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TECHNICAL REPORT ON THE EXPANDED
HARSHAW TBP EXTRACTION PROCESS

W.O. #46205

Issued May 11, 1954

AEC RESEARCH AND DEVELOPMENT REPORT

THE HARSHAW CHEMICAL COMPANY

CLEVELAND, OHIO

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TECHNICAL REPORT ON THE EXPANDED
HARSHAW TBP EXTRACTION PROCESS

BY

D. A. HAMMOND

(Work done by J.G.Barrick, M.F.Rejdak, J.R.Baird, and D. A. Hammond)

ABSTRACT

The tributyl phosphate extraction system installed at the Harshaw Chemical Company in September 1951 has been successfully modified to make available increased production capacity required by the Atomic Energy Commission as of September 1952. The following studies were made in the pilot plant to clearly establish the extent of the necessary modifications.

1. Maximum flow rates usable in the mixer-settler unit.
2. Optimum number of stages in the stripping section.
3. Effect of nitric acid concentration and effect of temperature on phase disengagement time in the stripping section.
4. The effect of various concentrations of tributyl phosphate upon the concentration of uranium in the OK liquor.
5. The effect of tributyl phosphate degradation products on the extraction, scrubbing, and stripping operations.
6. Reconditioning of impure solvent for extraction purposes.
7. Press cake solvent removal.

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8. Improved method for tributyl phosphate analysis.

Discussion of these studies is presented; complete data are in Appendix B. Data on and discussions of analytical methods are in Appendix A. Conclusions, together with recommendations for further work, precede the appendices.

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I. INTRODUCTION

A tributyl phosphate-kerosene extraction system was installed at Harshaw Chemical Company in September, 1951 for the production of UO_3 . The system was a mixer-settler counter current extractor with fifteen units, six extraction stages, three scrubbing stages, five stripping stages and one solvent washing stage.

The feed material was digested in nitric acid and adjusted to have a final composition of 300 g./l.U and 1 M free HNO_3 . This slurry was run into the extraction system unfiltered. The raffinate, containing 0.01 g./l.U or less, was run to the sewer. Most of the nitric acid insoluble matter was carried out with the raffinate.

The rate of flow of the solvent was constant after being set at some predetermined value. The rate of flow of feed slurry was automatically controlled within limits by means of an instrument recording the density of the solvent from the stage ahead of the one into which the feed slurry was entering. This solvent was approximately 50 percent saturated with uranium and gave the most sensitivity with the least time lag for a slurry control. In order to eliminate emulsion problems the recording instrument was designed to maintain a ratio of not more than one volume of slurry to four volumes of solvent.

The saturated solvent from the extraction section was then scrubbed with city water at a 15:1 ratio of solvent to water. This scrubbing removed any entrained slurry, the small amount of acid present

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in the solvent, and about 20 percent of the uranium. The scrubbing section was effective in almost complete removal of impurities, including the rare earths, from the solvent (1). The aqueous phase from this section, called N.G. liquor, was used to cut the digest liquor to the correct concentration for feed slurry.

The scrubbed solvent was stripped in 5 stages with condensate water from the boildown section. A water still supplied whatever make-up strip water was necessary. The ratio in this section was approximately 2:1 solvent to water. The condensate rate of flow was held constant at a certain set ratio by the solvent flowrator. The ratio was adjusted manually and periodically as the stripping conditions changed. The OK liquor from the stripping section contained about 80 g./l.U and ran by gravity into the first boildown tank. The stripped solvent contained little or no uranium.

The stripped solvent was washed in one unit with water held to a pH of 7 or above by a controlling and recording pH meter. Dilute NaOH (0.5%) was added by this device whenever the pH fell below seven. This washing stage removed residual uranium and the degradation products of the TBP kerosene mixture. The solvent was returned to the solvent storage tank and the aqueous solution pumped to a recovery tank. The precipitated diuranate was filtered through a plate and frame filter press.

The extraction system operated satisfactorily at its rated capacity. A few conditions arose which could be improved to give

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smoother operation:

1. The feed slurry flowrator would stick and not operate properly due to acid insoluble particles collecting in it.
2. Press cake from the uranium recovery operation contained a small amount of solvent which, when digested in nitric acid at 90°C, gave organic products which tended to increase the disengagement time of the two phases in the extraction section. This led to incomplete separation in each stage, and the slowing up of the flowrates. It was also necessary to completely replace the solvent periodically.
3. Emulsions formed when solvent, containing an excess of uranium, entered the washing stage and the addition of caustic solution was too rapid. Large losses of solvent occurred at this time.

In September, 1952 the Atomic Energy Commission asked Harshaw Chemical Company to make any necessary changes in order to increase the production between four and five times. It was also necessary to get these changes made in the shortest possible time and in the mean time continually increase the production.

To determine what changes would be necessary, considerable pilot plant investigation was required. It was necessary to determine the maximum flow rates usable in the mixer-settler unit. These flow-

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rates determined the concentration of U in the OK liquor necessary to achieve the production goal. Then solvent studies were run to determine the effect of various concentrations of TBP and ratios upon the concentration of uranium in the OK liquor.

The much larger volume of solvent used in the extraction system necessitated some pilot plant work on the reconditioning of impure solvent for extraction purposes.

This report is intended to be a technical guide to any future changes to be made in the extraction system at Harshaw and to give extraction data to aid the operation of the unit when changes occur, as a change in feed material.

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II. EXTRACTION EFFICIENCY

A. Fourteen Stage System

The shakeouts were set-up to duplicate the plant mixer settler units as nearly as possible. The disengagement times in the shakeouts were approximately two thirds that of the same stage in the extraction system. The centrifugal pump on the mixer tank, a LaBour type 12 DZT pump, apparently made a more finely divided mixture of the solvent and aqueous than could be obtained by shaking a separatory funnel for thirty seconds. The two phases reached equilibrium under these conditions.

In this series of shake-outs the first run included the extraction and scrubbing stages to set up the proper U and HNO_3 concentration in the saturated scrubbed solvent. Then, all of the stripping tests were run using this solvent. The solvent washing was not included in these runs. Figure 1 shows the profile of the first run (2). The points where the horizontal line indicating the stages on the profile crosses the curves, represent the equilibrium concentration of the two phases as they leave that stage. For example in figure 1, line 10 cuts the aqueous curve at 290, which indicates that the aqueous phase leaving stage 10 contains 290 milliequivalents of U. In this case the aqueous is the OK liquor from the stripping section and contains 725 m.e./l.U. The line 10 cuts the organic curve at 250 m.e. The solvent leaving stage 10 and entering stage 11 has a concentration of 500 m.e./l.U.

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FIG. 1

PILOT PLANT SHAKEOUT-14 STAGE-2320% TBP

FLOWS

ORGANIC—500 ML.
SLURRY—110 ML.
SCRUB—33 ML.
STRIP*—400 ML.

*DISTILLED WATER AT 25°C

AQUEOUS PHASE

ORGANIC PHASE

- ME U IN ORGANIC AND AQUEOUS
- ME HNO₃ IN ORGANIC
- ME HNO₃ IN AQUEOUS
- ORGANIC 0.8537M TBP

14
13
12
11
10
9
8
7
6
5
4
3
2
1

S
T
R
I
P
P
I
N
G

S
C
R
U
B

E
X
T
R
A
C
T
I
O
N

500 400 300 200 100 0 0 100 200 300 400 500
TOTAL MILLEQUIVALENTS

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Table 1 containing all the data obtained from the first test follows the report in appendix B.

The flow rates of this run were 500 ml. solvent, 110 ml. feed slurry, 33 ml. scrub water, and 400 ml. distilled water at 25°C as strip water. The flow rates quoted in this report were the volumes of incoming streams. As the solvent became saturated, its volume increased to approximately 510 ml., but the amount in any one stage varied because the wall effect of the funnels and the varying drain times. In the extraction plant there would be no wall or drain effects but it would be impossible to check the flowrates since many other factors are involved.

The saturated solvent from the last extraction stage was approximately 97% saturated. The scrubbing section removed nearly 17.5% of the uranium along with the impurities. As can be seen from the profile in figure 1, the stripping section was not effective in removing all the uranium from the solvent as 5.7% of the amount in the saturated solvent remained in the stripped solvent.

To check the effect of temperature on the stripping efficiency, the next run was made using a distilled water strip, which was maintained at 50°-55 °C during the stripping operation. The stripped solvent contained 0.7% of total U and the concentration of the OK liquor was 95.1 g./l.U as compared to 85.0 g./l.U for the cold strip. The disengagement time was lowered slightly by the higher temperature. The stripping operation was improved very significantly by the increase in temperature.

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FIG. 2

PILOT PLANT SHAKEOUT
5 STAGE STRIP
23.20% TBP

FLOWS

ORGANIC—500 ML.

AQUEOUS*—400 ML.

□ ME U IN ORGANIC AND AQUEOUS

▨ ME HNO_3 IN AQUEOUS

▨ ORGANIC 0.8537 M TBP

*DISTILLED WATER AT 25°C

AQUEOUS

PHASE

ORGANIC

PHASE

TOTAL MILLEQUIVALENTS

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In the preliminary pilot plant tests longer disengagement times were encountered than anticipated from previous experience. Clean solvent and city water were being used to get settling time data for the final stripping stage. Since the strip water used by the production plant was mainly boildown condensate, it contained a small amount of HNO_3 as well as some uranium. To simulate the condensate strip a small amount of HNO_3 (0.2g./l.) was added to distilled water. The acid strip water lowered the disengagement times approximately ten percent.

An added verification of this effect was given by the plant when it was necessary to use deionized water to make up for the loss of condensate during a period of expansion. As soon as the deionized water got to the stripping section, the production personnel had difficulty maintaining normal flow rates, due to slower disengagement of the two phases.

Since an acid strip was beneficial from the disengagement standpoint, it was necessary to check its effect upon stripping efficiency. A shake-out was made to compare the efficiency of this strip water at 50°C to that of distilled water at 50°C . The results indicated that the addition of HNO_3 aided the stripping of solvent, which appeared to be contrary to the theory that HNO_3 improves the extraction of U, from the aqueous phase to the organic phase. The OK liquor contained 101.4 g./l.U as compared to 95.1 g./l.U for the distilled water strip. The stripped solvent contained only 0.26% of the total U. Figures 2 and 3 show profiles of these two shake-outs.

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FIG. 3

PILOT PLANT SHAKEOUT
5 STAGE STRIP
23.20% TBP

□ ME U IN ORGANIC AND AQUEOUS

▨ ME HNO_3 IN AQUEOUS

▨ ORGANIC 0.8537 M TBP

▨ ME HNO_3 IN ORGANIC

FLOWS

ORGANIC — 500 ML.

AQUEOUS — 400 ML.

AQUEOUS

PHASE

ORGANIC

PHASE

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From the results of this test it was decided that an acid strip water, 0.2 g./l. HNO_3 , would be used in all the succeeding shake-outs and that the temperature would be maintained in the range of 50°C to 55°C. throughout the stripping section.

It was apparent that the flow rates in the stripping section would control the maximum production possible at the Harshaw installation. In order to determine the maximum U concentration of an OK liquor the system could produce, and the amounts of U the recovery room would have to handle, a series of runs were made with successively smaller flows. With a constant solvent flow of 500 ml. the stripping flows were as follows: 450 ml., 400 ml., 350 ml., and 300 ml. The U concentrations of the OK liquors were respectively as follows: 84.8 g./l., 101.4 g./l., 106.4 g./l., and 109.3 g./l. The amount of U in the stripped solvent was as follows: 0.16 g./l., 0.24 g./l., 2.50 g./l., and 8.40 g./l. Figure VII shows these results graphically. It appeared that a stripping flow between 400 ml. and 350 ml. would give a maximum throughput of uranium with a low recovery problem. Figures 3 to 6 show profiles of these runs.

Two shake-outs using different stripping ratios were run to compare the extraction data obtained from pilot plant shake-outs with the data from the production extraction system. The shake-outs were run after samples from the production plant had been taken, and it was necessary to make up pilot plant solvent to the same TBP concentration as the solvent used in the plant at the time of sampling. Even though

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FIG. 4

PILOT PLANT SHAKEOUT
5 STAGE STRIP
23.20% TBP

FLOWS

ORGANIC—500 ML.
AQUEOUS—450 ML.

□ ME U IN ORGANIC AND AQUEOUS

▨ ME HNO₃ IN AQUEOUS

▨ ORGANIC 0.8537 M TBP

AQUEOUS

PHASE

ORGANIC

PHASE

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FIG. 5

PILOT PLANT SHAKEOUT
5 STAGE STRIP
22.20% TBP

FLOWS

ORGANIC — 500 ML.

AQUEOUS — 350 ML.

□ ME U IN ORGANIC AND AQUEOUS

▨ ME HNO_3 IN AQUEOUS

▨ ORGANIC 0.8537M TBP

AQUEOUS PHASE

ORGANIC
PHASE

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FIG. 6

PILOT PLANT SHAKEOUT
5 STAGE STRIP
2320% TBP

FLOWS

ORGANIC—500 ML.
AQUEOUS—300 ML.

- ME UIN ORGANIC AND AQUEOUS
- ME HNO₃ IN ORGANIC
- ME HNO₃ IN AQUEOUS
- ORGANIC 0.8537 M TBP

AQUEOUS

PHASE

ORGANIC

PHASE

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FIG. 7

COMPARISON OF SEVERAL STRIPPING RATIOS

ORGANIC FLOW CONSTANT AT 500 ML.

AQUEOUS FLOW AS INDICATED

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the TBP concentrations of the two solvents were very close there were unknown factors which effected the solvent's ability to extract uranium. The saturated solvent from the shake-outs contained ten percent less uranium than the plant saturated solvent. Thus it was not possible to use the same ratios as the plant was using, but the distribution coefficients for the greater part were very close. The concentration of uranium in the two phases at a certain stage were not the same in two systems, but the overall picture was very similar as can be seen in figures 8, 9, and 10. Another comparison will appear later in this report concerning the expanded system.

When the results of the various tests using five stripping stages, and the results of several pilot plant flow rates experiments on a single mixer-settler were analyzed, it was evident that stripping capacity would have to be increased. To reach the required production quota, it was considered necessary to increase the number of stripping stages, and to increase the size of the settling tanks. Due to space limitations five four-foot diameter tanks and four five-foot diameter tanks could be added to the extraction system with a minimum interruption of production.

Since the stripping section was the only section of the extraction system which would have to handle high aqueous flow rates, the proposed nine new units would be stripping stages. The original fifteen units would then be split up into seven extraction stages, four scrubbing stages, two solvent washing stages, and two solvent settlers.

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FIG. 8

PILOT PLANT SHAKEOUT

14 STAGE

17.82% TBP

FLOWS

ORGANIC—500 ML.

SLURRY—92 ML.

SCRUB—33 ML.

STRIP—250 ML.

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FIG. 9

PILOT PLANT SHAKEOUT
14 STAGE
17.82% TBP

FLOWS

ORGANIC—500ML.

SLURRY—92ML.

SCRUB—33ML.

STRIP—300ML.

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FIG. 10

MIXER SETTLER UNIT-15 STAGE-17.84% TBP

FLOWS

ORGANIC — 500 ML.

SLURRY — 102 ML.

SCRUB — 34 ML.

STRIP — 318 ML.

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B. TWENTY STAGE SYSTEM

The next series of tests were set up to check the advisability of making the proposed equipment changes. One series of tests were run to show the effect of different concentrations of TBP in the solvent. Another series of tests were designed to show the overall efficiency of the stripping section each using a different number of stages.

Before any of these tests were run it was found necessary to develop a dependable method of TBP analysis of solvent. The method of analysis and the confirming data follows in appendix A. A rapid method of analysis of feed slurries for concentration of HNO_3 is also found in appendix A.

The initial run of the first series of shake-out tests checking the effect of TBP concentration, used a solvent containing 25.89% TBP. The feed slurry contained 505 g./l.U and 95 g./l. HNO_3 . The saturated solvent contained 104.0 g./l.U and had a specific gravity of 0.992 at 21°C. The scrubbed solvent contained 84.9 g./l.U which gave an OK liquor concentration of 120.9 g./l.U when the solvent was stripped. The profile of this run follows in figure 11.

This percentage of TBP in the solvent is nearly the maximum that will be feasible to operate with the mixer-settler units. As the specific gravity of the saturated solvent approaches 1.00, the phase disengagement times in the scrubbing stages becomes longer and clean separations of phases would be impossible.

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FIG. 11

PILOT PLANT SHAKEOUT

20 STAGE

25.89% TBP

FLOWS

ORGANIC—500 ML.

SLURRY—105 ML.

SCRUB—33 ML.

STRIP—350 ML.

ME U IN ORGANIC
AND AQUEOUS

ME HNO_3 IN ORGANIC

ME HNO_3 IN AQUEOUS

ORGANIC
0.9526M TBP

AQUEOUS
PHASE

ORGANIC
PHASE

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Shakeouts using solvents of the following TBP concentrations: 23.20 percent, 20.44 percent, 17.82 percent, and 14.93 percent, concluded the runs of this series. A profile of each run follows to give an overall picture of the effect of changing the TBP concentration of the solvent. The uranium concentration of the OK liquor, of the saturated, and of the scrubbed solvent, is plotted against the percent TBP in the solvent in figure 16. The 17.82 percent TBP solvent concentrations appear to be low for no apparent reason. There has not been sufficient time to determine the cause of this discrepancy. The OK liquor concentrations on this graph are from a nine stage stripping section.

To more or less complete the analysis of the variables in stripping efficiency, a series of shakeouts were run using different number of stages. The overall efficiency of the stripping section using six, seven, eight, and nine stages was obtained. Enough solvent was saturated and scrubbed by the 7 stage extraction and 4 stage scrub sections so that all the stripping shakeouts would be run using solvent with the identical U concentration. A ratio of flows of 500 ml. solvent to 350 ml. of strip water were set up. Profiles of these runs follow in figures 17 to 20. The results are shown graphically in figure 21 with the U concentration of the OK liquor vs. the amount of U left in the stripped solvent.

The result from the eight stage run appears to have too high an uranium content in the stripped solvent. Shakeouts previous to this series indicate less uranium in the stripped solvent for eight stripping,

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FIG. 12

PILOT PLANT SHAKEOUT

20 STAGE

23.20% TBP

FLOWS

ORGANIC—500ML.

SLURRY—110ML.

SCRUB—33ML.

STRIP—350ML.

- ME U IN ORGANIC AND AQUEOUS
- ME HNO₃ IN ORGANIC
- ME HNO₃ IN AQUEOUS
- ORGANIC 0.8537M TBP

AQUEOUS PHASE

ORGANIC PHASE

600 500 400 300 200 100 0 0 100 200 300 400 500
TOTAL MILLEQUIVALENTS

20

19

18

17

16

15

14

13

12

11

10

9

8

7

6

5

4

3

2

1

S

T

R

P

P

I

N

G

S

C

R

U

B

E

X

T

R

A

C

T

I

O

N

FIG. 13

PILOT PLANT SHAKEOUT
20 STAGE
20.44% TBP

- ME U IN ORGANIC AND AQUEOUS
- ME HNO₃ IN ORGANIC
- ME HNO₃ IN AQUEOUS
- ORGANIC 0.7521M TBP

FLOWS

ORGANIC—500ML
SLURRY—100ML
SCRUB—33ML
STRIP—325ML

AQUEOUS
PHASE

ORGANIC
PHASE

400 300 200 100 0 0 100 200 300 400
TOTAL MILLEQUIVALENTS

20

19

18

S

17

T

16

R

15

P

14

N

13

G

12

11

S

10

G

9

R

8

U

7

B

6

E

5

X

4

T

3

R

2

A

1

C

0

T

31

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FIG. 14

PILOT PLANT SHAKEOUT
20 STAGE
17.82% TBP

FLOWS

ORGANIC—500 ML.
SLURRY—91 ML.
SCRUB—33 ML.
STRIP—325 ML.

- ME U IN ORGANIC AND AQUEOUS
- ME HNO₃ IN ORGANIC
- ME HNO₃ IN AQUEOUS
- ORGANIC 0.6557 M TBP

AQUEOUS PHASE

ORGANIC PHASE

400 300 200 100 0 0 100 200 300 400
TOTAL MILLEQUIVALENTS

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FIG. 15

PILOT PLANT SHAKEOUT

20 STAGE

14.93% TBP

ME U IN
ORGANIC AND AQUEOUS

ME HNO_3 IN ORGANIC

ME HNO_3 IN AQUEOUS

ORGANIC
0.5493 M TBP

FLOWS

ORGANIC—500 ML.

SLURRY—100 ML.

SCRUB—33 ML.

STRIP—250 ML.

ORGANIC
PHASE

AQUEOUS
PHASE

400 300 200 100 0 0 100 200 300 400
TOTAL MILLEQUIVALENTS

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but at this time there is insufficient data available to warrant changing the result. The maximum U concentration possible using an aqueous flow of 350 ml. was approximately 112 g./l.U. If a smaller aqueous flow were used, the line would be above the present one and would have less slope. A flow of 325 ml. gave a theoretical maximum OK liquor concentration of 120.6 g./l.U. A five stage stripping result was not included on the graph since those shakeouts were run using solvent of a slightly higher concentration than the others. Previous work indicated that its result would fall nearly on the line at approximately 3.0 g./l. U in the stripped solvent.

From the above information a nine stage stripping section appears to be optimum. It is believed that the analysis would have been the same if some other reasonable stripping ratio had been chosen. A smaller number of stripping stages would give a less concentrated OK liquor, and to get the same throughput of uranium, a higher aqueous flow would have to be used. It has been seen since the expanded plant has been in operation that our previous estimated maximum aqueous flow rates were not too conservative.

The next series of profiles in figures 22, 23, and 24 shows a comparison of a pilot plant shakeout with two sets of samples taken from the production unit approximately five months apart. During this five months of operation the solvent in the production system had not been changed but only added to with fresh make-up solvent when necessary. The second set of samples were taken to determine if degradation products

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FIG. 17

PILOT PLANT SHAKEOUT
17 STAGE-23.20% TBP

FLOWS

ORGANIC—500 ML.

SLURRY—110 ML.

SCRUB—33 ML.

STRIP—350 ML.

□ ME U IN ORGANIC
AND AQUEOUS

▨ ME HNO₃ IN ORGANIC

▩ ME HNO₃ IN AQUEOUS

▤ ORGANIC
0.8537 M TBP

AQUEOUS
PHASE

ORGANIC
PHASE

400 300 200 100 0 0 100 200 300 400
TOTAL MILLEQUIVALENTS

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FIG. 18

PILOT PLANT SHAKEOUT
7 STAGE STRIP
23.20% TBP

FLOWS

ORGANIC—500 ML.
AQUEOUS—350 ML.

ME U IN ORGANIC AND AQUEOUS

ME HNO_3 IN AQUEOUS

ORGANIC 0.8537M TBP

AQUEOUS

PHASE

ORGANIC

PHASE

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FIG. 19

PILOT PLANT SHAKEOUT
8 STAGE STRIP
23.20% TBP

FLOWS

ORGANIC - 500 ML.
AQUEOUS - 350 ML.

□ ME U IN ORGANIC AND AQUEOUS

▨ ME HNO₃ IN AQUEOUS

▤ ORGANIC
0.8537 M TBP

AQUEOUS
PHASE

ORGANIC
PHASE

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FIG. 20

PILOT PLANT SHAKEOUT
9 STAGE STRIP
23.20% TBP

□ ME U IN ORGANIC AND AQUEOUS

▨ ME HNO_3 IN AQUEOUS

▤ ORGANIC
0.8537 M TBP

FLOWS

ORGANIC—500 ML.

AQUEOUS—350 ML.

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were building up and lowering the efficiency of the extraction system. Except for the fact that the solvents aqueous ratios were not identical, the two production profiles were very similar. The pilot plant shakeout profile agreed very well with that of the production unit. Since closer control was exercised over the solvent aqueous ratios than would be possible in the production unit, the OK liquor concentration was a little higher.

To show the effects of degradation products in the solvent, a shakeout was run using solvent, to which had been added one percent dibutyl phosphate. It was assumed that this concentration was in excess of anything to be expected in the production unit, but a positive result of the dibutyl phosphate was necessary.

The extraction section experienced little difficulty with the added DBP and a nearly normal saturated solvent concentration was obtained. In the scrubbing section the disengagement times were increased considerably. The biggest effect of the DBP was found in the stripping section. It seemed that as soon as the excess acid was stripped out of the solvent, the DBP was an excellent emulsifying agent. It was nearly impossible to reach any kind of an equilibrium in the stripping section. A more staggering problem arose with the necessity of stripping the uranium from each stage of the shakeout for analysis purposes. It was even necessary to change the analytical procedures slightly since the excess phosphate ions in the aqueous strips invalidated the analysis. Figure 25 shows a profile of this run. Better analytical stripping

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FIG. 22

PILOT PLANT SHAKEOUT

20 STAGE

23.20% TBP

FLOWS

ORGANIC—500 ML.

SLURRY—110 ML.

SCRUB—33 ML.

STRIP—325 ML.

ME UIN ORGANIC
AND AQUEOUS

ME HNO₃ IN AQUEOUS

ME HNO₃ IN ORGANIC

ORGANIC
0.8537M TBP

AQUEOUS
PHASE

ORGANIC
PHASE

600 500 400 300 200 100 0 0 100 200 300 400 500
TOTAL MILLEQUIVALENTS

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FIG. 23

MIXER SETTLER UNIT
22 STAGE - 23.20% TBP

FLOWS

ORGANIC — 500 ML.
SLURRY — 109 ML.
SCRUB — 36 ML.
STRIP — 336 ML.

AQUEOUS PHASE

ME U IN
ORGANIC AND AQUEOUS

ME HNO_3 IN ORGANIC

ME HNO_3 IN AQUEOUS

ORGANIC
0.8537 M TBP

ORGANIC
PHASE

359-11 KUFFEL & ESSER CO.
10 x 10 to the 12 inch, 5th floor, 5th floor.
NEW YORK, N.Y.

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FIG. 24

MIXER SETTLER UNIT
22 STAGE 23.7% TBP

FLOWS

ORGANIC — 500 ML

SLURRY — 144 ML

SCRUB — 32 ML

STRIP — 338 ML

- ME U IN ORGANIC AND AQUEOUS
- ME HNO₃ IN AQUEOUS
- ME HNO₃ IN ORGANIC
- ORGANIC 0.8526 M TBP

AQUEOUS
PHASE

ORGANIC
PHASE

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FIG. 25

PILOT PLANT SHAKEOUT

20 STAGE

22.30% TBP + 1.00% DBP

FLOWS

ORGANIC—500ML.

SLURRY—100ML.

SCRUB—33ML.

STRIP—500ML.

□ ME U IN ORGANIC
AND AQUEOUS

▨ ME HNO₃ IN ORGANIC

▩ ME HNO₃ IN AQUEOUS

▤ ORGANIC
0.8206M TBP

▥ ORGANIC
0.0507M DBP

AQUEOUS
PHASE

ORGANIC
PHASE

500 400 300 200 100 0 0 100 200 300 400 500
TOTAL MILL EQUIVALENTS

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possibly would have given a smoother profile. From the experience gained in this shakeout, it is thought that if any TBP degradation products are formed, they are removed in the solvent washing section or in the stripping section and go out with the OK liquor. Since the phosphates are very low in the UO_3 produced at Harshaw, either there is very little degradation of the TBP, or it is all washed out in the bicarbonate solvent washing section.

To summarize the results of the stripping efficiency tests it was found that stripping at $50^{\circ}C$ increased the uranium concentration of the OK liquor approximately ten percent. A small amount of nitric acid in the strip water improved the disengagement time of the two phases while not being detrimental to the distribution coefficient. In a five stage stripping section a ratio of 500 ml. solvent to approximately 375 ml. aqueous gave an optimum operating ratio with 23.2 percent TBP solvent for maximum uranium throughput.

Since other pilot plant data proved that more stripping stages were needed, a series of shakeouts were run to show the effect of TBP concentration on uranium throughput of the expanded system. From the resulting graph one can predict the various uranium concentrations to be expected when using a solvent of a certain TBP concentration. Another set of shakeouts were designed to show the overall efficiency of stripping sections with a different number of stages. A nine stage stripping section appears to be ideal since the OK liquor is nearly the maximum concentration possible. The extraction coefficient is the limiting

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factor.

The pilot plant shakeouts for both the fourteen stage system and the twenty stage system were compared to samples taken from the production systems. The distribution coefficient and profiles checked rather closely.

A shakeout was run with one percent dibutyl phosphate added to the solvent. This test was designed to show the effect of TBP degradation products upon the extraction system. The results showed that there must be a very small amount, if any, of these impurities in the solvent. If there were approximately one percent of the degradation products remaining in the solvent it would be disastrous from the extraction standpoint.

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III. EQUIPMENT DESIGN CHANGES

In order to determine disengagement times under actual flow rate conditions a mixer-settler unit, a duplicate of one stage in the production plant, was installed in the pilot plant. Since the largest total flow rates were to be used in the stripping section, the first tests were made with clean solvent and city water. This was to approximate the conditions at the end of the stripping section. One of the immediate problems was the question of being able to control the interface and gruel zone. At the high flow rates necessary, this became critical since the units were designed to operate with an aqueous underflow in the form of a jack leg. As the height of the jack leg controlled the interface in the tank, it was impossible to pick one height which would satisfy more than one unit.

Next, a moveable horseshoe which rotated between slip flanges was tried, by rotating the horseshoe between horizontal and vertical one was able to effectively move the interface up and down in the settling tank. The installation of this type of variable jack leg on all the plant units was considered too difficult and a weir box was suggested as a better means of controlling the jack leg height.

A weir box 18 inches long, 8 inches wide, and $15\frac{1}{2}$ inches deep, was placed on the up pipe from the settling tank underflow 49 inches from the floor. The drain line to the next stage was attached to the bottom of the weir box at the other end. Vertical channels were placed in the middle of the box to hold any height of weirs up to its top. One

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half inch and 3 inch stainless steel flat stock $\frac{1}{4}$ inch thick were cut to fit the channels and were used for weirs. With this arrangement the interface could be moved up or down with ease.

In the interest of cutting the disengagement time down with clean solvent and water, a small amount of nitric acid was added. The disengagement time was lowered by approximately 10 percent. This permitted an increase in the flow rates. The flow rates were considered maximum when the gruel zone nearly filled the settling tank. Another attempt to cut down the disengagement time and therefore increase flow rates involved the use of baffles. A comparison of maximum flow rates was made between using a set of baffles at the discharge line into the settling tank and using a swirl chamber at the end of the discharge line. Swirl chambers have been used in the production plant as standard equipment. The baffles were designed for an upright settling tank so that if they showed a large improvement over the swirl chamber, they could be installed in the present settling tanks. The baffles were placed four inches apart and about 15 degrees from horizontal. The baffle section was placed just above the aqueous layer in the Y tank. The openings of the discharge slits between the baffle plates were too large on the first run. These were narrowed up on subsequent runs but it was difficult to equalize the flows between the plates in the short time available for this demonstration. The baffles apparently improved the disengagement time somewhat. Other factors were involved which made numerical analysis of the improvement impossible. The improvement appeared to be approxi-

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mately 15 percent. More study is required before definite recommendations can be made.

The results of the pilot plant studies showed that nine stripping stages would be necessary. Since space limitations would allow five four-foot tanks and four five-foot tanks, these were added to the production plant. From the data available at the time the physical layout of the stripping section, it appeared that the longest disengagement time would be at the tail end of the stripping section. This placed the five-foot tanks at the four end stages of strippings. After the unit was in operation for some time it was found that the longer disengagement times were in the front end of the stripping section where the specific gravities were highest. Even though the production plant was able to maintain the production goal specified, it was decided to make the minor piping changes and use the five-foot tanks as the first four stripping settlers. This enables an even higher throughput in the production plant.

The previous production plant had the enviable design that it could be shut down by simply pushing a stop button and remain down for considerable time without any further changes. Then it could be started up by pushing three buttons in succession at the panel board.

Since the installation of the larger settling tanks, the mixer tanks will not hold the overflow from these tanks when the unit is shut down. Rubber plugs have to be placed in each of the weir boxes when the unit is shut down for more than a few minutes. Aside from this minor

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inconvenience the unit can be shut down and started up with ease. The important factor is that the equilibrium between aqueous and organic phases is not lost by shut downs.

It was decided to place an extra settling tank between the extraction section and the scrubbing section and one between the scrubbing section and the stripping section to remove entrained aqueous phase from the solvent.

The OK liquor lines were changes so that the OK liquor was run through a settling tank so that entrained solvent would be continually skimmed off and returned to the last scrubbing stage.

The solvent wash was changed from one to two stages counter-current. The aqueous phase is approximately a one half percent solution of sodium bicarbonate unless a stronger solution is called for by the fact that more metal is left in the stripped solvent. The amount of NaHCO_3 solution used in this section is regulated by a controlling pH meter which continuously reads the pH of the aqueous phase leaving the first wash stage. A small supplementary water stream is added continuously so that at times when only a small amount of NaHCO_3 solution is called for, there is no danger of losing the aqueous heels in the settling tanks.

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IV. SOLVENT CLEANUP

The experience at Harshaw with the solvent in the extraction system indicated that a method for solvent cleanup should be devised. The present method is periodically to replace the solvent completely. The use of certain feed materials, which apparently contain emulsifying agents as impurities made solvent replacement uneconomical. The expansion of the extraction system more than doubled the volume of solvent used which made the solvent cleanup method a necessity.

The solvent in the extraction system is in a closed system with only a water wash stage to remove any impurities or degradation products that it might pick up. A preferable solvent cleaner would be one in which the apparatus could be placed in the solvent stream. Since the solvent flow was quite large the greater part of the stream could bypass the solvent cleanup apparatus which would work continually. This would keep the apparatus small and inexpensive and the operation relatively simple.

In an attempt to get a suitable method many solvent washes were tried. A difficult problem arose when one tried to set up a standard solvent as a goal which could be checked in some manner analytically. Freshly made up solvent was water white whereas it became amber colored as soon as it was used in the extraction system a few hours. The amber solvent's extraction efficiency was in no way impaired by the color change. This solvent might be usable for months without an appreciable color change and then possibly in a few days, the disengagement

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times could begin to increase while the solvent became a decided reddish dark amber color. An infra-red spectrogram of the above three solvents was quite complex with a large number of absorption bands, but the three solvents had identical absorption bands. An ultra-violet spectrophotometric graph and a visible spectrophotometric graph were run on these solvents. The UV graph did not show anything significant while in the visible range the transmission cut off, which was sharp, frequency varied from 389 millimicrons for fresh solvent to 530 millimicrons for the unusable solvent. A sample of fresh solvent which had been used only a short time gave a cut-off frequency of approximately 420 mu.. The cut off frequency was then used as a measure of solvent improvement with 420 mu. as the goal.

A series of cleanup tests were run on a used solvent with the following results:

<u>Clean-up Method</u>	<u>Transmission Cut-off</u>
(1) Original used solvent.	460 mu.
(2) Scrubbed with NaHCO_3	458 mu
(3) Scrubbed with NaOH , H_2O , and treated with Nuchar	442 mu
(4) Treated with Nuchar, scrubbed with NaOH , H_2O , and retreated with Nuchar	432 mu
(5) Treated with Nuchar, scrubbed with NaOH , H_2O , Nuchar, HNO_3 , & H_2O .	432 mu
(6) Scrubbed with NaOH , H_2O , HNO_3 , H_2O , & treated with Nuchar.	441 mu
(7) Scrubbed with NaOH , H_2O , HNO_3 , H_2O , & treated, passed thru anion exchange column Dowex #2.	432 mu

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Clean-up Method

Transmission Cut-off

(8) Original used solvent passed thru dowex #2

457 mu

From these tests it was seen that a treatment with activated carbon, Nuchar, with a caustic scrub and a water wash followed by a Nuchar treatment gave the best clean-up by our arbitrary analysis.

Another series of tests were run using a small commercial filter unit. Various combinations of Nuchar treatments with scrub of NaOH, Na₂SO₄, Na₃PO₄, H₂SO₄, HNO₃, and H₂O were tried. The best results came from a caustic scrub and water wash followed by a Nuchar treatment and dilute nitric acid wash.

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V. PRESS CAKE SOLVENT REMOVAL

It was found that the press cake from the recovery room, precipitated sodium diuranate, contained considerable solvent adsorbed to it which became nitrated when the press cake was dissolved in strong nitric acid preceeding the addition to the feed slurry. When this feed slurry reached the extraction system, the nitrated organics acted as weak emulsifying agents which in a short time built up in the solvent to the point that the solvent was unusable. The press cake from that time on was stockpiled until a means of removing the solvent was developed.

It has recently been found that the solvent could be effectively removed from the press cake by adding a Johns-Mansville celite to an acid solution of the press cake and filtering off the celite. The solvent was adsorbed by the celite. Since the material being filtered off was slimy, it plugged the filter in a short time. Demonstrations were then carried out which showed that if the filtration were carried out on a precoated rotary filter with a moving doctor knife, which removed the slimy material plus a thin slice of precoat the press cake solution was easily filtered. The filtrate showed no trace of solvent. It was found that if Johns Manville 's Hi-flow Super-cel or Celite 501 were used, the precoat could be washed free of uranium without any solvent being washed through. A nine hundred gallon batch of press cake solution was filtered free of solvent in the Pilot Plant and then transferred to the production department to be run through the extraction system. No difficulty was experienced with this batch of press cake solution.

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A recommendation was turned in to get the necessary equipment to process the stockpile of press cake and also to change the recovery room procedure so as not to have to manually handle press cake in the future.

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VI. SUMMARY

Shakeout tests were run using a five stage stripping section to obtain equilibrium concentrations from which an estimate of the maximum throughput of uranium could be made. These tests were designed to find the optimum stripping conditions. By maintaining the stripping operation at 50°C to 55°C and by using strip water acidified with nitric acid, the most satisfactory results were obtained.

Pilot plant flow rate tests indicated that more stripping stages would be necessary in order to reach the proposed production output. A nine stage stripping section was found to be necessary in order to obtain the maximum concentration of uranium in the OK liquor. Another series of shakeouts were run to study the effect of variations in the solvent tributyl phosphate concentration as related to uranium concentration in the OK liquor. The data obtained shows clearly that the uranium equilibrium concentrations vary as a function of the percent tributyl phosphate concentration in the solvent.

One shakeout was run to check the effects of degradation products of the tributyl phosphate on the extraction of uranium. A solvent spiked with dibutyl phosphate was used. Because the presence of even slight amounts of dibutyl phosphate seemed to promote emulsification, the data obtained was not considered reliable. A method for reconditioning of the used solvent was proposed, together with a method for testing the suitability of the solvent after any period of use.

A method of removing adsorbed solvent from sodium diuranate press

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cake was proposed. Several suggestions can be offered which may result in further improvements in the extraction process.

1. A more detailed study of the effects of degradation products in the solvent and the means of removing them is needed.
2. The design and positioning of baffles in the solvent settler tanks requires more pilot plant investigation, should higher flow rates be necessary.
3. Modification of the existing extraction system may be required if it becomes necessary to use feed materials having high percentages of nitric acid insolubles. A method for the agitation of the acid insolubles in the settler tanks of the extraction section seems indicated.

REFERENCES

1. NYO-1456
2. ORNL-1367
3. FMPC-66
4. C. J. Rodden, "Analytical Chemistry of the Manhattan Project," p.74, McGraw-Hill Book Co., Inc., New York, 1950.

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APPENDIX A

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APPENDIX A - TBP ANALYSIS

The method of analyzing the solvent for TBP at Harshaw was not too satisfactory. The method developed at National Lead Co. (3.) appeared to require too precise an analysis to obtain the accuracy necessary for plant control. Since the electrodes of a pH meter often give erratic results when used with TBP solvent, some work was carried out to determine a pH indicator which would give a sharp and good color change at the equivalence point.

To increase the accuracy of the method without any essential changes, the volumes of solvent and 8 N nitric acid were increased six fold and the aliquot of solvent used was increased tenfold.

Upon investigation of the possible indicator for use at this end point Bromthymol Blue, Cresol Red and Phenolphthalein appeared to be the better ones. In actual use phenolphthalein gave a poor color change and the results were slightly high. The Cresol Red had a fairly good color change and was quite sharp. By far the best of the three indicated was Bromthymol Blue which gave a good color change and was sharp. The following data show the comparison between the different indicators and a pH meter. The same solvent was used in each case and the TBP factor used in each case was the one determined using a pH meter.

Percentage TBP From End Points Using

Sample #	pH Meter	Phenolphthalein	Bromthymol Blue	Cresol Red
1	21.94%	22.02		

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Percentage TBP From End Points Using

Sample #	pH Meter	Phenolphthalein	Bromthymol Blue	Cresol Red
2	21.95%	22.04		
3	21.94	22.04		
4	21.92	22.03		
5	21.95		21.98%	
6	21.94		21.95	
7	21.95		21.98	
8	21.93		21.95	
9	21.95			21.99
10	21.95			22.00
11	21.95			21.99
12	21.95			22.00
Average	21.94%	22.03%	21.97%	21.99%

The TBP factor is determined by making analyses of standard TBP solvents over the range for which the method is to be used. The factor is nearly constant for concentration range of at least five per-cent. The factor determined by two analysts using Bromthymol Blue is as follows:

Analyst	Std. Solvent 20.13% TBP	Std. Solvent 22.45% TBP
A	27.70% 27.64	27.64 27.64
B	27.61 27.57 27.60	27.60 27.55 27.53
Average factor	27.60	

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To test the reliability of the method five analysts made a duplicate analysis of the same solvent. Results as follows:

Analyst	% TBP	Analyst	% TBP
A	22.05% 22.04	D	22.05 22.04
B	22.05 22.09	E	22.04 22.04
C	22.02 22.07	Mean Average	22.04 22.05

As a result of the preceding data a method of analyzing solvent for TBP concentration was written up to be used in the analytical laboratory.

It is as follows:

REAGENTS

8 N HNO_3 - Dilute equal volume of conc. C.P. HNO_3 with distilled water accurately.

1.000 N NaOH

Brom Thymol Blue Indicator - Dissolve 0.100 GM of Brom Thymol Blue (Dibromothymolsulfonphthalein) in 50 ml of 90% alcohol, and dilute to 100 ml with distilled H_2O .

PROCEDURE

The solvent to be analyzed should be uranium free; if not, scrub the solvent with equal volumes of 1% NaHCO_3 until spot test shows no uranium present.

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1. With a graduate, add 150 ml of the solvent into a 1000 ml separatory funnel.
2. With a graduate add 300 ml. of 8 N HNO_3
3. Shake the funnel vigorously for 3 min. (clock)
4. Allow the layers to separate, and discard the aqueous layer.
5. Allow the solvent to sit for 5 min., and discard any more aqueous that may have settled out.
6. Into two 600 ml. beakers which contain 300 ml. of distilled water each, pipette 50.00 ml. portions of the sample. (Let pipette drain $\frac{1}{2}$ min. by clock).
7. Add 10 drops of Bromthymol Blue Indicator.
8. Agitate with electric stirrer until mixture is homogeneous. (About $\frac{1}{2}$ min. with fast agitation by clock).
9. Continuing this agitation, titrate with 1 N NaOH to a color change (Lime colored if used solvent, light blue if fresh) This end point should persist for two minutes (by clock) with the same agitation.
10. Record titration.
11. To calculate the amount of HNO_3 in the solvent, and from this percentage of TBP use the following method.

$$\frac{\text{Titration X Normality of NaOH X ME HNO}_3 \text{ X 1000 X Factor}}{\text{Equive. HNO}_3 \text{ X 50 (sample vol.)}} = \% \text{ TBP}$$

EXAMPLE

$$\frac{36.26 \text{ X } 1.1019 \text{ X } 0.06302 \text{ X } 1000 \text{ X } 27.52}{63.02 \text{ X } 50} = 21.99\% \text{ TBP}$$

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Reduced Version of Calculation

$$\frac{36.26 \times 1.1019 \times 27.52}{50} = 21.99\% \text{ TBP}$$

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A method of analyzing feed slurries for grams per liter of nitric acid and uranium, which was developed by Harshaw research and development group in 1951 for production personnel to use to control the feed slurry strength, was restudied and refined in order that the pilot plant group could get a rough analysis of a sample in the matter of a few minutes. The method is simply the titration of a sample with 1 N NaOH to a pH of 2.5 for nitric acid and titration of pH 2.5 to pH 6.0 for uranium. The theoretical factor for uranium is 2 NaOH- 1 U but due to occlusion and other effects a tactical factor (4) of 2.22 NaOH- 1 U should be used.

It was found that even with the tactical factor, analyses of solutions with low uranium concentrations gave high results and solutions with very high uranium concentrations gave results slightly low. Taking these limitations into consideration, this method of analysis is helpful in extraction equilibrium work since one can follow the concentrations of uranium in the different stages of operation while the extraction is going on due to the short time necessary for the analysis. By stripping the uranium from a known volume of solvent and titrating the aqueous strip, one also gets the uranium concentration in the solvent.

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APPENDIX B

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TABLE 1. PILOT PLANT SHAKEOUT - 14 STAGE - 23.20% TBP

Using a Distilled Water @ 25° C. Strip

Stage No.	Aqueous					Organic				
	Sp. Gr. @ 22°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 22°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow
1	1.040	34.77	60.69	0.02	0.02	0.848	6.14	48.70	0.02	0.10
2	1.055	62.19	108.55	0.09	0.09	0.852	11.30	89.65	0.08	0.40
3	1.080	110.87	193.51	0.29	0.26	0.854	20.12	159.60	0.50	2.10
4	1.095	133.74	233.43	0.30	0.28	0.865	22.28	176.75	7.48	31.40
5	1.275	151.40	264.26	123.80	114.43	0.962	10.78	85.50	86.24	362.35
6	1.530	98.57	172.05	309.0	285.63	0.970	3.64	28.85	91.20	383.15
7	1.345	13.24	6.93	236.0	65.44	0.964	0.0	0.0	90.10	378.55
8	1.275	4.41	2.31	182.0	50.47	0.962	0.0	0.0	87.32	366.85
9	1.195	0.0	0.0	124.0	34.39	0.947	0.0	0.0	77.56	325.85
10	1.116	0.0	0.0	85.0	285.68	0.926	0.0	0.0	59.08	248.20
11	1.084	0.0	0.0	63.0	211.76	0.907	0.0	0.0	47.20	198.30
12	1.063	0.0	0.0	47.0	157.96	0.890	0.0	0.0	32.30	135.70
13	1.048	0.0	0.0	36.0	121.00	0.873	0.0	0.0	21.92	92.10
14	1.021	0.0	0.0	17.0	57.12	0.852	0.0	0.0	5.32	22.35

Flows: Organic - 500 ml; Slurry - 110 ml; Scrub - 33 ml; Strip - 400 ml

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TABLE 2. PILOT PLANT SHAKEOUT - 23.20% TBP

5 Stage Strip (Distilled Water @ 50°C)

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow
10	1.122	1.62	10.28	95.10	319.64	0.913	0	0	51.24	215.25
11	1.078	0.35	2.20	62.20	209.04	0.896.	0	0	33.70	141.55
12	1.055	0	0	44.00	147.88	0.876	0	0	22.14	93.00
13	1.032	0	0	27.30	91.76	0.855	0	0	7.96	33.40
14	1.006	0	0	9.00	30.24	0.841	0	0	0.62	2.60

Flows: Organic - 500 ml; Strip - 400 ml

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TABLE 3. PILOT PLANT SHAKEOUT - 23.20% TBP

5 Stage Strip (Distilled Water + 0.2 gms. HNO_3 /l. @ 50°C)

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow
10	1.134	1.55	9.80	101.40	340.84	0.916	0.66	5.20	54.65	229.60
11	1.092	0.64	4.04	70.70	237.64	0.897	0	0	39.22	164.75
12	1.068	0.15	0.92	52.70	177.12	0.882	0	0	26.36	110.75
13	1.041	0	0	34.40	115.60	0.861	0	0	11.69	49.10
14	1.016	0	0	15.30	51.40	0.847	0	0	0.24	1.00

Flows: Organic - 500 ml; Strip - 400 ml

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TABLE 4. PILOT PLANT SHAKEOUT - 23.20% TBP

5 Stage Strip (Distilled Water + 0.2 gms. HNO_3 /l. @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow
10	1.120	3.29	23.49	84.80	320.67	0.907	0	0	47.36	198.95
11	1.060	1.29	9.18	50.60	191.34	0.881	0	0	26.40	110.90
12	1.042	1.11	7.92	30.10	113.81	0.859	0	0	10.80	45.35
13	1.016	0.29	2.07	11.40	43.07	0.847	0	0	1.40	5.85
14	1.001	0.21	1.49	1.09	4.10	0.846	0	0	0.16	0.65

Flows: Organic - 500 ml; Strip - 450 ml

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TABLE 5. PILOT PLANT SHAKEOUT - 23.20% TBP
5 Stage Strip (Distilled Water + 0.2 gms. HNO₃/l. @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow
10	1.139	0.86	4.76	106.40	312.94	0.921	0	0	59.00	250.40
11	1.099	0.95	5.25	76.00	223.51	0.906	0	0	44.70	187.80
12	1.076	0.84	4.66	58.50	172.03	0.887	0	0	33.20	139.45
13	1.054	0.42	2.31	43.20	127.05	0.868	0	0	18.17	76.30
14	1.025	0	0	21.60	63.53	0.851	0	0	2.50	10.50

Flows: Organic - 500 ml; Strip - 350 ml

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TABLE 6. PILOT PLANT SHAKEOUT - 23.20% TBP

5 Stage Strip (Distilled Water + 0.2 gms. HNO_3 /l. @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g/l.	ME. U x Flow
10	1.144	3.33	15.84	109.30	275.52	0.924	0.53	4.20	59.86	251.50
11	1.108	1.69	8.04	83.30	210.00	0.912	0.33	2.60	49.50	207.95
12	1.088	1.31	6.21	65.90	166.11	0.897	8	0	40.00	168.05
13	1.062	0.35	1.65	50.30	126.78	0.882	0	0	26.42	111.00
14	1.036	0.32	1.50	30.40	76.62	0.856	0	0	8.40	35.25

Flows: Organic - 500 ml; Strip - 300 ml

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TABLE 7.

STRIPPING COMPARISONS 23.20% TBP

Stage No.	350 cc		400 cc		450 cc		300 cc	
	U-Aq.	U-Org.	U-Aq.	U-Org.	U-Aq.	U-Org.	U-Aq.	U-Org.
10	106.40	59.60	101.40	54.65	84.80	47.36	109.30	59.86
11	76.00	44.70	70.70	39.22	50.60	26.40	83.30	49.50
12	58.50	33.20	52.70	26.36	30.10	10.80	65.90	40.00
13	43.20	18.17	34.40	11.69	11.40	1.40	50.30	26.42
14	21.60	2.50	15.30	0.24	1.09	0.16	30.40	8.40

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TABLE 8. PILOT PLANT SHAKEOUT - 17.82% TBP

Distilled Water Strip (0.2 gm. HNO_3 /l.) @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow
1	1.030	48.33	70.55	0.01	0.01	0.840	4.58	36.30	0.13	0.50
2	1.053	84.86	123.88	0.01	0.01	0.843	9.69	76.85	0.02	0.05
3	1.073	118.88	173.54	0.03	0.03	0.845	15.33	121.60	0.04	0.15
4	1.089	152.77	223.02	0.14	0.11	0.849	19.11	151.60	2.11	8.85
5	1.123	181.93	265.59	10.20	7.88	0.901	8.38	66.45	47.60	200.00
6	1.145	118.46	172.93	253.90	196.29	0.933	0.39	3.85	70.00	294.10
7	1.308	25.83	13.52	226.20	62.73	0.931	0	0	71.20	299.15
8	1.260	7.92	4.14	187.56	52.01	0.922	0	0	66.40	278.95
9	1.190	0.42	0.22	132.20	36.66	0.916	0	0	59.20	248.70
10	1.140	1.42	5.63	102.10	214.48	0.900	0	0	50.00	210.05
11	1.116	0.78	3.08	82.70	173.73	0.890	0	0	41.20	173.10
12	1.094	0.51	2.00	67.30	141.38	0.876	0	0	31.60	132.75
13	1.070	0.38	1.50	50.60	106.30	0.864	0	0	18.80	78.95
14	1.038	0	0	28.00	58.80	0.843	0	0	4.40	18.45

Flows: Organic - 500 ml; Slurry - 92 ml; Scrub - 33 ml; Strip - 250 ml

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TABLE 9. PILOT PLANT SHAKEOUT - 17.82% TBP

Distilled Water Strip (0.2 gm. HNO₃/l.) @ 50° C

Stage No.	Aqueous					Organic				
	Sp. Gr. @ 21° C	HNO ₃ g/l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21° C	HNO ₃ g/l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow
1	1.030	48.33	70.55	0.01	0.01	0.840	4.58	36.30	0.13	0.50
2	1.053	84.86	123.88	0.01	0.01	0.843	9.69	76.85	0.02	0.05
3	1.073	118.88	173.54	0.03	0.03	0.845	15.33	121.60	0.04	0.15
4	1.089	152.77	223.02	0.14	0.11	0.849	19.11	151.60	2.11	8.85
5	1.123	181.93	265.59	10.20	7.88	0.901	8.38	66.45	47.60	200.00
6	1.145	118.46	172.93	253.90	196.29	0.933	0.39	3.05	70.00	294.10
7	1.308	25.83	13.52	226.20	62.73	0.931	0	0	71.20	299.15
8	1.260	7.92	4.14	187.56	52.01	0.922	0	0	66.40	278.95
9	1.190	0.42	0.22	132.20	36.66	0.916	0	0	59.20	248.70
10	1.125	0.37	1.74	90.50	228.15	0.894	0	0	44.40	186.55
11	1.098	0.42	1.98	70.90	178.71	0.882	0	0	33.20	139.45
12	1.070	0.19	0.90	50.40	127.05	0.863	0	0	20.00	84.00
13	1.046	0	0	32.80	82.68	0.849	0	0	8.20	34.45
14	1.014	0	0	10.70	26.97	0.837	0	0	0.42	1.75

Flows: Organic - 500 ml; Slurry: - 92 ml; Scrub - 33 ml; Strip - 300 ml

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TABLE 10 PLANT MIXER SETTLER UNIT- 17.84% TBP 14 STAGE

Aqueous						Organic				
Stage No. 1	Sp. Gr. @ 21°C 2	HNO ₃ g/l. 3	ME HNO ₃ x Flow 4	U g/l. 5	ME. U. x Flow 6	Sp. Gr. @ 21°C 7	HNO ₃ g/l. 8	ME HNO ₃ x Flow 9	U g/l. 10	ME. U. x Flow 11
1	1.050	90.76	146.89	0	0	0.839	9.29	73.70	0	0
2	1.075	133.66	216.33	1.62	1.39	0.841	15.04	119.30	0	0
3	1.085	160.02	258.99	2.54	2.17	0.842	18.60	147.55	2.16	9.05
4	1.090	172.20	278.70	3.04	3.12	0.845	19.56	155.15	3.71	15.55
5	1.105	175.94	284.76	9.10	7.79	0.883	10.30	81.70	34.54	145.10
6	1.265	141.28	228.66	161.00	137.99	0.922	2.22	17.70	64.77	272.10
7	1.250	32.30	17.43	183.31	52.37	0.920	0	0	64.07	265.20
8	1.220	12.48	6.73	155.43	44.49	0.915	0	0	59.18	246.05
9	1.165	3.08	1.66	114.45	32.69	0.903	0	0	50.19	210.85
10	1.115	0.80	4.01	78.97	211.02	0.882	0	0	37.19	156.29
11	1.085	0.80	4.01	58.70	156.84	0.865	0	0	23.76	99.60
12	1.055	0.80	4.01	41.55	111.01	0.849	0	0	11.74	49.30
13	1.025	0.64	3.21	19.66	52.53	0.835	0	0	2.48	10.40
14	1.005	0	0	5.46	14.56	0.833	0	0	0.16	0.65

Flows: Organic - 500 ml; Slurry - 102 ml; Scrub - 34 ml; Strip - 318 ml

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TABLE 11. PILOT PLANT SHAKEOUT - 25.89% TBP

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow
1	2	3	4	5	6	7	8	9	10	11
1	1.035	56.11	93.48	0.01	0.01	0.855	8.89	70.50	0.08	0.30
2	1.060	101.10	168.44	0.01	0.01	0.860	18.92	150.10	0.02	0.10
3	1.100	164.71	274.43	0.01	0.01	0.865	31.55	250.30	0.07	0.25
4	1.135	231.24	385.28	0.05	0.04	0.871	41.89	332.35	2.03	8.50
5	1.170	296.65	494.26	2.60	2.29	0.922	29.39	233.15	43.60	183.15
6	1.340	254.15	423.44	143.00	126.18	0.983	17.92	142.15	76.80	322.65
7	1.700	115.27	192.06	441.40	389.47	0.992	0.67	5.30	104.00	436.95
8	1.395	30.41	15.92	280.10	77.67	0.988	0	0	101.50	426.45
9	1.340	12.49	6.54	244.20	67.72	0.984	0	0	99.00	415.95
10	1.280	2.78	1.46	203.10	56.32	0.981	0	0	96.50	405.45

Flows: Organic - 500 ml; Slurry - 105 ml; Scrub - 33 ml; Strip - 350 ml

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TABLE 11.

Aqueous					Organic					
1.	2.	3.	4.	5.	6.	7.	8.	9.	10	11
11	1.205	0	0	146.90	40.74	0.969	0	0	84.80	356.30
12	1.174	2.64	14.63	120.90	355.57	0.949	0	0	72.40	304.20
13	1.139	2.04	11.31	96.40	283.50	0.938	0	0	64.00	268.90
14	1.128	1.79	9.94	89.30	262.64	0.932	0	0	61.20	257.10
15	1.114	1.80	9.98	80.10	235.59	0.920	0	0	50.40	211.95
16	1.100	1.39	7.70	71.80	211.16	0.911	0	0	43.20	181.50
17	1.077	1.21	6.72	55.10	162.05	0.894	0	0	32.00	134.45
18	1.066	0.96	5.32	47.00	138.22	0.886	0	0	28.00	117.60
19	1.045	0.96	5.32	32.60	95.87	0.868	0	0	12.60	52.90
20	1.025	0.79	4.38	18.20	53.52	0.853	0	0	3.00	12.60

Flows: Organic - 500 ml; Slurry - 105 ml; Scrub - 33 ml; Strip - 350 ml

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TABLE 12 PILOT PLANT SHAKEOUT - 23.20% TBP
Distilled Water Strip (0.2 gms. HNO_3 /l) @ 50°C

Aqueous					Organic					
Stage No. 1	Sp. Gr. @ 21°C 2	HNO_3 g./l. 3	ME HNO_3 x Flow ³ 4	U g./l. 5	ME. U. x Flow 6	Sp. Gr. @ 21°C 7	HNO_3 g./l. 8	ME HNO_3 x Flow ³ 9	U g./l. 10	ME. U. x Flow 11
1	1.040	38.48	67.16	0.77	0.70	0.847	6.22	49.30	0.26	1.05
2	1.055	69.20	120.78	0.17	0.15	0.849	12.12	96.15	0.10	0.40
3	1.080	109.72	191.51	0.21	0.19	0.852	20.22	160.40	0.11	0.45
4	1.095	141.29	246.61	0.19	0.17	0.854	25.98	206.10	0.70	2.90
5	1.125	190.75	332.98	1.90	1.75	0.891	23.20	184.05	31.82	133.65
6	1.340	169.12	295.19	179.60	166.01	0.960	4.38	34.75	91.66	385.10
7	1.620	102.75	179.34	410.30	379.26	0.967	2.60	20.65	94.42	396.70
8	1.364	16.18	8.47	253.90	70.41	0.968	0.98	7.75	91.52	384.50
9	1.314	7.36	3.85	213.60	59.23	0.967	0.95	7.50	88.90	373.50
10	1.264	0.80	0.42	193.80	53.74	0.960	0.80	6.30	86.04	361.50

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TABLE 12

Aqueous						Organic				
1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.
11	1.204	0	0	145.80	40.43	0.949	0	0	77.76	326.70
12	1.154	3.20	17.75	111.40	327.64	0.927	0	0	61.00	256.30
13	1.128	2.31	12.81	92.30	271.46	0.917	0	0	52.88	222.15
14	1.110	1.19	6.58	79.40	233.52	0.906	0	0	46.32	194.60
15	1.096	1.13	6.27	70.10	206.15	0.895	0	0	33.80	142.00
16	1.077	0.81	4.48	56.20	165.27	0.884	0	0	27.70	116.35
17	1.058	0.39	2.14	42.90	126.18	0.868	0	0	17.28	72.60
18	1.038	0.33	1.82	28.20	82.92	0.857	0	0	8.74	36.70
19	1.015	0	0	10.70	31.47	0.846	0	0	2.25	9.40
20	1.003	0	0	2.20	6.44	0.846	0	0	0.12	0.50

Flows: Organic - 500 ml; Slurry - 110 ml; Scrub - 303 ml; Strip - 350 ml

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TABLE 13. PILOT PLANT SHAKEOUT - 20.44% TBP

Distilled Water Strip (0.1 gm. HNO₃/l) @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow
1	2	3	4	5	6	7	8	9	10	11
1	1.045	45.97	72.94	0	0	0.845	5.34	42.35	0.03	0.15
2	1.065	76.25	120.99	0.01	0.01	0.848	11.40	90.40	0.02	0.10
3	1.085	114.02	180.92	0.02	0.02	0.852	18.02	142.95	0.02	0.10
4	1.105	155.55	246.82	0.03	0.03	0.856	24.06	190.85	0.57	2.35
5	1.160	187.91	298.17	2.10	1.76	0.888	18.96	150.40	27.92	117.30
6	1.270	174.02	276.13	117.40	98.65	0.949	2.96	23.45	75.02	315.20
7	1.585	81.24	128.91	381.90	320.92	0.957	0.79	6.25	83.45	350.60
8	1.355	15.14	7.93	258.80	71.77	0.953	0	0	83.82	352.15
9	1.305	2.08	1.09	218.30	60.54	0.949	0	0	80.23	337.10
10	1.255	0	0	189.20	52.47	0.945	0	0	78.12	328.20

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TABLE 13.

Aqueous				Organic						
1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.
11	1.190	0	0	136.90	37.76	0.934	0	0	68.32	287.05
12	1.158	1.76	9.07	113.10	308.88	0.916	0	0	55.60	233.60
13	1.126	1.53	7.87	89.50	244.43	0.906	0	0	45.60	191.50
14	1.110	1.19	6.11	78.40	214.11	0.896	0	0	40.40	169.70
15	1.098	0.79	4.06	68.50	187.07	0.887	0	0	34.40	144.50
16	1.084	0.53	2.73	59.60	162.76	0.873	0	0	24.00	100.80
17	1.058	0.32	1.63	41.50	113.33	0.864	0	0	16.40	68.90
18	1.038	0.07	0.36	27.30	74.56	0.852	0	0	7.00	29.40
19	1.018	0	0	13.10	35.75	0.844	0	0	1.19	5.00
20	1.001	0	0	7.10	19.37	0.842	0	0	0.43	1.80

Flows: Organic - 500 ml; Slurry - 100 ml; Scrub - 33 ml; Strip - 325 ml

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TABLE 14 PILOT PLANT SHAKEOUT - 17.82% TBP

Distilled Water Strip (0.1 gm HNO₃/l.) @ 50°C

Aqueous						Organic				
Stage No. 1	Sp. Gr. @ 21°C 2	HNO ₃ g./l. 3	ME HNO ₃ x Flow 4	U g./l. 5	ME. U. x Flow 6	Sp. Gr. @ 21°C 7	HNO ₃ g./l. 8	ME HNO ₃ x Flow 9	U g./l. 10	ME. U. x Flow 11
1	1.025	49.72	71.84	0.003	0.009	0.839	4.58	36.30	0.05	0.20
2	1.050	81.52	117.71	0.007	0.009	0.841	9.49	75.25	0.03	0.10
3	1.070	107.22	154.82	0.02	0.009	0.843	13.32	105.65	0.06	0.25
4	1.085	138.46	199.93	0.09	0.06	0.846	17.57	139.40	1.28	5.35
5	1.100	156.66	226.21	3.80	2.90	0.880	11.77	93.35	29.60	124.35
6	1.312	125.83	181.69	168.10	128.55	0.930	1.28	10.15	69.20	290.75
7	1.486	83.74	120.91	311.60	238.27	0.934	0.39	3.05	70.00	294.10
8	1.335	11.39	5.96	245.30	68.02	0.929	0	0	68.40	287.35
9	1.285	1.94	1.01	202.80	56.23	0.926	0	0	66.40	278.95
10	1.245	0	0	179.90	49.88	0.923	0	0	66.40	278.95

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TABLE 14.

Aqueous					Organic					
1	2	3	4	5	6	7	8	9	10	11
11	1.175	0	0	122.00	33.83	0.913	0	0	52.00	218.45
12	1.148	6.11	31.49	106.90	291.95	0.895	0	0	42.40	178.15
13	1.114	4.17	21.48	84.10	229.67	0.883	0	0	35.40	147.85
14	1.096	1.94	9.98	68.20	186.26	0.875	0	0	29.60	124.35
15	1.082	0	0	58.90	160.84	0.864	0	0	22.00	92.40
16	1.058	0	0	41.20	112.52	0.852	0	0	11.20	47.05
17	1.030	0	0	21.20	57.88	0.840	0	0	1.60	6.70
18	1.008	0	0	6.50	17.75	0.834	0	0	0.24	1.00
19	1.002	0	0	0.54	1.46	0.836	0	0	0.02	0.05
20	1.000	0	0	0.03	0.07	0.836	0	0	0.01	0.05

Flows: Organic - 500 ml; Slurry - 91 ml; Scrub - 33 ml; Strip - 325 ml

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TABLE 15. PILOT PLANT SHAKEOUT - 14.93% TBP
Distilled Water Strip (0.1 gm. HNO₃/l.) @ 50° C

Aqueous						Organic				
Stage No. 1	Sp. Gr. @ 21°C 2	HNO ₃ g./l. 3	ME HNO ₃ x Flow 4	U g./l. 5	ME. U x Flow 6	Sp. Gr. @ 21°C 7	HNO ₃ g./l. 8	ME HNO ₃ x Flow 9	U g./l. 10	ME. U x Flow 11
1	1.030	48.19	76.46	0.01	0.01	0.834	3.47	1.75	0.13	0.55
2	1.045	73.88	117.23	0.03	0.03	0.835	6.94	55.05	0.02	0.10
3	1.060	96.38	152.93	0.02	0.02	0.837	9.80	77.75	0.14	0.60
4	1.067	110.13	174.75	0.20	0.17	0.840	10.67	84.65	2.21	9.25
5	1.095	117.35	186.21	19.00	15.96	0.885	2.56	20.30	38.40	161.30
6	1.275	90.69	143.90	165.10	138.73	0.907	0	0	56.00	235.25
7	1.430	65.27	103.57	282.20	237.14	0.913	0	0	62.00	260.50
8	1.320	9.58	5.02	223.60	62.00	0.910	0	0	55.79	234.45
9	1.275	0	0	197.60	54.80	0.907	0	0	53.43	224.35
10	1.230	0	0	164.30	45.56	0.903	0	0	50.31	211.30

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TABLE 15.

Aqueous					Organic					
1	2	3	4	5	6	7	8	9	10	11
11	1.170	0	0	113.50	31.47	0.891	0	0	43.78	184.00
12	1.152	1.89	7.48	107.30	225.40	0.879	0	0	31.20	131.05
13	1.120	1.28	5.08	84.20	176.88	0.873	0	0	30.40	127.70
14	1.104	1.07	4.23	70.00	147.05	0.868	0	0	26.80	112.60
15	1.092	1.07	4.23	63.50	133.40	0.862	0	0	20.80	87.35
16	1.070	0.99	3.93	49.20	103.35	0.850	0	0	12.00	50.40
17	1.047	0.98	3.88	32.50	68.28	0.843	0	0	7.20	30.25
18	1.022	0.68	2.68	15.20	31.93	0.835	0	0	1.58	6.65
19	1.010	0.59	2.33	5.80	12.18	0.833	0	0	0.21	0.90
20	1.003	0.56	2.20	0.52	1.10	0.832	0	0	0.13	0.55

Flows: Organic - 500 ml; Slurry - 100 ml; Scrub - 33 ml; Strip - 250 ml

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TABLE 16 URANIUM CONCENTRATION versus PERCENT TBP

% TBP	Saturated Solvent	Scrubbed Solvent	O.K. Liquor
14.93	62.00	43.78	107.30
17.82	70.00	52.00	106.90
20.44	83.45	68.32	113.10
23.20	94.42	77.76	116.10
25.89	104.00	84.80	120.90

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TABLE 17 PILOT PLANT SHAKEOUT - 23.20% TBP
Distilled Water Strip (0.2 gm. HNO_3 /l.) @ 50° C

Aqueous					Organic					
Stage No. 1	Sp. Gr. @ 21° C 2	HNO_3 g/l. 3	ME HNO_3 x Flow 4	U g./l. 5	ME. U x Flow 6	Sp. Gr. @ 21° C 7	HNO_3 g./l. 8	ME HNO_3 x Flow 9	U g/l. 10	ME. U. x Flow 11
1	1.040	38.48	67.16	0.77	0.70	0.847	6.22	49.30	0.26	1.05
2	1.055	69.20	120.78	0.17	0.15	0.849	12.12	96.15	0.10	0.40
3	1.080	109.72	191.51	0.21	0.19	0.852	20.22	160.40	0.11	0.45
4	1.095	141.29	246.61	0.19	0.17	0.854	25.98	206.10	0.70	2.90
5	1.125	190.75	332.98	1.90	1.75	0.891	23.20	184.05	31.82	133.65
6	1.340	169.12	295.19	179.60	166.01	0.960	4.38	34.75	91.66	385.10
7	1.620	102.75	179.34	410.30	379.26	0.967	2.60	20.65	94.42	396.70
8	1.364	16.18	8.47	253.90	70.41	0.968	0.98	7.75	91.52	384.50

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TABLE 17.

		Aqueous				Organic				
1										
9	1.314	7.36	3.85	213.60	59.23	0.967	0.95	7.50	88.90	373.50
10	1.264	0.80	0.42	193.80	53.74	0.960	0.80	6.30	86.04	361.50
11	1.204	0	0	145.80	40.43	0.949	0	0	77.76	326.70
12	1.142	0.77	4.27	104.50	307.34	0.924	0	0	58.52	245.85
13	1.106	1.05	5.81	75.10	220.85	0.911	0	0	47.30	198.70
14	1.087	0.64	3.54	62.60	184.10	0.899	0	0	38.70	162.60
15	1.070	0.35	1.93	52.10	153.23	0.885	0	0	26.96	113.25
16	1.052	0	0	37.40	109.97	0.867	0	0	16.00	67.20
17	1.025	0	0	18.90	55.58	0.849	0	0	1.90	7.95

Flows: Organic - 500 ml; Slurry - 110 ml; Scrub - 33 ml; Strip - 350 ml

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TABLE 18 PILOT PLANT SHAKEOUT - 23.20% TBP
7 STAGE STRIP (DISTILLED WATER + 0.2 gms. HNO₃/l.) @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U X Flow
12	1.148	2.18	12.08	108.30	318.50	0.923	0	0	58.28	244.85
13	1.114	1.77	9.80	83.00	244.09	0.911	0	0	49.76	209.10
14	1.094	1.02	5.64	68.40	201.15	0.897	0	0	41.86	175.85
15	1.082	0.47	2.59	59.20	174.09	0.887	0	0	32.26	135.50
16	1.059	0.43	2.38	43.10	126.74	0.873	0	0	22.04	92.60
17	1.043	0	0	31.70	93.21	0.860	0	0	11.02	46.30
18	1.015	0	0	11.60	34.09	0.846	0	0	1.12	4.70

Flows: Organic - 500 ml; Strip - 350 ml

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TABLE 19 PILOT PLANT SHAKEOUT - 23.20% TBP
8 Stage Strip (Distilled Water + 0.2 gms. HNO₃/l.) @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO ₃ g./l.	ME HNO ₃ x Flow	U g./l.	ME. U. x Flow	Sp. Gr. @ 21°C	HNO ₃ g/l. ³	ME HNO ₃ x Flow	U g./l.	ME. U. x Flow
12	1.153	2.10	11.66	109.30	321.44	0.929	0	0	58.88	247.35
13	1.123	1.13	6.27	88.90	261.45	0.918	0	0	51.98	218.40
14	1.103	0.77	4.27	76.40	224.70	0.908	0	0	45.48	191.05
15	1.091	0.74	4.10	66.40	195.27	0.893	0	0	34.30	144.10
16	1.068	0.39	2.14	49.80	146.44	0.882	0	0	28.16	118.30
17	1.054	0.05	0.25	38.40	112.91	0.871	0	0	19.24	80.80
18	1.036	0	0	26.60	78.23	0.857	0	0	7.68	32.25
19	1.016	0	0	11.30	33.22	0.847	0	0	1.12	4.70

Flows: Organic - 500 ml; Strip - 350 ml

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TABLE 20 PILOT PLANT SHAKEOUT - 23.20% TBP
9 Stage Strip (Distilled Water + 0.2 gm. HNO_3 /l.) @ 50°C

Aqueous						Organic				
Stage No.	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	ME. U x Flow	Sp. Gr. @ 21°C	HNO_3 g./l.	ME HNO_3 x Flow	U g./l.	Me. U x Flow
12	1.154	3.20	17.75	111.40	327.64	0.927	0	0	61.00	256.30
13	1.128	2.31	12.81	92.30	271.46	0.917	0	0	52.88	222.15
14	1.110	1.19	6.58	79.40	233.52	0.906	0	0	46.32	194.60
15	1.096	1.13	6.27	70.10	206.15	0.895	0	0	33.80	142.00
16	1.077	0.81	4.48	56.20	165.27	0.884	0	0	27.70	116.35
17	1.058	0.39	2.14	42.90	126.18	0.868	0	0	17.28	72.60
18	1.038	0.33	1.82	28.20	82.92	0.857	0	0	8.74	36.70
19	1.015	0	0	10.70	31.47	0.846	0	0	2.24	9.40
20	1.003	0	0	2.20	6.44	0.846	0	0	0.12	0.50

Flows: Organic - 500 ml; Strip - 350 ml

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TABLE 22 PILOT PLANT SHAKEOUT - 20 STAGE- 23.20% TBP

Distilled Water Strip (0.2 gm. HNO_3 /l.) @ 50° C

Aqueous						Organic				
Stage No. 1	Sp. Gr. @ 21° C 2	HNO_3 g./l. 3	ME HNO_3 x Flow 4	U g./l. 5	ME. U x Flow 6	Sp. Gr. @ 21° C 7	HNO_3 g./l. 8	ME HNO_3 X Flow 9	U g./l. 10	ME. U X Flow 11
1	1.040	38.48	67.16	0.77	0.70	0.847	6.22	49.30	0.26	1.05
2	1.055	69.20	120.78	0.17	0.15	0.849	12.12	96.15	0.10	0.40
3	1.080	109.72	191.51	0.21	0.19	0.852	20.22	160.40	0.11	0.45
4	1.095	141.29	246.61	0.19	0.17	0.854	25.98	206.10	0.70	2.90
5	1.125	190.75	332.98	1.90	1.75	0.891	23.20	184.05	31.82	133.65
6	1.340	169.12	295.19	179.60	166.01	0.960	4.38	34.75	91.66	385.10
7	1.620	102.75	179.34	410.30	379.26	0.967	2.60	20.65	94.42	396.70
8	1.364	16.18	8.47	253.90	70.41	0.968	0.98	7.75	91.52	384.50
9	1.314	7.36	3.85	213.60	59.23	0.967	0.95	7.50	88.90	373.50
10	1.264	0.80	0.42	193.80	53.74	0.960	0.80	6.30	86.04	361.50

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TABLE 22.

1	Aqueous					Organic				
11	1.204	0	0	145.80	40.43	0.949	0	0	77.76	326.70
12	1.160	3.10	15.95	116.10	317.07	0.930	0	0	62.38	262.10
13	1.133	2.33	11.99	95.20	260.00	0.920	0	0	53.50	224.75
14	1.116	1.73	8.91	83.70	228.57	0.912	0	0	50.40	211.75
15	1.105	1.40	7.22	73.30	200.17	0.902	0	0	43.92	184.50
16	1.086	0.94	4.84	62.10	169.59	0.892	0	0	34.20	143.65
17	1.071	0.85	4.36	50.30	137.35	0.880	0	0	24.08	101.15
18	1.050	0.36	1.85	34.40	93.93	0.865	0	0	14.94	62.75
19	1.032	0.19	0.98	23.30	63.60	0.853	0	0	5.50	23.10
20	1.008	0	0	6.90	18.82	0.846	0	0	0.38	1.55

Flows: Organic - 500 ml; Slurry - 110 ml; Scrub - 33 ml; Strip - 325 ml

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TABLE 23 PLANT MIXER SETTLER UNIT - 20 STAGE - 23.30% TBP

Aqueous						Organic				
Stage No.	Sp. Gr. @ 24°C	HNO ₃ g./l.	ME HNO ₃ x Flow ³	U g./l.	ME. U. x Flow	Sp. Gr. @ 24°C	HNO ₃ g./l.	ME HNO ₃ x Flow ³	U g./l.	ME.U x Flow
1	1.040	72.60	125.57	0	0	0.850	11.45	90.80	0	0
2	1.075	133.80	231.40	2.48	2.27	0.855	22.40	177.70	0	0
3	1.100	180.10	311.50	3.78	3.45	0.858	29.20	231.80	0	0
4	1.115	210.86	364.70	5.05	4.62	0.863	32.74	259.75	5.10	21.40
5	1.145	224.14	387.66	20.37	18.65	0.939	12.83	101.75	75.49	317.50
6	1.470	125.62	217.27	300.70	275.42	0.969	2.30	18.55	91.79	385.65
7	1.600	81.30	140.61	416.96	381.91	0.971	2.34	18.20	91.90	386.10
8	1.335	17.12	9.78	245.80	74.36	0.965	0.37	2.90	89.76	375.00
9	1.280	7.74	4.42	205.92	62.29	0.961	0	0	87.47	367.50
10	1.235	6.14	3.51	173.61	52.52	0.956	0	0	81.86	343.90

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TABLE 23.

Aqueous					Organic					
1	2	3	4	5	6	7	8	9	10	11
11	1.180	2.94	1.68	122.68	37.11	0.943	0	0	74.31	312.20
12	1.145	1.98	10.55	99.55	281.06	0.930	0	0	63.73	267.75
13	1.125	1.17	6.22	86.76	244.94	0.919	0	0	53.49	224.70
14	1.105	0.51	2.69	73.11	206.40	0.908	0	0	44.63	187.50
15	1.090	0.24	1.28	64.46	181.98	0.893	0	0	35.78	150.30
16	1.070	0	0	51.44	145.22	0.880	0	0	24.90	104.60
17	1.060	0	0	39.60	111.78	0.867	0	0	14.25	59.85
18	1.035	0	0	23.99	67.70	0.852	0	0	5.19	21.80
19	1.005	0	0	7.92	22.34	0.844	0	0	0.34	1.40
20	1.000	0	0	1.50	4.23	0.843	0	0	0.11	0.45

Flows: Organic - 500 ml; Slurry - 109 ml; Scrub - 36 ml; Strip - 336 ml

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TABLE 24 PLANT MIXER SETTLER UNIT - 23.17% TBP - 20 STAGE

Aqueous						Organic				
Stage No. 1	Sp. Gr. @ 21°C 2	HNO ₃ g./l. 3	ME HNO ₃ x Flow 4	U g./l. 5	ME. U x Flow 6	Sp. Gr. @ 21°C 7	HNO ₃ g./l. 8	ME HNO ₃ x Flow 9	U g./l. 10	ME. U x Flow 11
1	1.049	82.63	188.80	0.002	0.01	0.858	12.49	99.05	0.01	0.05
2	1.082	147.49	337.00	0.003	0.01	0.863	24.53	194.60	0.01	0.05
3	1.108	197.90	452.19	0.004	0.03	0.866	32.41	257.10	0.05	0.20
4	1.125	228.59	522.32	0.15	0.19	0.873	34.89	276.80	4.20	17.60
5	1.155	237.90	543.59	15.50	18.75	0.950	11.25	89.25	58.80	247.05
6	1.473	134.29	306.85	296.21	358.43	0.977	0.78	6.15	92.29	387.75
7	1.626	94.74	225.62	422.44	511.19	0.980	0	0	94.37	396.50
8	1.364	16.25	8.24	251.10	67.52	0.976	0	0	89.55	376.25
9	1.303	3.33	1.69	220.10	59.18	0.971	0	0	86.43	363.15
10	1.248	0	0	180.80	48.62	0.965	0	0	81.13	340.85

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TABLE 24

Aqueous			Organic							
1	2	3	4	5	6	7	8	9	10	11
11	1.173	0	0	127.00	34.15	0.951	0	0	71.29	299.50
12	1.150	0	0	107.56	305.48	0.929	0	0	54.61	229.45
13	1.125	0	0	83.21	236.33	0.917	0	0	45.86	192.65
14	1.100	0	0	70.92	201.41	0.906	0	0	38.30	160.90
15	1.085	0	0	59.57	169.17	0.895	0	0	30.50	128.15
16	1.070	0	0	47.99	136.32	0.882	0	0	21.42	90.00
17	1.060	0	0	34.04	96.67	0.868	0	0	10.83	45.50
18	1.025	0	0	17.73	50.33	0.856	0	0	2.36	9.90
19	1.005	0	0	4.02	11.39	0.853	0	0	0.24	1.00
20	1.000	0	0	0.71	2.00	0.853	0	0	0	0

Flows: Organic - 500 ml; Slurry - 144 ml; Scrub - 32 ml; Strip - 338 ml

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TABLE 25 PILOT PLANT SHAKESOUT - 22.30% TBP + 1.00% DBP- 20 STAGE

Aqueous						Organic				
Stage No. 1	Sp. Gr. @ 21°C 2	HNO ₃ g/l. 3	ME HNO ₃ x Flow ³ 4	U g./l. 5	ME. U. x Flow 6	Sp. Gr. @ 21°C 7	HNO ₃ g./l. 8	ME HNO ₃ x Flow ³ 9	U g./l. 10	ME.U x Flow 11
1	1.035	55.41	87.92	0.007	0.01	0.853	9.33	74.00	0.009	0.05
2	1.075	99.90	158.52	0.009	0.01	0.856	17.58	139.45	0.07	0.30
3	1.100	147.97	234.79	0.04	0.03	0.859	26.30	208.65	0.009	0.05
4	1.125	197.35	313.15	0.03	0.02	0.861	32.97	261.55	0.02	0.10
5	1.150	237.07	376.18	0.20	0.16	0.865	37.58	298.15	0.04	0.15
6	1.175	261.79	415.40	4.50	3.78	0.927	19.72	156.45	45.60	191.55
7	1.440	165.27	262.25	258.00	216.80	0.973	4.73	37.50	90.00	378.15
8	1.335	43.89	22.98	197.60	54.79	0.970	0	0	75.60	317.60
9	1.300	17.49	9.16	194.00	53.79	0.968	0	0	74.80	314.25
10	1.250	5.83	3.05	151.40	41.98	0.964	0	0	70.80	297.45

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TABLE 25

Aqueous							Organic			
1	2	3	4	5	6	7	8	9	10	11
11	1.190	0	0	117.00	32.44	0.953	0	0	68.00	285.70
12	1.126	1.78	14.10	78.00	327.70	0.920	0	0	63.20	265.50
13	1.088	0.90	7.10	53.00	222.65	0.906	0	0	37.80	158.80
14	1.070	0.83	6.55	43.20	181.50	0.890	0	0	34.20	143.65
15	1.042	0.49	3.85	25.50	107.10	0.875	0	0	31.20	131.05
16	1.030	0.37	2.90	17.90	75.20	0.870	0	0	18.80	78.95
17	1.020	0.35	2.75	12.30	51.65	0.867	0	0	14.40	60.50
18	1.014	0.39	3.05	8.20	34.45	0.864	0	0	12.00	50.40
19	1.006	0.49	3.85	2.90	12.15	0.862	0	0	11.20	47.05
20	1.004	0.49	3.85	1.60	6.70	0.858	0	0	9.20	38.65

Flows: Organic - 500 ml; Slurry - 100 ml; Scrub - 33 ml; Strip - 500 ml

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