuge (14-2)	1.7		2.5	
14-4-P-2	Wash acid after agitation in solution tank (14-4)	1.7		2.5	
14-4-P-3	Regular product sample on AW	2.6*	1.81	4.4*	0.0
14-3-W	Regular waste sample on AW	0.01		0.03	
16-1-P	Dissolving acid after circulation in 2nd by-product precipitator (16-1)	0.02		0.03	
16-4-BP	Regular waste sample on AW	0.06		0.09	
16-3-P	Product sample on acid wash	3.3*	0.7	8.7*	2.8
17-1-P	Wash acid in 2nd product precipitator (17-1)	(4.4)		(10.9)	
17-4-P-1	Wash acid after transfer from precipitator through centrifuge (17-2)	1.5		1.8	
17-4-P-2	Wash acid after agitation in solution tank (17-4)	1.5		1.7	
17-4-P-3	Regular product sample on AW	4.4*	1.1	10.4*	1.7
17-3-W	Regular waste sample on AW	0.02		0.04	

* Cumulative values.

** Based on previous run (280 units).

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Acid Washes - T Canyon

Sample	Description	T-5-08-AW-1 Total	T-5-08-AW-1 % Sec.** Increase	T-5-11-AW-1 Total	T-5-11-AW-1 % Sec.*** Increase
8-1-P	Wash acid in extraction precipitator (8-1)	0.54		0.89	
8-4-P-1	Wash acid after transfer from precipitator through centrifuge (8-2)	0.88		0.67	
8-4-P-2	Wash acid after agitation in solution tank (8-4)	0.86*	0.86	0.65*	0.65
12-6-P	Wash acid after agitation in crude product storage tank (12-6)	3.48*	2.62	4.14*	3.49
12-7-P	Wash acid after agitation in oxidation tank (12-7)	3.37*	0.0	4.49*	0.35
13-1-P	Dissolving acid after circulation in 1st by-product precipitator (13-1)	0.40		0.09	
13-4-BP	Regular waste sample on AW	0.55		0.16	
13-3-P	Product sample on acid wash	3.44*	0.0	4.35*	0.0
14-1-P	Wash acid in 1st product precipitator (14-1)	0.53		(42.7)	
14-4-P-1	Wash acid after transfer from precipitator through centrifuge (14-2)	0.44		3.67	
14-4-P-2	Wash acid after agitation in solution tank (14-4)	0.41		3.78	
14-4-P-3	Regular product sample on AW	3.72*	0.28	7.90*	3.55
14-3-W	Regular waste sample on AW	0.05		0.73	
16-1-P	Dissolving acid after circulation in 2nd by-product precipitator (16-1)	0.02		0.02	
16-4-BP	Regular waste sample on AW	0.15		0.14	
16-3-P	Product sample on acid wash	3.85*	0.13	8.59*	0.69
17-1-P	Wash acid in 2nd product precipitator (17-1)	0.28		(12.1)	
17-4-P-1	Wash acid after transfer from precipitator through centrifuge (17-2)	0.44		2.14	
17-4-P-2	Wash acid after agitation in solution tank (17-4)	0.43		2.11	
17-4-P-3	Regular product sample on AW	4.80*	0.95	12.0*	3.41
17-3-W	Regular waste sample on AW	0.05		0.06	

* Cumulative values.
** Based on average of previous runs (287 units).
*** Based on average of previous runs (273 units).

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Slow Speed Skimming (B)

Slow speed skimming (650 rev./min.) on the extraction (7-2) and first cycle product (14-2) centrifuges was discontinued on Run B-5-11-D-10. No consistent effect on waste losses was observed.

Ventilating Fan (B)

The inboard bearing on the No. 1 30,000 cu.ft./min. Ventilating Fan in the East Area has been replaced (5 minutes/man working time) and the fan is ready for testing. No obvious problems have arisen during the 1-week period in which the plant operated through necessity, with only one ventilating fan in service.

Concentration Buildings

LaF_3 Product Cake Removal

Use of the hot KOH slurry method of LaF_3 product cake removal from Centrifuge E-2 at T Plant has continued to provide efficient cake removal. Empty E-2 Beckman meter readings have been below 3×10^{-14} amps. for the last 7 runs.

At B Plant, the new 5-nozzle high-pressure spray header has been installed in E-2 and tested. This spray, when connected to 2 Taber pumps in series, delivers approximately 15 gal./min. at a pressure of 190 lbs./sq.in. The fifth nozzle is a spur nozzle designed to spray the floor of the centrifuge bowl.

Four runs (B-5-11-B-4-5-6-7) were carried out using both the high pressure spray and the hot KOH slurrying. The Beckman meter readings at the end of the initial 700-lb. water spray were reduced to an average of 5×10^{-14} amps., whereas the low pressure spray usually gives a reading of about 20×10^{-14} amps. at this point. Final readings after the KOH slurry-water spray cycle were all lower than 1×10^{-14} amps.

In an effort to determine if straight water spraying alone will provide satisfactory cake removal, the next 3 runs are being made in this manner, with no KOH slurry treatment.

LaF_3 By-Product Waste Losses

Recently it has become more noticeable at T Plant that there is a spread between the LaF_3 by-product (D-4-BP) waste losses for runs carried out with and without recycling respectively. The average loss for October was 0.92% (corrected to performance basis) for runs containing a recycle of 9.0 lbs. of lanthanum. The average of 5 runs made without recycling (6.0 lbs. of gallery lanthanum) was 0.64%. Both types of precipitation involve the use of a double-batch slurry cake wash. The October average for B Plant (6.0 lbs. of gallery lanthanum plus displacement wash) was 0.73%. In order to determine if the difference in waste losses is due alone to the larger cake in the recycling process, the next five runs at T Plant (T-5-11-F-11-15) will be carried out with a 6.0 lb. lanthanum recycle.

Acid Wash Runs

Following Run 11-B-8 at both T and B Plants, a full-volume acid-wash run was carried through the Concentration Buildings, originating in the Canyon Buildings. At present, the T Plant run (T-5-11-AW-1) is the only one completed. This run arrived at the Concentration Building with 12% product pick-up and gained an additional pick-up of approximately 8% in the course of passing from Centrifuge E-2 into and through Cell F.

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The data are summarized below:

T Plant Concentration Building Acid Wash Run (T-5-11-AW-1)

Process Vessel or Step	Sample Code	Product Balances Total % 1.95×10^{13} T.C.	% Pick-Up
Basis			
Canyon Building	17-4-P	12.0	--
C-4	C-4-0	12.5	+0.5
A-1	A-1-0	13.9	+1.4
$BiPO_4$ B.P. Loss	A-4-BP	0.30	--
A-3	A-3-OS	12.6	-1.0
D-1	D-1-0	12.7	+0.1
LaF_3 B.P. Loss	D-4-BP	0.07	--
D-3	D-3-OS	13.2	+0.6
E-1	E-1-R	11.9	-1.3
LaF_3 Prod. Waste	E-3-WS	0.03	--
F-1 (plus E-2 to F-1 line)	F-1-PS	20.0	+8.1
15% KOH Metathesis Loss	F-7-WS	0.10	--
2% KOH Metathesis Loss	F-9-WS	0.05	--
Final Yield	F-10-P	21.0	+1.1
		Net Total Pick-Up	+9.3

Note: % Product pick-up corrected for waste losses.

E-2 Skimmer

During the completion of the LaF_3 product cycle in Cell E at B Plant on Run B-5-11-B-7, it was found impossible to skim Centrifuge E-2. Subsequent inspection of the bowl after cake removal showed the skimmer to be torn and bent at the curve from the vertical to horizontal section. This skimmer pipe failed after approximately 75 runs had been carried through the cell, almost exactly the same number of runs which had been processed with the skimmer which failed previously at this point. The second skimmer had been used for the greater part of its operational life at bowl skimming speeds of 650-700 rev./min., as compared to the use of the initial skimmer at 870 rev./min.

The third skimmer which has been installed possesses additional bracing at both the vertical section and at the main plane-bend.

Metallurgical inspection of the previously broken skimmer pipes revealed that all of them were extensively corrosion-pitted in the fusion zone between the weld and the base metal along the inside of the pipe. In some cases, approximately 75% of the wall thickness was dissolved away in this zone. Laboratory modifications of heat-treatment have been unsuccessful. Their recommendations are that future skimmer pipes be made from seamless pipe obtained by drilling out 25-12 bar stock or that welded tubing, if used, be heavily reinforced and braced to absorb applied stresses.

Isolation Building

Relationship of High Recycles and H_2O_2 Concentration

In an effort to determine the cause for the generally higher recycles recently encountered, regular routine analyses have been made on the carboy H_2O_2.

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Nov. 6 - Nov. 12 '45

These data show that in the range under investigation from 6.74% to 8.77% H_2O_2, the H_2O_2 concentration has no perceptible effect on the size of the recycle. Further data will be available at a later date.

Oxalate Recycle Method

The average of the eight oxalate runs from T-5-10-B-35 to T-5-11, B-5 have shown a 2.6% loss (~ 0.1% of starting assay 8-1-MR) and a 102% material balance for this step. However, there are still large discrepancies in individual cases.

Process Chemistry

Appraisal of Activity Deposited on 100-F Area Slugs

A sample of an oil emulsion of the film surrounding the slugs in the F Pile was taken at the time of pushing on November 8. Decay data are being collected on the gross beta and gross gamma constituents. Analyses have been completed for the purpose of isolating radio-active Ca, Mn, Cu and Na. No radioactive calcium, barium, or strontium was detected in the calcium fraction. The manganese and sodium fractions have activities roughly corresponding to 2.6h Mn and 14.8h Na, but more accurate evaluation must wait until the longerlived constituents accompanying them can be established. Copper has been definitely isolated and identified as 12.8h Cu; this fraction was essentially free from contamination of other activities, and appears to contribute 15-20% of the total activity 12 hours after removal from the pile. A search for longer lived activities is continuing.

Soil Retention Studies

An additional 3.5 gallons of 110 Tank waste solution has been passed through the column. Approximately 30 gallons of waste from the tank farm has been obtained, to provide sufficient solution for saturating the soil, since additional samples can be obtained only with difficulty after the tanks are partially closed by goosenecks to prevent spray from reaching the air.

Surface Decontamination Survey

A study is underway to collect all available information for establishing the best procedure or procedures to be followed by decontaminating various types of surfaces, such as cement, wood, stainless steel, etc., and various types of equipment. Laboratory studies will follow if warranted.

Radio-Active Xenon Absorption

A preliminary examination has been made of the feasibility of assaying radioxenon in the atmosphere by absorption in a liquid solvent. It was calculated that in water at 20°C a maximum concentration of 3.7 d/min./liter could be obtained if the xenon in the atmosphere were at tolerance level (10^{-11} curies/ml.). Scarcely any data were obtainable on xenon solubility in other liquids, but by analogy with radon, for which solubilities in a large number of liquids have been measured, it is reasonable to think that solubilities up to 100 times that in water could be obtained in certain organic derivatives such as olive oil. This solubility would correspond to 370 d./min./l. when the xenon in air is at the tolerance level.

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Isolation of Europium

The processing of the 10 cc. of 8-1-M Solution has been completed, and while the radio-assay for purity of the europium has not been completed as yet, the indications are that sufficient decontamination has been obtained to enable the solution to be used for mass assignment studies in the mass spectrograph.

M F Acken
M.F.Acken, Chief Supervisor
200 Areas Technical Division

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#10	B.H.Mackey-D.A.Miller-100 Area	#22	J.D.Ellett
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November 23, 1945

This Document consists of
2 Pages
4 Copies, Series A

200 AREAS
November 13 Through November 19

Canyon Building

Basis Assay (B)

Sampling the 7-1 Precipitator in the B Canyon at 35°C instead of 85°C reduced the discrepancy between this assay and the quantity based on the 6-1 transfer weight or the sum of the 7-4 and 7-3 assays (extraction cake plus waste) but did not result in complete agreement. Sampling at 85°C has therefore been resumed. Samples were also taken from the 6-3 intermediate transfer tank and on several runs the specific gravity of the solutions were determined in the laboratory. The best agreement continues to be between the 6-1 transfer weight and the sum of the extraction yield and loss:

Run Number	Transf. from 6-1		6-3 Assay	Assayed in 7-1		7-4P + 7-3W
	Sample Temp.	Assay		Sample Temp.	Assay	Assay
B-5-11-B-9	75°	100%	103.6%	35°	103.0%	99.0%
B-5-11-D-10	75°	100%	100.5%	35°	103.0%	100.5%
B-5-11-D-11	75°	100%	100.5%	35°	100.5%	99.5%
B-5-11-D-12	75°	100%	102.0%	35°	105.5%	100.0%

Run Number	Transf. from 6-1		Assayed in 7-1			7-4P + 7-3W Assay
				Assay		
	Sample Temp.	Assay	Sample Temp.	Sp.Gr.Meter	Lab.Sp.Gr.	Sp.Gr.Meter
B-5-11-D-13	75°C	100%	35°C	106.5%	105.0%	100.0%
B-5-11-D-14	75	100	35	106.0	102.5	100.5
B-5-11-D-15	75	100	50	102.0	96.5	98.5
B-5-11-D-16	75	100	85	105.5	103.0	100.5
B-5-11-D-17	75	100	85	109.0	108.0	94.5
B-5-11-D-18	75	100	85	102.5		96.5
B-5-11-D-19	75	100	85	107.0		101.0
B-5-11-D-20	75	100	85	106.5		101.0
B-5-11-D-21	75	100	85	107.0		99.0

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The material balance across the canyon is being checked on a number of runs, on which the specific gravity of the 17-4-P product sample is being determined in the laboratory. On the basis of two runs checked, the best agreement is obtained using the 7-4-P plus 7-3-W as the basis:

Run Number	6-1 Assay	7-1 Assay		7-4P + 7-3W	Total Waste + 17-4P	
	Sp.Gr.Meter	Sp.Gr.Meter	Lab.Sp.Gr.	Sp.Gr.Meter	Sp.Gr.Meter	Lab.Sp.Gr.
B-5-11-D-16	100%	105.5%	103.0%	100.5%	102.5%	100.5%
B-5-11-D-17	100%	109.0%	108.0%	94.5%	96.0%	94.0%

Extraction Waste Loss (B and T)

During the past month several variables have been introduced into the extraction step which may have affected the extraction waste loss:

1) Metal solution of lower free nitric acid, resulting from dissolving to a final specific gravity of 1.82 instead of 1.81.

2) Increased sulfuric acid concentration (1.0N) in the metal solution at the time of the strike.

3) Slow speed (650 rev./min.) skimming of the extraction cake.

4) Reduced phosphoric acid concentration (0.6M) at the time of the strike.

Only changes (1) and (2) were made in the T Canyon, these being made concurrently. In the B Canyon, the changes were made as indicated below, with the following results:

Summary of Extraction Variables

Change	Canyon	No. of Runs	Waste Loss (%) Average	Waste Loss (%) Range
Previous condition	T	34	1.27	0.62-2.4
Previous condition	B	39	1.07	0.40-2.1
(1) and (2)	T	37	0.94	0.54-1.8
(2) and (3)	B	10	1.00	0.75-1.4
(2), (3) and (4)	B	7	1.10	0.62-2.2
(1) and (2)	B	8	0.94	0.54-1.8

It is impossible to separate the effect produced by each of the variables, but the combination of a higher gravity end-point on metal dissolving and increased sulfuric acid concentration during the strike has reduced the waste losses significantly to the same values in each canyon.

Decontamination in the Canyon (B and T)

The B Canyon continues to produce slightly poorer decontamination than the T Canyon although this is fully compensated for in the B Concentration Building, the overall decontamination being slightly higher in the B Plant than in T Plant:

Range of Decontamination Factors (dF)

	B Plant	T Plant
Through Canyon Building	5.00 - 5.10	5.20 - 5.40
Through Concentration Bldg.	7.30 - 7.50	7.10 - 7.30

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That this difference in Canyon decontamination represents a real difference in the amount of activity sent to the Concentration Building is indicated by the following comparison between gamma activity per unit of product and decontamination factors:

Decontamination Factor (dF)	Run No.	Gamma/Product*
4.94	B-5-11-D-15	55.5
5.12	B-5-11-D-8	48.2
5.19	B-5-11-D-13	38.1
5.22	T-5-11-F-10	30.0
5.23	T-5-11-F-13	29.4
5.29	T-5-11-F-18	25.6
5.40	B-5-11-B-9	26.5
5.42	T-5-11-F-12	20.0

* gamma counts/million alpha counts

Since this slight difference in decontamination presents no problems to the operation of the Concentration Building, no change in process is contemplated because of it.

Concentration Buildings

LaF$_3$ Product Cake Removal

At B Plant, the use of the high-pressure water sprays alone for LaF$_3$ product cake removal from Centrifuge E-2 was attempted in Runs B-5-11-B-8, AW-1, and B-9. The acid-wash run (AW-1) was not very informative since the Beckman meter levels were too low. The other two runs, however, produced Beckman meter readings definitely higher than the KOH-slurry method, indicating product hold-up. The 5-nozzle spray was connected to two Taber pumps in series, developing 190 lbs./sq.in. pressure at a delivery of 15 gal./min.

Beginning with Run B-5-11-D-10, a return to the KOH-slurry method was made, using the 5-nozzle spray and one pump only. This system develops about 90-95 lbs./sq.in. pressure. Cake removal has been satisfactory thus far, with the Beckman readings at the end of the initial 700-lb. water spray averaging lower than those formerly produced by the 4-nozzle spray.

Continued use of the KOH-slurry method and 4-nozzle spray at T Plant has been producing satisfactory cake removal.

LaF$_3$ By-Product Waste Losses

As pointed out in the last weekly report, the LaF$_3$ by-product (D-4-BP) waste losses at T Plant appear to be larger from the 9.0-lb. La recycle than the 6.0-lb. gallery La runs. In an effort to determine whether this is due to the difference in respective cake weights or to something inherent in the new type of recycle solution, a series of runs has been made in which 6.0 lbs. of lanthanum was recycled from the Storage Tank E-4, instead of the usual 9.0 lbs.

The loss data for these runs are shown in the following table. The data appear inconclusive in that the average loss of the 6.0-lb. lanthanum recycle runs is 0.76%, as compared to an October average of 0.64% for 6.0 lbs. of gallery lanthanum. The average of three 9.0-lb. lanthanum recycle runs made during this same period was 1.01%, which is definitely higher. More test runs have been recommended.

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LaF_3 By-Product (D-4-BP) Waste Losses (% 8-1M)

Run Numbers	Lbs. La in B.P.Cake	Source of La	% 11-4-BP
T-5-10 Ave.	9.0	Recycle	0.92*
T-5-10 Ave.	6.0	Gallery	0.64
B-5-10 Ave.	6.0	Gallery	0.73
T-5-11-F-11	6.0	Recycle	0.73*
T-5-11-F-12	6.0	Recycle	0.79*
T-5-11-F-13	6.0	Recycle	0.66*
T-5-11-F-14	6.0	Gallery	0.63
T-5-11-F-15	9.0	Recycle	0.88*
T-5-11-F-16	6.0	Recycle	0.85*
T-5-11-F-17	9.0	Recycle	1.22*
T-5-11-F-18	9.0	Recycle	0.93*
T-5-11-F-19	6.0	Gallery	0.49

* Corrected to performance basis for recycle.

A-4-BP Losses

Recently at T Plant there have been $BiPO_4$ by-product (A-4-BP) waste losses frequently ranging as high as 1.0%. Both incomplete oxidation in Tank C-4 and/or incomplete removal of the product heel from precipitator A-1 at the end of jetting to Centrifuge A-2 are suspect. To check these suspicions, C-4-O samples are being assayed for total and reduced product. In addition, a sample of the cake solution acid after it has been circulated in the precipitator is being assayed for product content. The results of several recent assays are shown in the table below. With the exception of Run T-5-11-F-19, no high A-4-BP losses have occurred recently, so the study is being extended.

C-4-O, A-1 Heel, and A-4-BP Product Assays (%8-1M)

Run No.	% Reduced Prod. in C-4-O	% Prod. in A-1 Heel A-1-AW	% A-4-BP Loss
T-5-11-F-15	0.27	0.05	0.26
T-5-11-F-16	0.13	0.07	0.23
T-5-11-F-17	0.19	0.04	0.29
T-5-11-F-18	2.4 (?)	0.05	0.22
T-5-11-F-19	24.0	0.46 (?)	22.4

In Run T-5-11-F-19, a 22.4% A-4-BP Loss was obtained. A check of the C-4-O sample showed 25% reduced product. A further check of the Cell C operation revealed that the agitator was inadvertently left off for 15 minutes after the $NaBiO_3$ addition. Apparently the $NaBiO_3$ decomposed during this time in the bottom of the 7.5N HNO_3 solution, without adequate mixing contact with the product. The A-4-BP was successfully reworked (0.2% loss) by returning it to precipitator A-1, adding HNO_3 to increase its volume to agitator level at 5N HNO_3, reoxidizing, reprecipitating, and carrying on as an 80% volume run.

B Area Acid-Wash Run

Following Run B-5-11-B-8, an acid-wash run (AW-1), originating in the extraction cell of the Canyon Building, was carried through the Concentration Building at B Plant. This run arrived at the Concentration Building with 11% product pick-up and picked up an additional 5% only in passing through the building and all of that in Cell F, as in the T Plant acid-wash run.

The data are summarized as follows:

[redacted]

B Plant Concentration Building Acid-Wash Run (B-5-11-AW-1)

Process Vessel or Step	Sample Code	Product Balances	
		Total %	% Pick-up
Basis		2.00×10^{13} T.C.	
Canyon Building	17-4-P	10.4	--
C-4	C-4-0	10.9	+0.5
A-1	A-1-0	11.3	+0.4
$BiPO_4$ B.P. Loss	A-4-BP	0.07	--
A-3	A-3-OS	11.3	+0.1
D-1	D-1-0	11.5	+0.2
LaF_3 B.P. Loss	D-4-BP	0.09	--
D-3	D-3-OS	11.3	-0.1
E-1	E-1-R	11.2	-0.1
LaF_3 Prod. Waste	E-3-WS	0.28	--
F-1 (plus E-2 to F-1 line)	F-1-PS	16.0	+5.1
15% KOH Meta. Loss	F-7-WS	0.11	--
2% KOH Meta. Loss	F-9-WS	0.07	--
Final Yield	F-10-P	14.6	-1.2
	Net Total Pick-Up		+4.9

Isolation Building

Oxalate Method of Recycle Handling

For the 75 runs completed to date, 3.7% of the product handled has been sent to waste and 94% returned to the Concentration Building with a material balance of 97.7%. This loss is 0.14% on the 8-1 MR basis.

In an attempt to ascertain the magnitude of error inherent in the present waste sampling and analysis procedure which must deal with alkaline solutions containing suspended solids that carry product, the wastes have been acidified and re-assayed on four runs chosen at random:

Run No.	% of CT-1-W in			
	1st Waste		2nd Waste	
	Alkaline	Acidified	Alkaline	Acidified
T-5-10-F-27	0.07	0.24	0.08	0.39
T-5-10-F-31	1.3	1.76	2.0	2.6
T-5-11-B-2	0.64	0.20	0.80	0.28
T-5-11-B-7	6.4	1.4	5.6	6.0
Weighted Ave.	1.8	0.8	1.9	2.0

These results substantiate the belief that more consistent and reasonable assays are obtained when the wastes are acidified and the suspended solids dissolved.

Weight of Product Recycled

An effort has been made to correlate the effect of H_2O_2 concentration of the reagent used with the weight of product recycled. Graphing the latter as a function of the former does not show correlation; the data are widely scattered. There does appear, however, a slight trend for higher recycles to result when weaker H_2O_2 is used; perhaps one unit more of recycle is caused by a drop of 1% H_2O_2 in P-1 (or of 3% H_2O_2 in the carboy). The substitution of "Albone" for "Becco" H_2O_2 should be considered since the former contains a better stabilizer and retains the 30% strength satisfactorily.

It is of interest to note that the weight of product recycled dropped for two recent B Plant runs in which the KOH addition was made in Cell F instead of in Cell E. These runs, B-5-11-B-8 and 9, recycled 7.7 and 5.0 units respectively, whereas the previous 7 runs from B Plant recycled an average of 8.8 units/run. This confirms the fact, previously reported, that the weight of product recycle was increased by about 2 to 4 units when the Concentration Building metathesis procedure was changed to provide for KOH addition in Cell E instead of Cell F.

One Cycle Isolation Process

The possibility of shortening the isolation process by eliminating the second cycle and adding additional washes to the first cycle to obtain 95% purity was considered. A survey of available information has been made on this subject. This included the Memo Report SE-PC-41 with laboratory data and various results of plant runs. The general conclusion is that a single cycle procedure with extra washes would probably not furnish sufficient purity; such a procedure would increase cycle time by 3 to 5 hours.

A review of SE-PC-41 leads to the following comments:

1) The laboratory work on a one cycle process resulted in purities of 95.3% and 99.3% on two separate experiments where a total of five washes were made, two with 0.2N H_2SO_4, two with 1.0N HNO_3, and one with water. These purity measurements are high, however, since they were made by the old α count vs. weight method. This procedure was found to give purity values greater than 100%. It was replaced by the present hexone extraction procedure which weighs the impurities which are water soluble but hexone insoluble. The routine plant purity measurements dropped about 4% when the change of method was made (6 runs by old method averaged 102.1% pure while the next 8 runs, by new method, averaged 97.9% pure). This suggests that the purity values actually obtained in the two laboratory experiments mentioned above were about 91% and 95%.

   If these revised purity data are accepted, it becomes apparent that agreement was poor between the gross purity and the purity with respect to lanthanum alone. Using radio-lanthanum, it was found that 0.15% of the original La remained with the product after 1 precipitation and 5 washes. This means 98.6% purity with respect to lanthanum alone. The average gross purity found by the old method was 97.2% which would be about 93% by the new method. This would seem to indicate that elements other than lanthanum were predominant in determining purity. This is not indicated by the spectrographic analyses which showed lanthanum to be the major impurity.

2) The statement that elimination of the second cycle would decrease processing time is misleading. Although the time to isolate a given batch would be shortened, additional washes would lengthen the first cycle and decrease the number of runs processed per week per cell. This could of course be avoided by making piping changes to by-pass Nutsche N-2 and doing some

[redacted]

of the washes in the second precipitator, P-2. Additional piping changes would then be required to filter the cake solution from P-2 through N-2, a step that would probably be essential.

3) The laboratory work was all done with P-1 solutions containing 1140 grams of lanthanum/batch. Current usage of 1800 grams of La would make the removal of La at least 60% more difficult.

4) The work was also done at a product level of about 100 units/batch. It has been assumed that present levels of 250 units/batch would make attainment of 95% purity much easier. This belief is apparently substantiated by lower than average purities on occasional recent batches of low product content such as T-5-9-R-1. Such cases, however, have involved two precipitations which permit better removal of adsorbed or carried La than is possible in multiple washing. It is possible that increased product concentrations would proportionally raise the amount of impurities carried along by adsorption or similar phenomena. It is this type of impurity that is most difficult to remove by washing.

5) "Albone" H_2O_2 was used in all work discussed in this report. Current use of "Becco" H_2O_2 might be said to favor increased purity after one cycle. This is doubtful, however, for the improvement resulting from the change to Becco was a decrease in insoluble solids in the AT solution. This factor is of no import in purity measurements since the AT sample is centrifuged and a portion of the supernate tested.

Plant data not available for SE-PC-41 also throw doubt on the success of a one-cycle process with 5 or 6 washes. The process used before May 31, 1945 when a change in washing procedure was made, was yielding occasional low purities. The preceding 15 runs averaged 97.2% with one value of 93.5% purity. Actual purity was considerably lower than theoretical. After the change in washing procedure (three washes of 6 liters in each cycle), the next fourteen runs averaged 98.7% purity with a low of 97.9%. Theoretically, the fraction of La left would be 1/8000. This would leave 0.14 grams of La for 99.95% purity. These factors would suggest a five-fold improvement in purity, whereas the impurities were reduced by only a factor of two (from 2.8% to 1.3%).

Calculating the fraction of La left after a single precipitation and 5 washes of 6 liters each, one gets 1/680. Six washes would only lower this to 1/1600, a factor that was insufficient in the runs before May 31 discussed above, even though a second precipitation was included. This furthers the general view that a successful one cycle process is improbable.

Process Chemistry

Appraisal of Activity Deposited on 100-F Area Slugs

A sample of water extract of the oil emulsion from 100-F Area was analyzed for alpha activity. Only 6.5 c/m/ml. of emulsion were found. This indicates that essentially no product was in the film.

Radiochemical analyses have been continued on the oil emulsion (from November 8 push at 100-F). Analyses for chromium, iron, molybdenum, barium, zirconium, and columbium have been made, and the decay of activities accompanying carriers used in the attempt to isolate these activities is being followed. The major part of the activity has not yet been accounted for at any time after receipt of the sample.

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No fission products have as yet been identified in this sample. This is further proof that the film activity could be accounted for by uranium dust is quite unlikely.

Soil Retention Studies

The second cycle waste solution is still being fed into the column in continuous flow. An additional 3.5 gallons of effluent has been collected, the analysis of which showed 132 α counts/min./cc breaking through as compared to 900 c/m/cc in the starting solution. The flow rate has dropped to the very low figure of 1.5 cc/minute.

There is evidence of a bismuth compound precipitating from the solution around the valves and flanges, likely caused by a lower pH of the solution resulting from CO_2 absorption from the air. There is, therefore, a possibility that the same has happened within the column, thus lowering the flow rate and the amount of product appearing in the effluent.

Surface Decontamination Survey

Material is still being gathered for a critical survey of the existing information on decontaminating various types of surfaces as a preliminary for possible laboratory work.

Effect of 85°C -50% KOH on Crystalline LaF_3 on Stainless Steel Surface

Since 50% KOH at 85°C is used to wash the centrifuge prior to the metathesis to remove the last traces of lanthanum fluoride, the question was raised as to whether the same treatment would dislodge crystalline lanthanum fluoride from the centrifuge. Laboratory tests were run in 250 cc stainless steel beakers using gelatinous LaF_3 as a control in the experiments. The following conclusions were obtained:

1) The hot KOH treatment is about twice as effective in removing gelatinous LaF_3 as the crystalline derivative.
2) Two treatments of the hot KOH generally will soak the crystalline LaF_3 from stainless steel surface.
3) The hot KOH treatment removes crystalline LaF_3 infinitely faster from stainless steel than does water treatment under the same conditions.

It is impossible to tell from laboratory results what could be expected in a plant trial. Indications are favorable, however, for the success of a plant trial.

Corrosion of 25-12 Cb and 18-8 Steel in 2% Sulfuric

As a wash for plutonium peroxide, 2% sulfuric acid is used in Isolation Building operations. A large make-up and storage tank will be installed, and corrosion tests have been initiated to determine a suitable, non-corrosive fabricating material. As can be seen from the following data, neither 25-12 or 18-8 stainless steel are corroded by 2% acid.

Corrosion of 25-12 and 18-8 Stainless Steel in 2% Sulfuric Acid (48-Hr. Exposure)

Composition of Coupon	Temp., °C	Penetration inches/month
25-12	25	0.0000
18-8	25	0.00002

[redacted]

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[redacted]

Composition of Coupon	Temp., °C	Penetration inches/month
25-12	65	0.00002
18-8	65	0.00002

M.F.Acken, Chief Supervisor
200 Areas Technical Division

MFA:jd

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#1 R.M.Evans
#2 R.M.Evans
#3 B.H.Mackey for The Area Engineer
#4 B.H.Mackey for The Area Engineer
#5 B.H.Mackey for The Area Engineer
#6 B.H.Mackey for The Area Engineer
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November 30, 1945

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200 AREAS
November 20 through November 26

Canyon Buildings

Basis Assay (B)

The basis for runs in the B Plant continues to be the 7-1-MR assay, which, during the past week, ranged from 103% to 108% of the "transferred weight from 6-1" and "7-4-P + 7-3-W" bases. Several means have been suggested for correcting this situation:

1) Return to the use of Section 8, permitting Section 7 to be maintained as a spare.
2) Obtaining the basis assay in Tank 6-3, the total contents of which are transferred to the precipitator.
3) Use of either the "transferred weight from 6-1" or the 7-4-P + 7-3-W" assays as the run basis.

The material balance across the canyon has been checked on four additional runs:

Run No.	6-1 Assay	7-1 Assay	7-4P + 7-3W Assay	17-4P + Total Waste	
	Sp.Gr.Meter	Sp.Gr.Meter	Sp.Gr.Meter	Sp.Gr.Meter	Lab.Sp.Gr.
B-5-11-D-18	100%	102.5%	96.5%	97.5%	94.0%
B-5-11-D-19	100%	107.0	101.0	102.0	102.0
B-5-11-D-20	100%	106.5	101.0	100.0	98.5
B-5-11-D-21	100%	107.0	99.0	101.5	100.0

These data add more confirmation to the inaccuracy of the 7-1-MR basis assay. The difference between the laboratory determined and instrument determined specific gravity of the 17-4-P (second cycle product) solutions is approximately 1.5%, the laboratory values being higher.

Neutralized Waste Storage (B and T)

The first storage tank in each series of three for the respective wastes is full at each plant; in addition, the second of the metal waste tanks was filled at the T Plant during the past month. A summary of the temperatures and reserve capacities in each area are presented below:

[redacted]

		Temperature (F°)						
		Metal Waste			1st Cycle		2nd Cycle	
Area	Date	101	102	103	107	108	110	111
T	6/19/45	108	--	--	90	--	88	--
	7/17/45	118	--	--	94	--	88	--
	8/21/45	132	--	--	102	--	90	--
	9/20/45	138(full)	102	--	94(full)	86	89	--
	10/23/45	146(full)	92(66%full)	--	95(full)	90(53%full)	89(full)	93 (17%full)
	11/26/45	151(full	115(full)	90(11%full)	93(full)	84(95%full)	85(full)	85 (48%full)
B	6/19/45	97	--	--	75	--	71	--
	7/17/45	110	--	--	80	--	78	--
	8/21/45	136	--	--	90	--	84	--
	9/20/45	142	--	--	96	--	86	--
	10/23/45	138(full)	106(36%full)	--	100(full)	78(19%full)	82 (56%full)	--
	11/26/45	143(full)	118(83%full)	--	104(full)	80(58%full)	80(full)	73 (12%full)

			Reserve Capacity as of 11/26/45			
			B Plant		T Plant	
Tanks	Type Waste	Gal./Run	Gallons	Runs	Gallons	Runs
X-101,-2,-3	Metal	5700	620,000	108	470,000	82
X-107,-8,-9	Ctng. & 1st Cycle	4700	750,000	159	556,000	118
X-110,-11,-12	2nd Cycle	3600	990,000	275	805,000	223

The spare tanks (X-104,-5,-6) have not been included in the above calculations of reserve storage capacity.

Concentration Buildings

LaF_3 Product Cake Removal

Extended use of the KOH-slurry method for LaF_3 product cake removal from Centrifuge E-2 at both T and B Plants has continued to provide satisfactory cake removal. At B Plant, the 5-nozzle spray, connected to a single Taber pump, is still in operation. The highest recent "empty" Beckman reading at T Plant has been 2.5×10^{-14} amperes and 2.0×10^{-14} amperes at B Plant. There have been no centrifugation delays because of cake hold-up.

F-2 High-Pressure Spray

The 2-nozzle high-pressure spray was installed in Centrifuge F-2 at B Plant and put into service with Run B-5-11-D-13. It has regularly performed satisfactorily since its installation, reducing the "empty" F-2 Beckman reading below 5×10^{-14} amperes.

F-10 Beckman Tube Position

Recently, the G.E. tubes for the F-10 Beckman meters have been changed in position from the former point opposite the narrow end of the tank to a point broadside to the long edge of the tank. This change in position has increased the sensitivity of the metering system by 2.5 to 3 fold. The correlation between the F-2 and F-10 Beckman meter readings has improved since the F-10 meter sensitivity has been increased.

Skimming Speeds

At T Plant, skimming of Centrifuge A-2 at the reduced speed of 650 rev./min. was initiated with Run T-5-11-F-23. No operating difficulties have been experienced. At present, all of the 40-inch centrifuges in both Concentration Buildings are being skimmed at an inched speed of 650 rev./min. instead of the former speed of 870 rev./min.

A-4-BP Waste Losses

In view of past waste loss history and present inability to obtain a clear-cut definition of the occasionally high losses, it is recommended that a change be made in the C-4 oxidation acid normality from the present 7.5N to 6.5N HNO_3. This is the same acid concentration as used in the Canyon Building oxidations and should retard the present rapid decomposition of the $NaBiO_3$. It is intended that precipitator acid heel samples (A-1-AW) be continued and held for assay only when the A-4-BP losses are high, in order to check this source as a possibility if losses continue to be erratically high.

LaF_3 By-Product Losses (T)

Recent LaF_3 by-product losses in the T Plant have averaged 0.2 - 0.3% higher than those encountered in the past. In the following table an attempt has been made to correlate these fluctuations with process variables. These data indicate that the following factors may be associated with the upward trend in this waste loss:

1) Increased amounts of La now being used in the LaF_3 product step and returned in the recycle.
2) Increased amounts of product now being returned with the recycle.
3) Impurities normally associated with the recycled La (comparison with results using fresh La solution from gallery).

Further investigation of these points will be made.

T Plant LaF_3 By-Product (D-4-BP) Waste Losses (% 8-1-M)

Run No.	No. of Runs in Correlation	Lbs. La in B.P.Cake	Source of La	% Prod.in Recycle	% D-4-BP Loss
T-5-08-Ave.	18	9.0	Recycle	7.2	0.72 *
T-5-11-Ave.	4	6.0	Gallery	None	0.62
T-5-11-Ave.	4	6.0	Recycle	7.5	0.76*
T-5-11-Ave.	19	9.0	Recycle	10.1	0.98*

* D-4-BP losses corrected for recycling to a "performance" basis.

Isolation Building

Oxalate Method of Handling Recycles

The first draft of a memorandum report summarizing the results of Production Test SE-231-PA-6 to date has been prepared.

[redacted]

Supernatant solutions from two runs in the 5-11 Series contained unusually large amounts of product, 48% in T-6 and 14% in T-12. As a result of the first of these two instances, the practice of dumping the waste before analysis has been discontinued. The T-12 waste was put in an RC can and later reworked (oxalate destroyed with permanganate) and sent tack to T Plant. This type of waste recovery procedure was also applied to T-19, although the loss was low.

Weight of Product Recycled

Analysis of each carboy of H_2O_2 is being continued to prevent the use of weak H_2O_2 since occasional concentrations of 22% and one of 15% have been found. It appears desirable to revert to "Albone" in place of the current "Bocco" H_2O_2.

In spite of these checks on H_2O_2 concentration occasional high recycles are encountered. In the 44 runs of the 5-11 Series completed to date, there were three, T-16 at 21.2 units, B-12 at 24.0, and B-20 at 20.3. No reasons are shown.

One Cycle Isolation Process

In the discussion under this topic last week, it was stated that additional washes in the first cycle would lengthen it sufficiently to decrease the number of runs processed per week per cell. While this would be strictly true, it would not be practically so, for cycle time would not be increased beyond 16 or 18 hours which represents the current maximum rate at which runs are received into any one cell.

Process Chemistry

Appraisal of Activity Deposited on 100-F Area Slugs

Decay curves, of radiochemical fractions of film removed from 100-F Area slugs on November 8, are still being followed to identify the longer lived radio constituents.

Solution Reduction Time Prior to LaF_3 Product Precipitation

It is plant practice to allow 1 hour for reduction after the addition of oxalic acid and before the commencement of the lanthanum strike. The question has arisen as to whether all or part of that reducing time could be saved. In a 1 liter scale laboratory run, reduction of the permanganate and dichromate oxidants was complete 5 minutes after the addition of the oxalic acid. Since the lanthanum strike lasts twenty minutes, and an additional hour is allowed for digestion, it appears that the lanthanum strike could be begun immediately after the addition of the reducing agent, thereby saving 1 hour of plant operation in the E Cell.

Determination of Solubility of $BiPO_4$ in Certain Process Steps

As a method of decreasing consumption of essential materials, the suggestion has been made that the phosphoric acid concentration could be lowered in the extraction step and in the $BiPO_4$ product precipitation steps. Prior to high product runs, tests were made on synthetic solutions to determine whether reducing the acid concentration would result in an abnormally large amount of bismuth remaining in solution. As can be seen from the following data, the quantity is increased, but not objectionally so. The high-product level runs are now in progress.

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Step	H_2PO_4 Concentration	Solubility Bi- mg/l
Extraction	0.8M	125
Extraction	0.6M	250
Product Precipitation	0.6M	80
Product Precipitation	0.4M	190

Isolation of 95^{241}

From process solutions in Isolation Building product purifications, 95^{241} has been isolated. A series of steps separating the 95 from lanthanum are now in progress. The purified element will be used for mass assignment studies.

Surface Decontamination Survey

The literature search, necessary for writing a memorandum giving specific instructions for decontaminating various surfaces, is still in progress.

Corrosion of 25-12 and 18-8 Stainless Steel in 2% H_2SO_4

The corrosion study of stainless steels in 2% sulfuric acid reported last week has been concluded. On the basis of these laboratory data, both 18-8 and 25-12 stainless steel are perfectly satisfactory for fabricating storage tanks for 2% sulfuric acid.

Composition of Coupon	Temp., °C	Penetration Inches/Month		
		First Exposure	Second	Third
25-12	25	0.0000	0.0000	0.0000
18-8	25	0.00002	0.00002	0.00001
25-12	65	0.00002	0.0000	0.00001
18-8	65	0.00002	0.00003	0.00001

M. F. Acken
M.F.Acken, Chief Supervisor
200 Areas Technical Division

MFA:jd

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#1 R.M.Evans
#2 R.M.Evans
#3 B.H.Mackey for The Area Engineer
#4 B.H.Mackey for The Area Engineer
#5 B.H.Mackey for The Area Engineer
#6 B.H.Mackey for The Area Engineer
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#18 R.S.Apple
#19 R.H.Beaton
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#23 J.J.Urban- 700 File
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December 7, 1945

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200 AREAS

November 27 Through December 3,1945

Canyon Buildings

Dissolver Performance (B and T)

The dissolving time cycles on the 4-5-L Dissolver in the T Canyon have been increasing, indicating depletion of the metal heel. Accordingly, beginning with Charge T-5-11-Fd-42, the weight of 60% nitric acid charged to this dissolver was reduced from 5400 lbs. to 5100 lbs., the quantity being used in the 3-5-R Dissolver. In the B Canyon, the weights of acid charged to 3-5-R and 4-5-L are 5000 lbs. and 4900 lbs. respectively.

Basis Assay (B)

Beginning with Run B-5-11-D-34 the unsatisfactory 7-1-MR assay was replaced with the "7-4-P plus 7-3-W" assay as the bases for the runs.

Beckman Activity Measurements on the Ventilating Fans (B and T)

Ionization chambers installed near each electric ventilating fan and connected to micromax-Beckman recorders in the Fan House have been placed in service at both the B and T Plants. At the T Plant, the installation has not yet been satisfactorily adjusted. At the B Plant, the idle fan (No. 1) gives a slightly lower reading than does the operating fan (No. 2), the reading on the No. 1 Fan shows a very small increase when it is started for routine checking and the readings on both fans have shown a gradual decrease during the past week. This latter may be due to decay of fan activity, change in activity of material processed in the canyon, or a drift in the instrument. The time interval has been too short to permit any definite conclusions to be drawn. There has been practically no fluctuations in readings during any 24-hour period.

Concentration Buildings

Weight of La in LaF_3 By-Product

In an effort to minimize the LaF_3 by-product (D-4-BP) waste losses and to decrease simultaneously the use of lanthanum ammonium nitrate, a series of test runs

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s being carried out at B Plant. In these runs, the weight of lanthanum added to Cell D for the LaF_3 by-product is being successively decreased from 6 to 5 to 4 to 3 lbs. in 5-run group tests. It is felt that the use of a smaller by-product precipitate would not injure the decontamination unduly and would, in turn, cause less product hold-up in the centrifuge cake.

Thus far, a reduction of 1 lb. of lanthanum (6 to 5 lbs.) has been made in 5 runs. A possible decrease of 0.1% in waste loss is indicated. No noticeable effect on decontamination factor has been observed. A further reduction to a 4 lb. cake is in progress.

The test data thus far collected are shown below.

LaF_3 By-Product Waste Losses (% D-4-BP)

Run No.	No. of Runs	Lbs. La in B.P. Cake	% D-4-BP Ave.	% D-4-BP Range	dF (Cell D) Ave.	dF (Cell D) Range
Ave. of B-5-11-D-17 to D-26	10	6	0.68	0.54-0.90	8.8	8.0-9.7
Ave. of B-5-11-D-27 to D-31	5	5	0.58	0.34-0.73	8.1	6.7-11.8

Study of Plant Reduction

In an effort to determine if it is possible to eliminate the present separate digestion time (1 hour) for the oxalate reduction preceding the LaF_3 product strike in Precipitator E-1, a study of plant reduction conditions is in progress at T Plant. Samples are being taken from E-1 at measured time intervals during both the normal reduction period and the first lanthanum strike digestion. These samples are being analyzed for completeness of reduction and carrying.

F-1-PS Sampling

Arrangements are being initiated at T Plant to have constructed and installed for testing a modified version of the Smith type of external sampler. This equipment will be used for sampling the F-1-PS LaF_3 product slurries, a frequent point of irregular assay believed to be due to poor plant sampling.

Crystalline LaF_3 Tests

Plans are being made for carrying out dummy runs in the near future in Cell B of B Plant with crystalline LaF_3. These initial tests are designed to study the problems of plant preparation of the 2a reagent and the removal of the cake from the centrifuge. If successful, these preliminary tests are expected to lead to plant trials in production runs.

Isolation Building

Product Content of Recycle Solutions

The amount of product recycled to the Concentration Building has decreased during the past month and is currently less than 10 units per batch for both B and T material as indicated in the following tabulation. On the basis of the overall information to date, little correlation can be found with fluctuations in the amount of peroxide used or the use of KOH in removing LaF_3 product cakes.

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Runs	H_2O_2 Used, kg.	Av.Units Recycled/ Run	Cell
T-5-9-ECW-1 through T-5-10-D-8	15.75	11.5	4
T-5-10-D-9 through T-5-10-B-36	18.9	11.0	4
T-5-10-B-37 through T-5-11-F-16	15.75	10.2	4
T-5-11-F-17 through T-5-11-B-33	15.75	7.5	4
B-5-9-D-14 through B-5-10-B-12	15.75	10.5	3
B-5-10-B-13 through B-5-10-D-41	18.9	9.1	3
B-5-11-B-1 through B-5-11-D-13	15.75	10.9	3
B-5-11-D-14 through B-5-11-D-29	15.75	9.0	3

One Cycle Isolation Process

As a first check on the validity of the conclusions reached in the report of November 20, four runs have been sampled for "purity" assays after the regular present first cycle. The theoretical purity attainable at this point, based on volumes of solutions, heels, and washes with no carrying or adsorption, is 92% for a 250-unit batch. The purity values obtained on the special P-2-P samples were as follows:

T-5-11-F-28	85.8%
B-5-11-D-23	93.9%
B-5-11-D-29	92.3%
T-5-11-B-34	82.1%

Two facts are immediately apparent; 1) the amount of impurities differs between extremes by a factor of 3 (17.9% on -T-B-34 vs. 6.1% on B-D-23); and 2) both low purities occurred on Cell 4 material from T Plant. The Cell 3 values are encouraging to the possibility of 2 or 3 additional washes furnishing purity greater than 95%, whereas the Cell 4 values are not.

The iron content of the P-1 and AT solutions reveals no direct relation with purity either of P-2-P or AT samples, although the run with highest P-1 iron content showed the lowest P-2-P purity and that with lowest P-1 iron had highest P-2-P purity.

The data indicate that any removal of Fe which occurs in the second peroxide cycle is probably wasted effort since evaporation in the still returns much iron by corrosion. It, therefore, seems that a single cycle isolation process would be satisfactory as regards iron. The main impurity to consider is therefore lanthanum. The present process theoretically leaves 1/4300 of the La with the product. This is 0.43g. La/250g. product or 1700 ppm. A single cycle process with two extra washes (total of 5) would theoretically leave 1/410 of the La or 4.5g.; this would be 18,000 ppm. One more wash and increased volume of washes could improve this picture if the product peroxide has not adsorbed or carried too much La.

For further information, it is planned to determine purity and iron after one cycle and after both cycles (but before evaporation) for comparison with P-1 and AT data.

Process Chemistry

Appraisal of Extraction Conditions

Lowering of the H_3PO_4 concentration in the extraction is a potential means of

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decreasing operational costs. Liter-scale extraction runs were made from 24% UNH containing 25 mg. Pu/liter. As can be seen from the following data, the combination strike gave the lowest product loss:

H_3PO_4 Concentration	Type of Strike	% Pu in Effluent
0.6M	Combination	0.1
0.6M	Direct	1.6
0.8M	Direct	0.31

Dummy runs were made to determine whether any differences in the rate of solution of the $BiPO_4$ from the runs under various precipitating conditions could be detected. The rates of solution were identical.

The carrying results will be checked before any recommendations will be made to the plant.

Laboratory runs are in progress for information on the effect of lowered H_3PO_4 concentration on carrying in the $BiPO_4$ product precipitation steps in the decontamination cycles.

Isolation of 95^{241}

A sample of 95^{241} has been isolated and carried through three peroxide-hydroxide cycles. Purity assays are now in progress.

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M.F.Acken, Chief Supervisor
200 Areas Technical Division

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#1 R.M.Evans
#2 R.M.Evans
#3 B.H.Mackey for The Area Engineer
#4 B.H.Mackey for The Area Engineer
#5 B.H.Mackey for The Area Engineer
#6 B.H.Mackey for The Area Engineer
Attention: Patent Group
#7 N.Hilberry
#8 N.Hilberry
#9 N.Hilberry
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#24 W.K.MacCready

December 14, 1945

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200 AREAS
December 4 Through December 10

Canyon Buildings

Process Performance (B and T)

Process performance, as indicated by waste losses and decontamination factors has continued to be satisfactory in both B and T Canyons with no off standard operations.

Beckman Activity Measurements on the Ventilating Fans (B)

The ionization chambers as recently installed at the ventilating fans (under the discharge ducts) were located in such position as to give non-representative readings. This is illustrated by a comparison of the readings obtained with the "fixed" installation and a portable meter:

	Fan No. 1 (idle)	Fan No. 2 (running)
Fixed meter	9×10^{-11} amp.	13×10^{-11} amp.
Portable meter	700 mr/hr	3400 mr/hr

The "fixed" chambers are being relocated in order that they may "see" more of the fan housings and suitable shielding is being specified to prevent background interference of one fan on the other.

Of interest is the appreciable decay of radio-activity on the No. 1 Fan which has taken place since it went out of continuous service (November 3).

Concentration Buildings

Reduction of Lanthanum in LaF_3 By-Product

The test runs at B Plant involving a progressive reduction in the weigh of the LaF_3 by-product cake, partially described in last week's report, have been completed. In the test series, five runs were carried out with 5.0 lbs. of lanthanum and five runs with 4.0 lbs. The normal weight is 6.0 lbs. Product waste losses (D-4-BP) were not decreased more than 0.1% and both the LaF_3 by-product decontamination

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factor and the over-all decontamination factor were decreased slightly. The latter disadvantage outweighed the slight gain in product yield so a return to the 6.0-lb. lanthanum cake was made.

The results of the test runs are summarized below:

LaF_3 By-Product Waste Losses (% 7-1-M)

Average of Runs No.	No. of Runs	Lbs. La in B.P. Cake	% D-4-BP Ave.	% D-4-BP Range	LaF_4 BP dF Ave.	LaF_4 BP dF Range	Overall dF Ave.	Overall dF Range	PR Beck (mr/hr)
B-5-11-D-17 to D-26	10	6.0	0.68	0.54-0.90	8.8	8.0-9.7	7.41	7.30-7.51	31
B-5-11-D-26 to D-31	5	5.0	0.58	0.34-0.73	8.1	6.7-11.8	7.22	6.98-7.53	42
B-5-11-D-32 to 12-D-2	5	4.0	0.63	0.52-0.71	8.1	6.5-11.3	7.22	7.03-7.39	51

Reduction and Carrying of Product in LaF_3 Product Precipitation

During Run T-5-12-F-1, numerous samples were taken from Precipitator E-1 during the $H_2C_2O_4$ reduction step and the LaF_3 product precipitation digestion. These samples were assayed by the Process Chemistry Section as quickly as possible after being taken in the plant. Three types of measurements were carried out. In the first, E-1-R samples spaced throughout the reduction period (1 hour) were assayed for completeness of reduction vs. time. In the second, samples of the LaF_3 product slurry were centrifuged in the laboratory to measure completeness of reduction and carrying. In the third, HF and lanthanum were added in the laboratory to an E-1-R Sample taken immediately after the $H_2C_2O_4$ addition in the plant and small aliquots removed at regular time intervals during the lab digestion, also measured for completeness of reduction and carrying.

The objective of these tests was to determine if time savings could be gained in the plant by decreasing reduction or LaF_3 precipitation digestion time.

The test data obtained showed that the product is still as much as 25% unreduced at the end of the 1-hour $H_2C_2O_4$ digestion period in the plant. Shortening of the reduction period would, therefore, not be desirable. Addition of the lanthanum immediately after the $H_2C_2O_4$ verified this in the laboratory strikes.

Reduction and carrying appeared to be complete, however, immediately after the first-strike lanthanum addition in the plant. The time interval between the end of the reduction digestion and the end of the HF and lanthanum addition is normally about 30 minutes. On the basis of these results, a series of test runs is being made in Cell E at T Plant. Three runs will be made with only 30 minutes digestion after the second lanthanum strike, instead of the usual 1-hour. If waste losses (E-3-WS) are unchanged, 30 minutes will also be removed from the first-strike digestion for three more test runs. Further shortening of digestion time will be considered if these runs prove favorable.

The test data collected in Run T-5-12-F-1 are shown below:

Plant Reduction of Product by $H_2C_2O_4$ vs. time (T-5-12-F-1)

Sample No.	Time (min.) after $H_2C_2O_4$ Addition in Plant: Sampled in Plant	Time (min.) after $H_2C_2O_4$ Addition in Plant: Run in Laboratory	% Prod. Not Reduced or Not Carried
E-1-R-1	5	20	--
E-1-R-2	15	30	49.1
E-1-R-3	30	45	48.4
E-1-R-4	45	60	39.5
E-1-R-5	60	75	24.1

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Sample No.	Time (min.) after $H_2C_2O_4$ Addition in Plant		% Prod. Not Reduced or Not Carried
	Sample in Plant	Run In Laboratory	
E-1-PS-1*	95	110	0.47
E-1-PS-2**	125	140	0.30

* Sample at end of lanthanum addition in plant.
** Sampled 30 min. after lanthanum addition in plant.

Carrying of Product by Lanthanum Added in Laboratory to Plant E-1-R Sample Soon After $H_2C_2O_4$ Addition

Sample No.	Time (min.) after $H_2C_2O_4$ Addition in Plant		% Not Reduced Or Carried
	Sampled in Plant	Aliquot taken from lab. digestion samp.	
E-1-R-1-A	5	35	36
E-1-R-1-B	5	50	36
E-1-R-1-C	5	65	13.2
E-1-R-1-D	5	80	9.3

F-1-PS Sampler Modifications

In view of the decreased importance of the F-1-PS Sample, as caused by consistently satisfactory removal of the LaF_3 product cake from centrifuge E-2, the proposed modifications of the F-1-Sampler (conversion to Clinton type) are not to be made.

Crystalline LaF_3 Tests

Procedures for the preparation of crystalline LaF_3 on a plant scale for proposed test runs in Cell B at B Plant have been compiled. Sketches of the necessary equipment revisions have been prepared. A production test describing both program and equipment set-up is being prepared. At present the equipment visualized for use is as follows: Tank G-1 (E.P. 224-170-1) - capacity 55 gal. - to serve as the main mixing tank; E.P. 231-140 - capacity 20 gal. - to serve as the lanthanum salt solution make-up tank; E.P. 231-114 - capacity 7 gal. - to serve as the H_2SO_4 solution make-up tank.

The volumes of the reagent solutions involved are as follows, named in order for Cells D and E batches respectively: 0.71M NaOH - 0.77M Na_2SO_4 - 23.8 and 8.2 gal.; 15% lanthanum ammonium nitrate solution - 18.7 and 6.5 gal.; 3N H_2SO_4 - 5.6 and 1.9 gal.; total volumes - 48 and 16.7 gal.

Isolation Building

Product Content of Recycle Solutions

There has been no appreciable change in the level of product recycled per run in the past few weeks. The average for Runs T-5-11-B-34 through T-5-12-F-5 was 9.0 units per run while that for B-5-11-D-30 through B-5-12-D-5 was 8.0. Few values above 10 are currently found.

Acidity of P-1 Solution

The P-1 acidities appear to have come under as close control as is probably possible. The 37 runs from T Plant in November averaged 2.02N with 7 values below the lower limit of 1.8N and 9 above the upper limit of 2.2N. The 34 runs from B Plant in November averaged 2.16N with none low and 14 above 2.2N. If the December results are comparable and no process changes are made to obscure the results, a

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later reduction of the quantity of acid used at B Plant may be desirable.

One Cycle Isolation Process

No additional data are available. Further checks are underway with purity and iron analyses being obtained on the first product cake solution after filtration through N-2 (P-2-P-1) and on the second product cake solution before transfer to the still (P-2-P-2). These values, compared with P-1 and AT data, will show how much purification from iron and other soluble impurities (notably lanthanum) is obtained from each peroxide cycle as now constituted. The effect of evaporation in the still should also be evident.

Stability of H_2O_2 Reagent

After receiving word that tin introduced into AT solution from "Albone" grade of H_2O_2 causes no particular difficulty to our consumer, it is planned to substitute Albone for "Becoo" H_2O_2 now used since the latter is often too weak for effective precipitation.

Filtration of First Product Cake Solution

Plans are being laid to determine, if possible, (1) the necessity for continuing this filtration, (2) the necessity for using filter aid, and (3) the best manner of using filter aid.

Process Chemistry

Examination of Uranium Slug After Coating Removal

To determine whether flow-sheet conditions for removing the aluminum coating would leave the surface of an uranium slug free of scale, the coating of a rejected slug was removed in the laboratory. The lathe marks were very clear and distinct. Any mars or imperfections on the metal surface could be detected visually. An HF wash of the stripped slug results in a cleaner surface, but the lathe marks are partially obliterated.

Appraisal of $BiPO_4$ Product Precipitation Conditions

Lowering of the H_3PO_4 concentration in the $BiPO_4$ product precipitation steps is a potential means of decreasing operational costs. Liter-scale runs with process solutions containing 25 mg. Pu/liter were carried through the product precipitation step under flow-sheet conditions. In one run the ultimate H_3PO_4 concentration was 0.4M, whereas in the other run the final H_3PO_4 concentration was 0.6M. As can be seen from the following data, the concentration of the H_3PO_4 within the 0.4-0.6M range does not influence the completeness of carrying. A plant test based on a 0.4M H_3PO_4 concentration appears justified.

H_3PO_4 Concentration	Loss of Product in Effluent
0.6M	0.45%
0.4M	0.36%

Solutions for Preparation of Crystalline $NaLaF_3$

The Specific gravity data on solutions used to prepare cyrstalline sodium

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lanthanum sulfate were needed so that necessary weight-basis calculations could be made. The following results were obtained.

Nature of Solution	Specific Gravity by Hydrometer
0.71M NaOH - 0.77M Na_2SO_4	1.120 at 28°C
0.27 lanthanum ammonium sulfate	1.089 at 25°C
Effluent from preparation of $NaLaSO_4$	1.064 at 27°C

M. F. Acken

M. F. Acken, Chief Supervisor
200 Areas Technical

MFA:jd

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#1 - R. M. Evans
#2 - R. M. Evans
#3 - Area Engineer
#4 - Area Engineer
#5 - Area Engineer
#6 - Area Engineer
Attn.: Patent Group
#7 - N. Hilberry
#8 - N. Hilberry
#9 - N. Hilberry
#10 - B.H. Mackey-D.A. Miller-
700 Area File
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#24 - W. K. MacCready

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December 21, 1945

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200 AREAS
December 11 through December 17

Canyon Buildings

Time Cycles (B and T)

In order to increase the metal cooling time (before dissolving) from forty to sixty days, the runs are now being scheduled on a twenty-four hour basis instead of as rapidly as process time cycles permit (approximately eighteen hours).

Processing "Class C" Metal (T)

During the next few months the T Canyon will operate on "Class C" metal of approximately 400 gms/ton concentration. Preliminary to dissolving this material, one full charge of heel was dissolved out of each dissolver. It is planned to use Dissolver 3-5-R only for the "Class C" metal, maintaining 4-5-L in a "heel-less" stand-by condition. The extraction conditions to be used for this material are essentially those of the current runs. However, since limitations on batch size will permit processing only approximately one-half the present weight of metal, the runs will be made at about one-half volume. It is proposed to vary the bismuth/plutonium ratio and bismuth concentration on these runs between constant ratio with increased concentration and constant concentration with decreased ratio. These proposed changes are covered by Production Test SE-221-T-PA-10.

Centrifuge Difficulty (B)

During the second cycle product cake removal operation on Run B-5-12-F-13, the centrifuge (17-2) was plugged past the stop position and run backward. A probable wobble of the bowl caused the dip tubes to become wedged on the bottom of the bowl, rendering the machine inoperative. The bent dip tubes were replaced by new ones one-half inch shorter and the bent plows were replaced by a skimmer. These changes were made with the centrifuge in place; the radiation level was sufficiently low to permit 8-hour exposure. No permanent damage to the centrifuge resulted and it has continued in operation.

Beckman Activity Measurements on the Ventilating Fans (B)

The ionization chambers on the two electrically driven ventilating fans at the B Plant have been relocated and shielded so that each chamber "sees" approximately

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half of its fan rotor and case, yet cannot "see" the other fan. The initial readings (first day) are as follows:

Fan No. 1 (idle)	Fan No. 2 (running)
3×10^{-10} Amp.	43×10^{-10} Amp.

If this arrangement proves to be satisfactory, the chambers at the T Plant fans will be relocated similarily.

Neutralized Waste Storage (B and T)

The project covering the installation of transfer lines from the T to U and B to C waste storage tanks has been approved and construction started on the T and U line. Assuming no saving in the metal waste volume by virtue of processing the "Class C" metal and the twenty-four hour run schedule, this line must be ready for use by February 4, 1946.

Concentration Buildings

Reduction of Digestion Time in LaF_3 Product Precipitations

Analysis of special samples taken during the product LaF_3 cycle on Run T-5-12-F-1 indicated that the digestion time after the addition of lanthanum could be eliminated without affecting the product waste loss. On the basis of these results three runs (T-5-12-B-11, 12, 13) were made reducing the digestion time after the second strike from sixty minutes to thirty minutes. No effect on the waste losses was noted. A second set of three runs (T-5-12-B-15, 16, 17) was made shortening by thirty minutes the digestion time after both lanthanum strikes. No adverse effect was noted; in fact, a decrease in waste loss of about 1.0% resulted at the end of the first strike and 0.1% at the end of the second strike. Additional runs would be required to establish whether this small improvement was real. Further shortening of the digestion time will be recommended in view of the favorable results of these tests. This will take the form of a production test in view of the thirty-minute minimum digestion time specific in the Operating Standards.

The results of the test runs are summarized below.

Digestion Study in LaF_3 Product Precipitations (% 8-1M)

Run No.	First Strike			Second Strike		
	Digestion Time, Min.	% E-3WS-1		Digestion Time, Min.	% E-3W	
		Ave.	Range		Ave.	Range
T-5-12-F-5,6,7,8,9,10	60	---	---	60	0.96	0.88-1.17
T-5-12-B-11,12,13	60	6.7	6.4-7.1	30	0.97	0.95-0.99
T-5-12-B-15,16,17	30	5.3	4.7-6.9	30	0.85	0.72-0.94

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A-4-BP Losses

In T Plant, the $BiPO_4$ by-product (A-4-BP) waste losses have become erratic again. Assays of the cake solution acid after circulation in the precipitator do not indicate the presence of a larger than normal product heel. A change is being made in the C-4 oxidation acid normality from the present 7.5N to 6.5N HNO_3 in an effort to obtain better control of the waste loss at this point. This will begin in the T Plant with Run T-5-12-D-20. A similar change in B Plant will be recommended if T Plant results are favorable.

In Run T-5-12-B-15 the A-4-BP waste loss was 10.5%. Circumstantial evidence indicates incomplete oxidation. There was no heel left in the A-1 Precipitator and no indication of crud in the 17-4P product solution. A check of the Cell C and Cell A operation revealed no deviation from standard procedure. It is of interest to note that, in this case as well as with T-5-11-F-19 (A-4-BP = 22.4%), the very high waste loss followed a period of erratic A-4-BP waste losses in the range of 0.4 to 1.0%. Also, immediately following the very high loss, the A-4-BP losses returned to normal. The A-4-BP of T-5-12-B-15 was successfully reworked (0.12%) using the same procedure as used in T-5-11-F-19. It was returned to the A-1 Precipitator, HNO_3 added to increase the volume for good agitation, reoxidized at 5N HNO_3, reprecipitated, and carried on as an 20% volume run.

A-1 Heel and A-4 BP Product Assays (% 8-1M)

Run No.	% Prod. A-1 heel (A-1-AW)	% A-4BP
November Ave.	----	0.36
T-5-12-F-7	----	0.44
F-8	----	0.43
F-9	----	0.45
F-10	----	0.30
T-5-12-B-11	----	0.92
B-12	----	0.66
B-13	----	0.51
B-14	0.07	0.57
B-15	0.28	10.5
B-16	0.06	0.30
B-17	0.24	0.31
B-18	----	0.33

Isolation Building

Waste Loss - Run B-5-12-D-8

In this run the product solution in the still was inadvertantly jetted to the Still Receiver SR-1 before evaporation. This abnormally large volume of solution was partially drained off to the sump tank through the overflow line from SR-1. The product remaining in the overflow line and that was largely recovered but the sump tank material (containing above seventy-three units) was jetted to the dry well. No attempt was made to re-work the sump tank material because of the health hazards likely to be encountered in returning several thousand gallons from the sump to the Concentration Building in equipment not specifically designed to that purpose.

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Possible One Cycle Isolation Process

Additional analytical data have been obtained in the study of purification by the present isolation process. Samples of the first peroxide cake solution after filtration through Nutsche N-2 (coded P-2-P-1) and of the second peroxide cake solution before transfer to Still S-1 (coded P-2-P-2) were analyzed for iron and gross soluble impurities on the runs. These values are compared with similar data for P-1 (solution received from Concentration Building and filtered) and AT (concentrated product solution before loading to sample can) in the following table:

Run	B-5-12-D-6		T-5-12-F-7	
Analysis	% Soluble Impurities*	Total mols Fe	% Soluble Impurities*	Total mols Fe
Sample				
P-1 N-1	750**	0.25	750**	0.32
P-2-P-1 1st-N-2	25	0.014	26	0.01
P-2-P-2 2nd	4.1	0.015	10	0.037
AT AT	1.5	0.018	1.6	0.024

*Based on weight of product

**Approximately calculated from weight of lanthanum in standard P-1

The above values for P-2-P-1 impurities are higher than were found on four previously reported runs (14.2, 17.9, 6.1, and 7.7% for T-28, T-34, B-23, and B-29 in the 5-11 Series). It is interesting to note that the first cycle has reduced both the iron and gross impurity level by a factor of twenty-five to thirty in B-6 and T-7; this was also true if the iron removal in T-28, the only one of the previous four runs for which reliable iron data are available. The gross impurity level on the four previous runs, however, was reduced by factors varying from forty to one-hundred twenty-five. Calculations show that this is easily accountable by assuming that heel volumes after decanting differed by as little as 3.5 liters.

In any single precipitation isolation process with multiple washing, minimum possible volumes after decanting would be desirable for quality control. Opposed to this would be the desire to avoid recycling large amounts of product to the Concentration Building; this requires stopping a decanting when the upper fringe of the cake is reached, even if a heel of seven or eight liters is left.

Examination of the P-2-P-2 and AT data in the table above shows apparent discrepancies. The P-2-P-2 values are not felt to be accurate, since it is not conceivable that evaporation in the still could reduce the iron and gross impurity content. The concentrations of product and impurities in the P-2-P-2 samples are so low in comparison with those in AT samples that unsatisfactory results can be expected when only small volumes of solution are available for analysis and the methods used are those designed for AT solutions.

It is planned to sample another pair of plant runs for the same analyses. Heel volumes after each decanting will be requested and an effort made to correlate the results.

Oxalate Method of Handling Recycle

Several recent runs in Cell four have resulted in rather large final recycle volumes and, in one case (T-5-12-B-14), two RC cans were necessary. This volume increase is associated with larger weights of permanganate used in destroying oxalate and may have resulted from improper functioning of the potentiometric titrometer used to locate the end-points.

Filtration of First Product Cake Solution

Three questions were raised on this subject in the last weekly report. The first concerned the necessity for continuing the filtration through N-2. Examination of N-2 in Cells 3 and 4 (visually) and observation of several filtrations revealed considerable "crud" admixed with the filter aid. The filtration therefore seems justified in that this material is kept out of the second cycle and hence the sample can.

The second point related to the necessity of using filter aid. The records show that this was apparently justified during early months of plant operation. The current need for the use of filter aid could only be established by omitting its use on a number of plant runs. Such a test does not seem in order now because the N-2 filtration rate has been excellent in recent months. From thirty to fifty runs have processed in each cell since it was last necessary to backwash N-2.

The third item was specification of the test manner of using filter aid (concentration, degree of agitation, etc.) The amount of filter aid was varied in several production tests without any clear conclusion being reached. It thus seems that a wide latitude in filter aid concentration is permissible and that present conditions are probably as good as any. Although no formal test of degree of agitation was made, it appears that the current mode of operating (no agitation in N-2) is as satisfactory as any other tried.

Leak in Weigh Tank (Wt.) Water Seal

Some ten days ago it was discovered that water added to the seal on WT in Cell 3 was slowly leaking into WT. It was observed that 10 grams leaked through in a fifty-minute period. The majority of the liquid thus obtained in WT has in the past drained to a spare sample can before loading from AT to WT to SC.

To avoid the hazardous job of replacing this tank, it has been decided to abandon the addition of water to the WT water seals.

Process Chemistry

Appraised of Activity Deposited on 100 Area Slugs

All the experimental work on identifying the active constituents in the film from 100-F slugs (Push of 11-8-45) is now complete. A status report is being written that will furnish all the experimental details.

Influence of the H_3PO_4 Concentration on $BiPO_4$ Extruction Yield

The carrying of plutonium from 24% UNH was again tested in the laboratory on a liter-scale with 25 mg Pu/liter. The phosphoric acid concentration and method

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of strike were varied. The results are definitely in line with those obtained in the previous runs.

H_3PO_4 Concentration	Type of Strike	% Pu in Effluent New Data	Reported 12/3/45
0.8 M	Direct	0.49	0.31
0.6 M	Direct	1.16	1.6
0.6 M	Combination	0.24	0.1

Isolation of Pure 95^{241}

The isolation of a sample (λ) of pure 95^{241} has been completed. It was free of lanthanum and was satisfactorily separated from plutonium by hexone extraction of the latter. The sample will be used for pile irradiation studies.

Mass Assignment Studies on 95^{241}

The 95^{241} La sample was used in a mass spectrograph test. Both a target plate and an "alpha" plate was made. No lines were observed, either for 95^{241} or for the Th^{232}, which was run on the same plate as a standard. There still remains the possibility that the alpha tracks on the "alpha" plate will yield sufficient information to clarify some of the isotopic data.

M. F. Acken

M. F. Acken, Chief Supervisor
200 Area Technical Division

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#1	R. M. Evans
#2	R. M. Evans
#3	Area Engineer
#4	Area Engineer
#5	Area Engineer
#6	Area Engineer Attention: Patent Group
#7	N. Hilberry
#8	N. Hilberry
#9	N. Hilberry
#10	B. H. Mackey - D. A. Miller - '00 Area
#11	M. H. Smith
#12	L. Squires
#13	300 Area File
#14	Pink Copy
#15	Yellow Copy
#16	M. F. Acken
#17	W. C. Kay - F. B. Vaughn
#18	R. S. Apple
#19	R. H. Beaton
#20	W. H. Sullivan
#21	G. W. Struthers
#22	J. D. Ellett
#23	J. J. Urban
#24	W. K. MacCready

December 28, 1945

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200 AREAS
December 18 through December 26

Canyon Buildings

Removing Coating Materials From Special Slug (T)

As assistance to the 100 - 300 Area Technical group in determining the cause of "pimpling" on certain irradiated slugs, one such slug was decoated (Production Test SE-221-T-PA-7) and returned for inspection. The coating removal was done during the course of a regular coating removal operation, the single slug being lowered through a spare nozzle in the top of the dissolver by means of a special basket. No difficulty was encountered in inserting or removing the slug or basket and the coating removal was sufficiently complete to permit accurate observation of the slug surface.

Neutralized Second Cycle Wastes (B and T)

Samples were obtained from the neutralized second cycle waste tanks at each plant and analyzed with the following results:

Tank	110 T	111T	110B	111B
P H	9.3	9.0	7.9	6.5
A (c/mm)	700	348	273	58
B (c/mm)	58,000	18,000	14,000	6,800
G (c/mm)	18	10	13	71
Sp. Gr. @ 23° C	1.104	1.100	1.112	1.110

These data show that these wastes, when adjusted to essential neutality, have approximately the same composition as the neutralized wastes from the Concentration Building, which are now disposed of through a dry well:

A = 60 c/mm; B = 1,800 c/mm; G = 24 c/mm.

Neutralized Waste Storage (B and T)

Installation of the transfer lines from 241 T to 241 U is approximately 5% complete. A summary of the reserve capacities and solution temperatures in 241 B and

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Dec 18-26 '45

41 T are presented below:

Tanks	Type Waste	Gal/run	Reserve Capacity as of December 24, 1945 B Plant Gallons	 Runs	 T Plant Gallons	 Runs
X-101, -2, -3	Metal	5,700	445,000	78	250,000	73*
X-107, -8, -9	Ctng. & 1st Cycle	4,700	610,000	130	395,000	84
X-110, -11, -12	2nd. Cycle	3,600	880,000	244	670,000	186

*Assuming "class C" metal will be processed as 60% extraction volume. The spare tanks (X- 104, -5, -6) have not been included in the above calculations of reserve storage capacity.

Temperatures (F°)

		Metal WAste			First Cycle			Second Cycle	
Plant	Date	101	102	103	107	108	109	110	111
T	7/17/45	118	---	---	94	---	---	88	---
	8/21/45	132	---	---	102	---	---	90	---
	9/20/45	138 (Full)	102	---	94 (Full)	86	---	89	---
	10/23/45	146 (Full)	92 (66% Full)	---	95 (Full)	90 (53% Full)	---	89 (Full)	93 (17% Full)
	11/26/45	151 (Full)	115 (Full)	90 (11% Full)	93 (Full)	84 (95% Full)	---	85 (Full)	85 (48% Full)
	12/24/45	147 (Full)	118 (Full)	102 (53% Full)	93 (Full)	82 (Full)	80 (25% Full)	84 (Full)	82 (76% Full)
B	7/17/45	110	---	---	80	---	---	78	---
	8/21/45	136	---	---	90	---	---	84	---
	9/20/45	142	---	---	96	---	---	86	---
	10/23/45	138 (Full)	106 (36% Full)	--	100 (Full)	78 (19% Full)	---	82 (86% Full)	---
	11/26/45	143 (Full)	118 (83% Full)	--	104 (Full)	80 (58% Full)	---	80 (Full)	73 (12% Full)
	12/24/45	137 (Full)	121 (Full)	83 (16% Full)	107 (Full)	77 (85% Full)	---	76 (Full)	76 (33% Full)

Activity on the Ventilating Fans (B and T)

The relocated ionization chambers on the fans at the B plant continue to show about a factor of ten between the No. 1 (idle) and No. 2 (running) fans. However, as the No. 1 Fan is run occasionally, to permit dissolver operation during periods of low atmospheric dilution, no particular significance can be attached to its readings. Of interest is the fact that the activity on the No. 2 Fan has not varied significantly ($35 \pm 1 \times 10^{-11}$ Amp.) during the past two weeks. This may indicate that the decay rate is equal to the rate of increase. This same phenomenon has been observed with the poorly placed chambers on the fans at the T Plant. (The ionization hambers at the T-Plant fans will be relocated to a more favorable position as soon as experience at the B Plant establishes the optimum location.)

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Concentration Buildings (224)

F-10 Tank Flush

Partial plugging of the B Area F-10 drain valve has resulted in the setting up of a monthly flush of the F-10 system to remove solid matter. This crud is thought to be MnO_2 plus other acid insolubles which collect in the unagitated F-10 tank. The first routine flush was carried out this week and resulted in the removal of a considerable quantity of black crud. The flushing procedure consists of air sparging successive portions of nitric acid and water in the F-10 tank, draining the tank while agitating, and getting the flush to E-4.

Production Tests

Production test SE-224-B-PA-3 has been written covering the proposed dummy runs evaluating an improved crystalline LaF_3 carrier. These tests are to be carried out in the Cell B equipment of the East Area. If successful they will provide the basis for a second production test evaluating the use of crystalline LaF_3 in production runs as a tool for reducing product waste losses (E-3-W) in the product LaF_3 precipitation.

HF Spargers- T Area

The HF spargers in both Cell D (LaF_3 by-product cell) and Cell E (LaF_3 product cell) have been replaced after approximately 200 runs. The new spargers are like those in B area and consist of an inner one-inch dip tube extending ten inches below the end of an outer two-inch dip tube. The one-eighth inch inner dip tubes being replaced were badly corroded, losing twenty inches in cell D and thirty inches in Cell E. The Cell E two inch outer dip tube had shortened ten inches. The cell D outer pipe had been etched but was still in good condition and was not replaced. These spargers will be checked again after approximately 150 runs.

Isolation Building (231)

Product Content of Recycle Solutions

Data for fifty-one recent runs (since T-5-11-F-11 and B-5-11-B-9) were examined in an attempt to correlate the H_2O_2 concentration during the first cycle precipitation with the total amount of product recycled. The control of H_2O_2 reagent concentration was good during this period and other factors determined the weight of product recycled. This is evident from the following brief summary:

H_2O_2 Concentration in P-1	No. of runs in group	Ave. units recycled for run
< 8.0	8	9.2
8.0 - 8.1	6	9.6
8.1 - 8.2	16	9.6
8.2 - 8.3	11	8.9
> 8.3	7	9.1

Accounting of recycled product

In cross-checking the weight of product recycled per fun from the Isolation Building to the Concentration Building, the RC and E-4 data for several months were examined. Since no attempt is made to assay E-4 tank after every recycle is received it was necessary to study "month-end" numbers. It would seem that the amount of product in E-4 tank at the beginning of a month, plus the sum of the amounts added to E-4 (both recycles from 231 and kittens, washes, etc from 224), should balance the amount of E-4 at the end of the month plus the sum of the amounts jetted out of E-4 as recycles to production runs. In the following table the total units returned to E-4 plus the E-4 beginning inventory appears in column two while the total units removed from E-4 plus the E-4 ending inventory appears in column three for each month; the excess of column two values over column three appears in column four:

Month Ending	Col. 2	Col. 3	Col. 4
Aug. 25	385.4	366.4	19
Sept. 25	756.6	583.2	173.4
Oct. 25	1000.8	933.3	67.5
Nov. 25	960.3	851.7	108.6
Four Month Total	3,103.1	2,734.6	368.5

The discrepancy (column 4) is over ten per cent of the total amount of product handled. If it continues and no adjustment of the books is made, E-4 tank will appear richer than it actually is. Among the factors possibly responsible for the difference might be listed: (1) High assay of recycle in 231 building, due either to (a) high volume calibration of CT-1 tanks or (b) high values for CT-1-R analyses; (2) low assay of E-4 tank contents due either to (a) low weight factor readings or calibration, (b) high sp. gr. readings, or (c) low values for E-4 RC analyses; (3) transfer of lower volumes out of E-4 tank than indicated by instruments.

Process Chemistry

Problems - 200 Area

Analysis of Second Cycle Waste Now in Storage

On December 12, 1945 samples of waste were removed from the 110 W, 111W, 110 E, and 111 E tanks for analysis. The object was to determine whether the decrease in alkalinity had lowered the product content in solution, as predicted in SE- PC No. 82. The results obtained are as follows, and are compared with both previous and predicted results.

Measurement of Activity in Waste Solutions

Source	α/min/cc	M g Pu per liter	B/min/cc 2 nd Shelf	γ/min/cc 2 nd Shelf	P H	Sp G at 23 °C
110 W	700	9	5.6×10^4	18	9.30	1.104
111 W	348	4.5	1.73×10^4	10	8.95	1.100
110 E	273	3.5	1.1×10^4	13	7.85	1.112
111 E	58	0.8	6.5×10^3	71	6.45	1.110
As Measured July 13, 1945						
110 W	1090	15	4.9×10^4	30		
As Measured July 26, 1945						
110 W	1320	19	6.9×10^4	53	9.8	
110 E	600	8.5	7.0×10^4	35	9.6	

As Predicted in SE-PC 82 for low P H. Based on Laboratory Results.

	α/min/cc	M g Pu per liter	B/min/cc 2 nd. Shelf	γ/min/cc 2 nd Shelf	P H
110 W	980	13	5.0×10^4		8.2
[illegible]10 W	63	0.84	2.9×10^3		6.0

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As can be seen from the above data, the product content is lowered appreciably by lowering the pH. The assays of the solutions recently taken from the tank farm are close to the laboratory results at comparable pH values.

Corrosion of 25- 12 Cb Steel in H F Solutions

The corrosion of 25 - 12 Cb steel, previously treated with molten potassium dichromate to passify it, has been determined in H F solutions of several concentrations. The oxide layer was completely removed in a relatively short time, and the treatment does not passify the steel to H F corrosion.

A temperature of 45°C was chosen, since that is the maximum temperature that is reached in E1 - E2 operations. The treated steel appeared to have corroded slightly greater than the control but this is undoubtedly due to the loss in weight of the oxide film.

Mass Assignment Studies on 95^{241}

The results of the mass spectrograph run involving 5×10^7 Cts/min of 95^{241} indicated that the amount of lanthanum associated with the material was of such quantity that a blurred spread-out image on the alpha plate was produced. Another sample of 95^{241} is being prepared which will be free of lanthanum and plutonium, and the test will be repeated using the purified material.

Mass Assignment Studies on 1yr. Ruthenium

Since the mass spectrograph is now available for use, tests are being made to determine a suitable state in which ruthenium can be used in the instrument to determine the mass mnmber of the 1 y isotope formed in fission. Approximately 5×10^8 d/m of this isotope had previously been purified and concentrated in about 100 λ of HCl solution. It has been found (using stable Ru) that RuO_2 applied as a thick ethanol slurry emits well and appears to have a low loss factor. Tests will soon be made with the active Ru.

M. F. Acken per

M. F. Acken, Chief Supervisor
200 Area Technical Division

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#2	R.M.Evans	#14	Pink Copy
#3	D.A.Miller for The Area Engineer	#15	Yellow Copy
#4	D.A.Miller for The Area Engineer	#16	M.F.Acken
#5	D.A.Miller for The Area Engineer	#17	W.C.Kay - F.B.Vaughan
#6	D.A.Miller for The Area Engineer Attention: Patent Group	#18	R.S.Apple
#7	N. Hilberry	#19	E.R.Gilbert
#8	N. Hilberry	#20	W.H.Sullivan
#9	N. Hilberry	#21	G.W.Struthers
#10	D.A.Miller-T.N.Stapleton-700 Area	#22	J.D.Ellett
#11	M.H.Smith	#23	J.J.Urban
#12	L.Squires	#24	W.K.MacCready

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January 4, 1946

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200 AREAS
December 25 through December 31

Canyon Buildings

Processing "Class C" Metal (T)

The extraction conditions being employed for "Class C" metal are essentially those previously employed for "Class A" metal; they differ in the following respects: (1) volume is reduced to approximately 60% of previous volume and (2) bismuth concentration is either the same (with decreased Bi/Pu ratio) or greater (with the same Bi/Pu ratio).

The results to date show an abrupt and consistent increase in waste loss resulting from processing the greater enriched metal. The effect of increased bismuth concentration or decreased Bi/Pu ratio has not been apparent on either the extraction waste loss or the canyon decontamination factors:

Run No.	Cooling Time to Extraction, days	Canyon Decon. Factor, G-dF	Bi Conc., g/l	8-3-W, %	Bi/Pu
12-F-30	107	5.11	4.22	1.0	96
F-31	108	5.14	4.05	1.4	97
F-32	109	5.14	2.45	1.4	62
D-33	80	5.40	2.50	1.3	53
D-34	81	5.44	4.52	1.7	91
D-35	82		4.74	1.7	97
D-36	83		4.87	1.9	91
"Class A"(25 runs)	45	5.26	2.5	0.74	95

Neutralized Wastes (B and T)

The summary of neutralized second cycle wastes contained in the previous report showed the waste from the T Plant to have significantly higher pH than that from the B Plant. Less precise measurements made on the first cycle wastes showed the T Plant wastes to be about pH 9 and the B Plant wastes to be about pH 8. A survey of nitric acid and caustic strengths delivered to each plant during the last quarter of 1945 failed to provide a satisfactory explanation for this discrepancy:

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	B Plant		T Plant	
	Range	Ave.	Range	Ave.
HNO_3 (%)	59.22-64.50	60.76	59.98-62.94	61.14
NaOH (%)	47.22-50.34	49.16	48.13-51.50	49.44

Concentration Buildings

$BiPO_4$ By-Product Waste Losses (A-4-BP)

The reduction in nitric acid concentration from 7.5N to 6.5N during oxidation has resulted in lower product losses in the $BiPO_4$ by-product precipitation. Beginning with Run T-5-12-D-20, the nitric acid concentration during oxidation in the C-4 Tank was reduced in an attempt to correct erratic A-4-BP performance in T Plant. Slower decomposition of the sodium bismuthate oxidizing agent occurs at the lower acidity, thus lengthening the time during which oxidation can occur. Because of the apparent increase in oxidation safety factor obtained in this test , it will be recommended that B Plant use 6.5N acidity for oxidation even though no difficulty is being experienced at the present time.

The results of the test runs are tabulated below:

Effect of HNO_3 Concentration During Oxidation on A-4-BP Losses

Run No.	Number of Runs	HNO_3 Normality at Time of Oxidation	A-4-BP (% 8-1-MR)	
			Ave.	Spread
T-5-12-F-1 to T-5-12-B-19	18*	7.5	0.40	0.25-0.88*
T-5-12-D-20 to T-5-12-F-32	13	6.5	0.26	0.19-0.36

* Does not include 10.5% A-4-BP of T-5-12-B-15

Lanthanum Strike Digestion

A Production Test SE-224-T-PA-7 is being written covering the proposed elimination of the 1-hour digestion time following each lanthanum strike.

Isolation Building

Possible One Cycle Isolation Process

An attempt has been made to correlate "purity" found at several points in the present process with the theoretical purity predicted on the basis of volumes of heels left after decanting. Contrary to expectation, the data shown below fail to reveal any such correlation:

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	B-5-12-D-24		T-5-12-D-25	
	Theoretical	Experimental	Theoretical	Experimental
P-2-P-1 purity	92%	79%	89%	87%
P-2-P-2 purity	99.7%	95.5%	99.8%	95.4%
AT purity	<99.7%	98.8%	<99.8%	98.8%
P-1 Fe (mols)	--	0.276	--	0.206
P-2-P-1 Fe (mols)	0.0033	0.0084	0.0035	0.0168
P-2-P-2 Fe (mols)	0.0001	0.0095	0.00006	0.0104
AT Fe (mols)	>0.0001	0.0162	> 0.00006	0.0117

(P-2-P-1 sample after first cycle and subsequent filtration; P-2-P-2 sample after second cycle but before concentration in still).

If the analytical values are accepted, the conclusion is reached that factors other than volumes before and after decanting govern the rate of removal of lanthanum and iron by precipitations of product peroxide and multiple washes.

To summarize the data scattered through the past few reports, the following tables are presented:

Percent Purity

Sample	B-23*	T-28*	B-29*	T-34*	T-7‡	B-6‡	T-25‡	B-24‡
P-2-P-1	93.9?	85.8	92.3	82.1	74	75	87	79
P-2-P-2	--	--	--	--	100	95.9	95.4	95.5
AT	98.9	98.1	99.0	98.1	98.4	98.5	98.8	98.8

Total Mols of Iron Present

Sample	B-23*	T-28*	B-29*	T-34*	T-7‡	B-6‡	T-25‡	B-24‡
P-1	0.16	0.20	0.22	0.23	0.32	0.25	0.21	0.28
P-2-P-1	--	0.0083	<0.03	<0.03	>0.009	0.014	0.017	0.0084
P-2-P-2	--	--	--	--	0.037	0.015	0.010	0.0095
AT	0.023	0.022	0.0051	0.011	0.024	0.018	0.012	0.016

* 5-11 series
‡ 5-12 series

Among the conclusions that may be drawn are the following:

(1) Purity after the present first cycle is erratic, varying from 74 to 94% in 8 runs tested;
(2) Purity after two cycles and concentration is consistently high;
(3) Purity after two cycles but before concentration is apparently lower (in 3 of 4 cases) than after concentration;
(4) Iron removal is good in the first cycle but apparently very poor in the second;
(5) If item (4) is correct, then still corrosion during evaporation adds little iron to that already present.

Item (3) in the above list could only be true if the concentration in the still resulted in conversion of soluble to insoluble impurities (since the latter are not determined by the present "purity" assay). This would require that the AT solutions from Runs B-6, T-25, and B-24 contain 2100, 2700, and 2600 milligrams of insoluble solid per 80 units of product. The most recent (5-05 and 5-06 series) data showed less than 100mg. solids per 80 units, when Becco H_2O_2 was used (as at present). Even with Albone H_2O_2 no value above 1500 mg./80 units was found in tests made during the 5-04 and 5-05 series.

In view of the erratic fluctuations in purity throughout the process, it appears that a true appraisal of the merits of a single cycle process can only be evaluated by a production test in which material from one precipitation and five or six washes is taken to the still, evaporated, and transferred to AT for purity measurement. If further purification is required on the test runs, the AT solution can be dropped to ST-2 Tank and returned to Precipitator P-2.

M. F. Acken

M. F. Acken, Chief Supervisor
200 Areas Technical Division

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