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SUBJECT: THOREX PILOT PLANT RUN AWD-1 SUMMARY

TO: F. L. Culler

FROM: W. T. McDuffee and O. O. Yarbro

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## 1.0 INTRODUCTION

The processing of short-decayed thorium metal results in the collection of first cycle extraction column raffinate which contains 20 to 30 per cent of the original mass $^{233}$ as $Pa^{233}$. Ultimate recovery of the $U^{233}$ requires storage of the raffinate for decay of $Pa^{233}$. During the Thorex Pilot Plant short-decay scouting run HD-19, an estimated 27 g of $Pa^{233}$ was collected and stored as extraction column raffinate. This report summarizes the data resulting from the processing (run AWD-1) of the first cycle raffinate from run HD-19. A period of 346 days elapsed between the completion of run HD-19 and run AWD-1.

A one-cycle solvent-extraction flowsheet was used to separate $U^{233}$ from fission products and other contaminants contained in the raffinate. The $U^{233}$ was extracted into 6 per cent tributyl phosphate in Amsco 125-82 and subsequently stripped into dilute nitric acid. Feed for run AWD-1 was prepared by concentrating and adjusting the 1A-column raffinate from run HD-19 to feed specifications.

The primary objectives of run AWD-1 were as follows:

1. Demonstrate a flowsheet for the recovery of $U^{233}$ from first cycle extraction column raffinate after sufficient storage time to allow the $Pa^{233}$ to decay to $U^{233}$.
2. Recover approximately 27 g of $U^{233}$ with high isotopic purity from the first cycle raffinate from run HD-19.

## 2.0 RESULTS AND OBSERVATIONS

The first solvent-extraction cycle of the Thorex Pilot Plant was operated for approximately 8 hr to process the first cycle extraction column raffinate resulting from the short-decay scouting run HD-19. Synthetic feed prepared from aluminum nitrate was used for the startup and shutdown of the system. Uranium extraction losses averaged 9.6 per cent and stripping losses averaged 0.04 per cent. The extraction column raffinate was reprocessed to recover 91 per cent of the uranium lost during the first pass through the system. The high uranium losses were attributed to the low $U^{233}$ concentration in the feed. The uranium material balance around the solvent-extraction system was 102.4 per cent. A total of 21.37 g of $U^{233}$ was recovered as product which was 99.56 per cent $U^{233}$ compared to ~ 98 per cent routinely recovered in normal Thorex processing (Table 2.1). Further decontamination from both radive and ionic contaminants, principally corrosion products, will be required.

Table 2.1. Isotopic Analysis - Uranium Product

| | Uranium Isotope, wt % | | | |
|---|---|---|---|---|
| | $U^{233}$ | $U^{234}$ | $U^{235}$ | $U^{238}$ |
| Uranium Product | 99.56 | 0.08 | 0.00 | 0.36 |

The $U^{233}$ product was decontaminated from gross gamma activity by a factor of $1.9 \times 10^3$ (Table 2.2) yielding a product with an activity of $1.2 \times 10^7$ gross gamma cts/min/mg U (Table 2.3).

Table 2.2. Decontamination Factors

| | Decontamination Factors | | | | |
|---|---|---|---|---|---|
| | Gross $\gamma$ | Pa | Ru | Zr-Nb | TRE |
| 1AF/1CU | $1.9 \times 10^3$ | $1.3 \times 10^3$ | $1.6 \times 10^4$ | $1.2 \times 10^3$ | $2.7 \times 10^6$ |

Table 2.3. Activity Distribution

| | Activity, cts/min/mg U | | | | |
|---|---|---|---|---|---|
| | Gross $\gamma$ | Pa $\gamma$ | Ru $\gamma$ | Zr-Nb $\gamma$ | TRE $\beta$ |
| Feed | $2.3 \times 10^{10}$ | $5.5 \times 10^{8}$ | $5.9 \times 10^{8}$ | $1.3 \times 10^{10}$ | $3.8 \times 10^{10}$ |
| Product | $1.2 \times 10^{7}$ | $4.4 \times 10^{5}$ | $3.6 \times 10^{4}$ | $1.1 \times 10^{7}$ | $1.4 \times 10^{4}$ |

The assay of concentrated 1A-column raffinate after approximately 1 year storage was difficult owing to the formation of precipitates that interfered both with accurate analyses and with obtaining representative samples.

Although the feed processed during this run was characterized by the presence of higher than normal concentrations of degradation products of TBP and diluent, the Thorex Pilot Plant first cycle equipment operated smoothly. The flowsheet was operable and with further development would probably provide higher decontamination from fission products and $Pa^{233}$. Substitution of ion-exchange isolation for simple evaporation would probably reduce the ionic contamination found in the product and provide additional decontamination from fission products and $Pa^{233}$.

The extraction loss in the 1A-column in the first pass was 9.6 per cent and occurred when the 1A-column pulse frequency was 30 cycles/min; upon adjustment and reprocessing the raffinate from the first pass, using a 1A-column pulse frequency of 40 cycles/min, the losses were again ~ 9 per cent of the feed. However, during the second pass the uranium concentration in the raffinate was reduced to the level observed in normal Thorex processing (~ $10^3$ $U^{233}$ $\alpha$ cts/min/ml). It is believed that had the pulse frequency been 40 cycles/min during the first pass, the first pass losses would have been ~ 1 per cent.

## 3.0 EQUIPMENT AND FLOWSHEET

The equipment associated with the first solvent-extraction cycle of the Thorex Co-decontamination Flowsheet was used for run AWD-1.

The first cycle raffinate from short-decay scouting run HD-19 was transferred to the feed adjustment tank, concentrated, and adjusted to 1A-column feed conditions (Table 3.1). After feed adjustment, the solution was transferred to the feed tank and metered to the extraction column with the first cycle airlifts. Uranium was extracted into 6.1 per cent tributyl phosphate in Amsco in the 1A-column and stripped into 0.01 N nitric acid in the 1C-column. The uranium product from the stripping column was collected in the intercycle evaporator and batch-concentrated to 0.85 g uranium/liter. The flowrates and average compositions of the process streams are listed in Table 3.1.

Table 3.1. Composition and Flowrates---Process Streams

| Stream | Composition | Flowrate, 1/hr |
|---|---|---|
| 1AF | $H^+$, 1.0 N; Al, 1.8 M; Th, 3.57 g/1; U, 0.0238 g/1 | 126 |
| 1AX | TBP, 6.1%; Amsco, 93.9% | 59 |
| 1AS | $H^+$, 2.9 N; Al, 0.68 M | 21 |
| 1AW | $H^+$, 1.2 N; Al, 1.6 M | 147 |
| 1AP | TBP, 6.1%; Amsco, 93.9%; U, 0.0508 g/1[a] | 59 |
| 1CX | $H^+$, 0.01 N | 16 |
| 1CP | U, 0.187 g/1[a] | 16 |
| 1CW | TBP, 6.1%; Amsco, 93.9% | 59 |

a. Calculated from 1AF concentration and average flowrates. The uranium concentration reached ~ one-half of these equilibrium values during run AWD-1.