OAK RIDGE NATIONAL LABORATORY LIBRARIES
3 4456 0549263 2

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION

**OAK RIDGE NATIONAL LABORATORY**
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

ORNL-TM-1256
COPY NO. - 26
DATE - August 30, 1965

# EVALUATION OF SMALL MODIFIED CEN MIXER-SETTLERS FOR THE EXTRACTION OF URANIUM AND THORIUM IN A HOT CELL FACILITY

J. Klitgaard* and J. H. Goode

## ABSTRACT

In a study of a small modified version of the mixer-settlers used at the Centre d'Etude de L'Energie Nucleaire, at Mol, Belgium, a cascade of three mixer-settlers was made and tested with nonradioactive, simulated feeds adjusted to meet conditions for their purification by the Acid Thorex and Purex processes. The mixer-settlers were constructed from molded Homalite plastic and had 8 extraction, 8 scrub, 8 partitioning, 8 back-extraction, and 16 stripping stages. The chief objective of the study was to acquire performance data (throughput, stage efficiencies, and operating limitations, such as losses) before the equipment is used in a process demonstration with highly radioactive feed.

Results indicated that this type of mixer-settler should be satisfactory for hot-cell studies of aqueous nitrate solvent extraction systems. The uranium losses during a Purex run totalled 0.007%, and the overall stage efficiencies were 65 and 77% for extraction and stripping, respectively. In an Acid Thorex run, the uranium losses totalled 0.19% and the thorium losses 0.33%. The separation factors were: thorium from uranium, 360; uranium from thorium, 555. Methods were developed for adjusting the impellers (paddles) to ensure proper hydraulic performance of the units.

---

*Guest scientist from Mol, Belgium.

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION
LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this document, send in name with document and the library will arrange a loan.

**NOTICE** This document contains information of a preliminary nature and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

## 1. INTRODUCTION

Previous hot cell experience with the stainless steel and fluorothene KAPL "Mini" mixer-settler,[1] used for Purex and plutonium extraction studies[2] and for strontium recovery and Acid Thorex experiments,[3] pointed out limitations due to the low (4 ml/min) total flow capacity of the unit and the difficulty of pumping these small flows of reagents required. The ORNL "Immi" mixer-settler,[2,3] a stainless steel unit with a fluorothene window, had a total flow capacity of up to 578 ml/min and was much too large for use with limited quantities of highly-radioactive feed solutions. A low cost mixer-settler of intermediate capacity was desired for use in small scale hot cell studies since it could be discarded when repair or decontamination was impractical.

A molded Homalite plastic unit was constructed based on the original design of the 16-stage, box-type Centre d'Etude de L'Energie Nucleaire[4] (CEN) unit as modified at Oak Ridge National Laboratory for use in the Curium Recovery Program.[5] The Homalite plastic failed in the high temperature chloride system used in the latter program but proved satisfactory in the nitrate system used in the work outlined in this report.

## 2. DESCRIPTION OF EQUIPMENT

### 2.1 The Mixer-Settler

Four changes were made in the original CEN apparatus. (1) It was enlarged to provide a holdup of 1 liter and to permit a flow rate of 1 liter/hr; (2) the impeller was altered to improve mixing; (3) an interface-control weir was installed between the stages to allow a wider range of mixing speed without causing loss of interface, which results in decreased efficiency due to backmixing; and (4) the units were made by casting instead of machining as an economy measure. Figure 1 shows one of the units. Nylon gears mounted on each impeller shaft are interconnected, giving adjacent impellers opposite directions of rotation but the same speed. The gear train is driven by a variable-speed direct-current motor. Three mixer-settlers were arranged in a descending cascade to permit gravity flow. Figure 2 shows the assembly ready to use.

OAK RIDGE NATIONAL LABORATORY LIBRARIES

3 4456 0549263 2

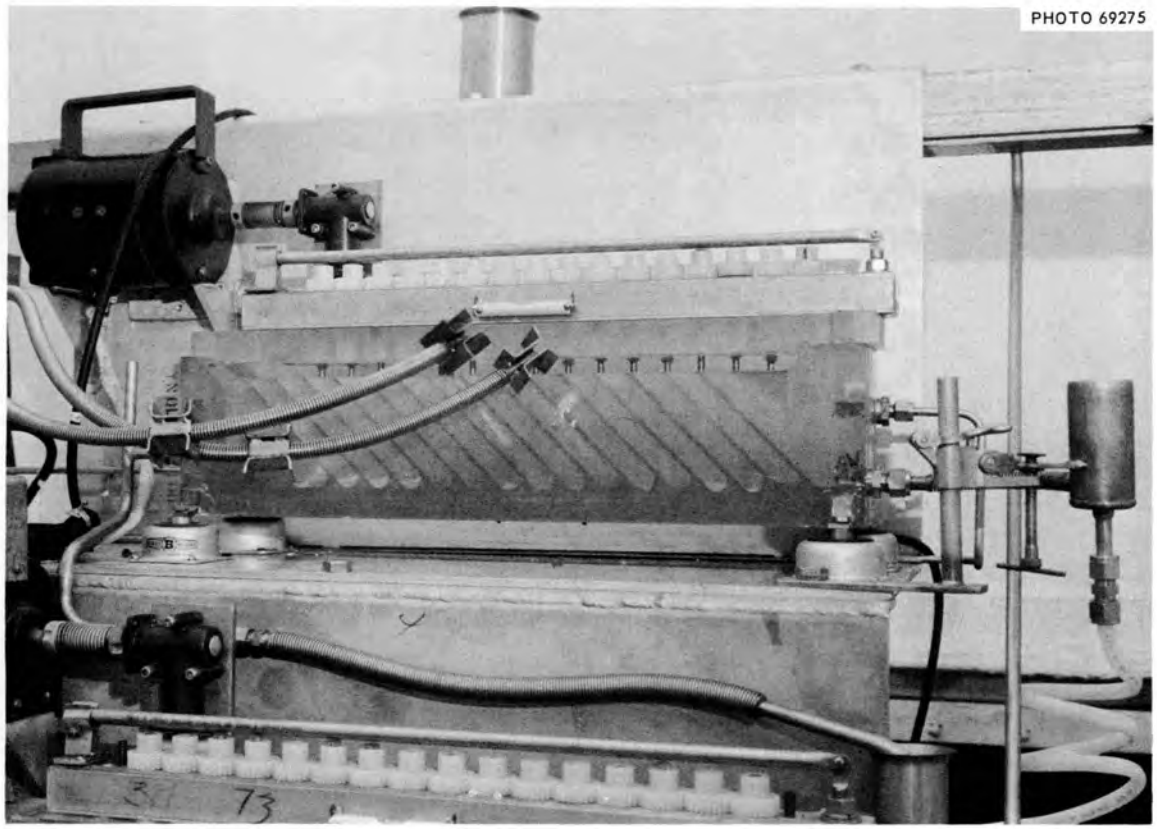


Fig. 1. Modified CEN 16-Stage Mixer-Settler Cast from Homalite.

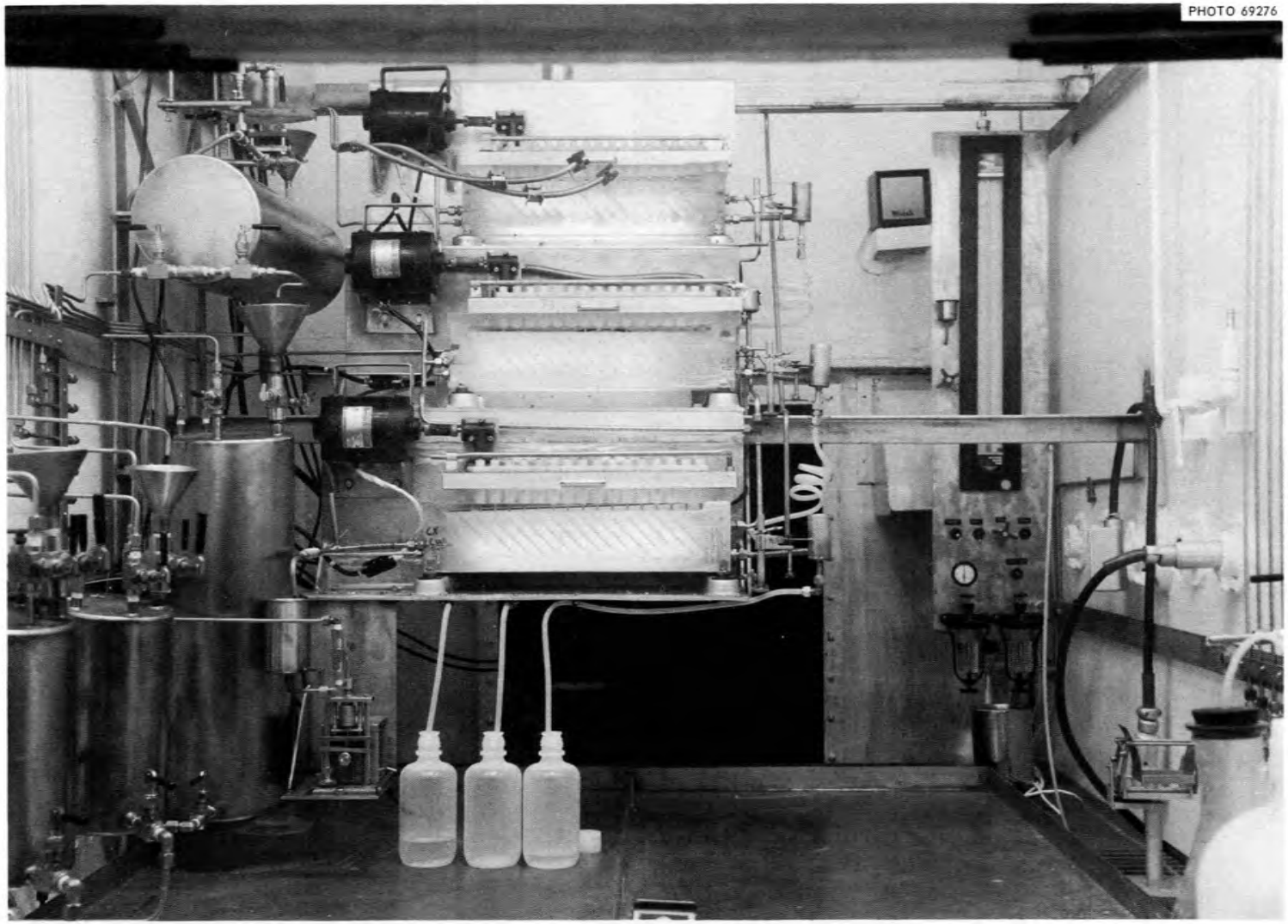


Fig. 2. Modified CEN Mixer-Settler Cascade in Hot Cell.

In making them, each mixer-settler was cast in four sections in Homalite-1100, a two-component epoxy-base resin, which were then glued together. The masters of each section were made of aluminum, by machining, and the final molds were made with silicone rubber.

### 2.2 The Sampling System

Figure 3 is a schematic drawing of the sampling system. It was made by drilling 32 holes in a wooden block on a 45° angle to accommodate two glass pipes that dipped into each settler, one ending above the interface, the other below. Short neoprene hoses were connected to the glass pipes, and hypodermic needles were placed in the ends of the hoses. Sample bottles with neoprene diaphragm caps were placed in holes drilled in the block. The needles were placed in the sample bottles, and all neoprene hoses were squeezed between the steel bars mounted on the block. Vacuum — the amount of which depended on the sample volume needed — was applied to each sample bottle through a hypodermic needle. With the block placed on a mixer-settler, 32 samples could then be taken simultaneously by releasing the pressure on the hoses.

### 2.3 Auxiliaries

Feed pumps (Lapp Model LS-10 and Model LS-20 "Pulsafeeder") and cold tanks were mounted on a rack placed outside the hood or hot cell. All tanks were equipped with a valve and a graduated sight glass to permit fast determinations of the pumping rate. The active feed tank proper was a closed tank placed next to the mixer-settler. The feed was metered by displacement with diluent pumped into the tank from outside the cell. Waste and product tanks were graduated cylinders to allow flow-rate determinations by the composite-volume method.

## 3. TESTS

### 3.1 Impeller Adjustments

Faulty operation during the initial hydraulic tests was first thought to be due to insufficient pumping by the cast Homalite paddle-type impellers,

ORNL DWG 65-7814

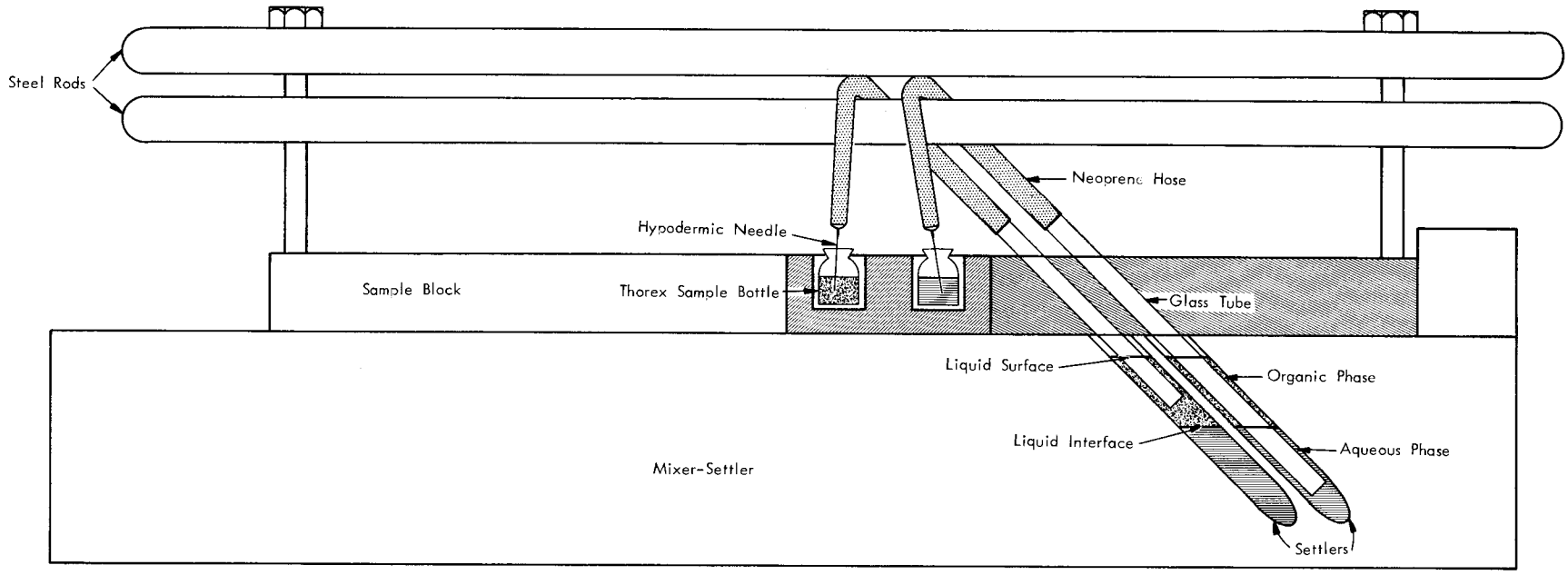


Fig. 3. The Sampling System — Schematic.

but it was found that the mixer-settler sections were not properly glued together, resulting in interconnection of several or all settlers in a bank. Re-gluing eliminated that source of trouble.

Experience with these cast Homalite-1100 units soon showed that the castings should be given time to shrink before the holes for the impeller shafts in the bearing block are drilled. This will prevent their being moved off center because of shrinkage. Eccentric impellers lead to poor flow patterns. We obtained stable operation by inserting washers in the bearing block (this changed the level of the impeller) and by adjusting the width of the impeller blades.

Future operators of the apparatus may find the following five suggestions useful:

1. The operation of the mixing compartment can be changed from organic continuous to aqueous continuous by lowering the impeller as far as possible while still maintaining sufficient pumping. (We finally used a smaller diameter impeller.)
2. The interface in a settler can be lowered by lowering the impeller in the mixing chamber that sucks aqueous phase from the settler. This permits a higher pumping rate. (Finally, we used a larger impeller.)
3. The interface in a settler can be raised by raising the impeller in the mixing chamber that draws aqueous phase from the settler. This change provides a lower pumping rate. (Eventually, a smaller impeller was used.)
4. When the interface level across the unit varies from low at the aqueous inlet to high at the aqueous outlet, reducing the impeller speed levels the interface heights in all the settlers.
5. However, if the interface levels vary from high at the aqueous inlet to low at the aqueous outlet, increasing the impeller speed levels the interface heights in all the settlers.

According to the above, it was possible to obtain stable, aqueous-continuous operation in two of the units. However, in the extraction unit, three of the settlers near the feed point had larger dispersion bands than the others (for instance, 60-mm vs 10-mm band widths, respectively). Continued operation under these conditions could have resulted in emulsion flooding or inversion plus subsequent flooding. However, small pieces of fritted glass filter were inserted in these settlers, breaking the emulsions. The dispersion bands then reached the same width as in the other settlers, resulting in trouble-free operation.

### 3.2 Extraction Runs

Extraction tests were made with standard Purex and Acid Thorex[6] flowsheets (Figs. 4 and 5). Plutonium could not be used in these tests, so the partitioning unit (B-bank) was bypassed during Purex runs. The AP stream was diluted with an amount of fresh organic equal to the BS flowrate to obtain correct concentration and flow conditions in the stripping bank (C-bank). The bypassing of B-bank is indicated on Fig. 4.

Six Purex runs — BMS-2 through BMS-7 — were performed. The first three were mostly designed to evaluate the hydraulics of the system. However, the few samples taken indicated an efficiency far below that expected, which then in turn led to the discovery of the excessive leaks between the settlers mentioned in Sec. 3.1.

The remaining runs were efficiency runs with flowrates of 100%, 75%, and 150%, respectively, of those indicated on the standard flowsheet (Fig. 4).

Three Acid Thorex runs were made: Thorex-1 was a scouting run with little sampling, and Thorex-2 and -3 were efficiency runs at flowrates of 100% and 200%, respectively, of the standard rates (Fig. 5).

## 4. RESULTS OF EFFICIENCY RUNS

### 4.1 Run Conditions

The operating conditions for the three Purex runs are given in Table 1. Table 2 gives the analyses of the samples taken. Similarly, Tables 3 and 4 give conditions and analyses, respectively, for the two Acid Thorex runs.

### 4.2 Losses and Separation Factors

The large number of actual stages in each mixer-settler bank resulted in very small losses. During the Purex runs the uranium losses to the waste streams never exceeded 0.007%. The highest uranium and thorium losses experienced during the Thorex runs were 0.19% and 0.073%, respectively.

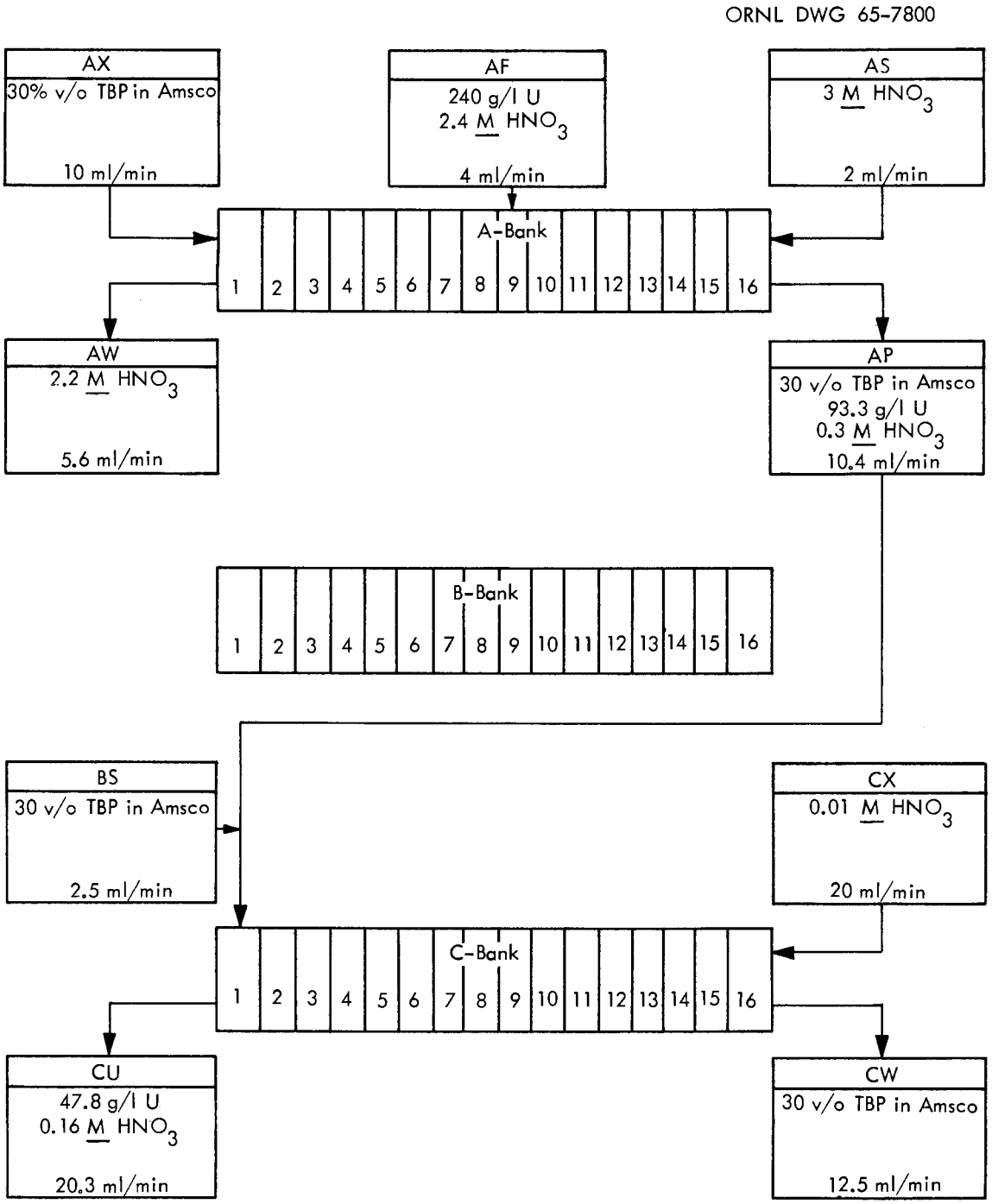


Fig. 4. Standard Flowsheet — Purex.

ORNL DWG 65-7801

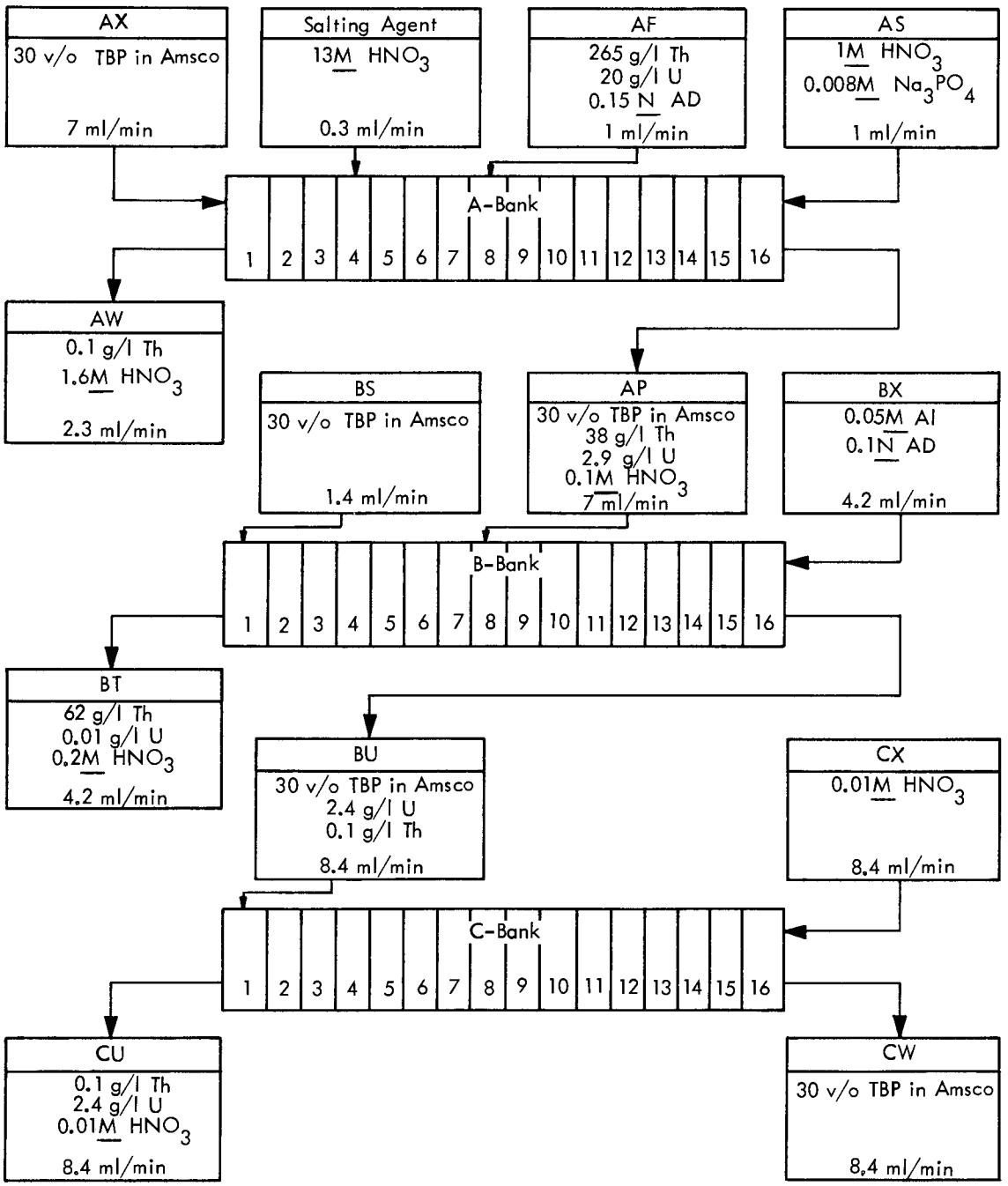


Fig. 5. Standard Flowsheet — Thorex.

Table 1. Purex Efficiency Runs. Run Conditions.

| | Run No. | | |
|---|---|---|---|
| | BMS-5 | BMS-6 | BMS-7 |
| Percent of Standard Flowrates | 100 | 75 | 150 |
| Length of Run, hours | 8 | 7.4 | 5.5 |
| Impeller Speed, A-bank, rpm | 660 | 685 | 660 |
| Impeller Speed, C-bank, rpm | 495 | 515 | 490 |
| Temperature, °C | 26 | 25.5 | 25.5 |
| Flowrates: | | | |
| AX, ml/min | 10.10 | 7.46 | 14.68 |
| AF, ml/min | 3.91 | 2.85 | 5.64 |
| AS, ml/min | 2.01 | 1.50 | 3.02 |
| BS, ml/min | 2.49 | 1.89 | 3.70 |
| CS, ml/min | 19.97 | 14.95 | 30.18 |
| AW, ml/min | 5.48 | 4.08 | 8.25 |
| CU, ml/min | 20.26 | 15.63 | 31.06 |
| CW, ml/min | 12.88 | 9.43 | 18.67 |
| Flowrates averaged over, hours | 5 | 5 | 3 |
| Flow Balance (flow, out/flow, in), percent | 100.4 | 101.7 | 101.3 |
| AX and BS, v/o TBP | 29.6 | 30.1 | 30.3 |
| AX and BS, $H^+$, M | | 0.0006 | |
| AF, g/l U | 241. | 247. | 242. |
| AF, $H^+$, M | 2.42 | 2.37 | 2.33 |
| AS, $H^+$, M | 3.44 | 3.51 | 3.19 |
| CX, $H^+$, M | 0.011 | 0.014 | 0.011 |

During the Acid Thorex tests, thorium was satisfactorily separated (as the BT stream) from the uranium using 8 stages of the "B", or partitioning, mixer-settler by factors of 360 and 210 using the standard and 200% flowrates, respectively. Uranium was separated from the thorium by a "normal" factor of 1460 during the 200% flowrate run; however, during the 100% flowrate run, the separation factor was only 555. This is probably due to the fact that equilibrium had not been reached when sampling took place because of the small flowrates in the scrubbing part of the B-bank. The results are summarized in Table 5.

Table 2. Purex Efficiency Runs. Samples.

| Stage No. | Run No. BMS-5 Aqueous U, g/l | BMS-5 Aqueous $H^+$, M | BMS-5 Organic U, g/l | BMS-5 Organic $H^+$, M | BMS-6 Aqueous U, g/l | BMS-6 Aqueous $H^+$, M | BMS-6 Organic U, g/l | BMS-6 Organic $H^+$, M | BMS-7 Aqueous U, g/l | BMS-7 Aqueous $H^+$, M | BMS-7 Organic U, g/l | BMS-7 Organic $H^+$, M |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A-1 | 0.001 | 2.47 | | | 0.005 | 2.48 | | | 0.002 | 2.48 | 0.0005 | 0.540 |
| A-2 | 0.002 | 3.31 | 0.008 | 0.705 | 0.0007 | 3.35 | 0.0008 | 0.690 | 0.0003 | 3.25 | 0.0009 | 0.690 |
| A-3 | | | | | | | | | 0.0002 | 3.51 | 0.0003 | 0.741 |
| A-4 | 0.0004 | 3.68 | 0.004 | 0.775 | 0.0004 | 3.70 | 0.002 | 0.769 | 0.0005 | 3.59 | 0.010 | 0.760 |
| A-5 | | | | | | | | | 0.007 | 3.61 | 0.053 | 0.760 |
| A-6 | 0.032 | 3.71 | 0.368 | 0.770 | 0.032 | 3.74 | 0.373 | 0.758 | 0.121 | 3.62 | 0.798 | 0.762 |
| A-7 | 0.886 | 3.70 | 11.8 | 0.720 | 0.749 | 3.74 | 12.3 | 0.703 | 1.28 | 3.63 | 18.3 | 0.772 |
| A-8 | 22.0 | 3.58 | 89.9 | 0.265 | 24.8 | 3.59 | 89.9 | 0.323 | 30.3 | 3.48 | 94.0 | 0.280 |
| A-9 | | | | | | | | | 149 | 2.86 | 93.3 | 0.270 |
| A-10 | 13.8 | 3.41 | 86.0 | 0.252 | 15.2 | 3.48 | 86.0 | 0.315 | 18.7 | 3.17 | 90.9 | 0.281 |
| A-11 | | | | | | | | | 20.0 | 3.14 | 92.9 | 0.282 |
| A-12 | 15.3 | 3.36 | 86.8 | 0.247 | 15.7 | 3.44 | 86.8 | 0.310 | 21.4 | 3.12 | 92.6 | 0.275 |
| A-13 | | | | | | | | | 20.7 | 3.13 | 94.6 | 0.270 |
| A-14 | 15.1 | 3.46 | 86.5 | 0.243 | 15.2 | 3.44 | 87.3 | 0.310 | 19.6 | 3.07 | 93.8 | 0.280 |
| A-15 | | | | | | | | | 18.8 | 3.12 | 92.9 | 0.286 |
| A-16 | 12.4 | 3.46 | 85.9 | 0.252 | 12.7 | 3.45 | 87.1 | 0.320 | 15.4 | 3.14 | 90.5 | 0.295 |
| C-1 | 44.6 | 0.188 | | | 45.4 | 0.219 | 55.9 | 0.014 | 46.8 | 0.152 | 74.4 | 0.034 |
| C-2 | 36.6 | 0.086 | 43.4 | 0.008 | 37.3 | 0.119 | 39.7 | 0.006 | 41.0 | 0.040 | 48.9 | 0.011 |
| C-3 | | | | | 25.9 | 0.041 | 24.0 | 0.0002 | 31.4 | 0.016 | 34.3 | 0.002 |
| C-4 | 17.0 | 0.054 | 14.9 | 0.001 | 15.1 | 0.035 | 11.0 | 0.001 | 22.3 | 0.015 | 20.9 | 0.004 |
| C-5 | | | | | | | | | 13.2 | 0.015 | 11.2 | 0.002 |
| C-6 | 3.55 | 0.034 | 1.98 | 0.002 | 2.97 | 0.037 | 1.73 | 0.003 | 6.87 | 0.011 | 4.49 | 0.003 |
| C-7 | 1.28 | 0.033 | 0.755 | 0.002 | | | | | 2.89 | 0.012 | 2.47 | 0.001 |
| C-8 | 0.52 | 0.025 | 0.275 | 0.001 | 0.656 | 0.013 | 0.248 | 0.0008 | 1.77 | 0.011 | 0.685 | 0.0007 |
| C-9 | | | | | | | | | 0.461 | 0.011 | | |
| C-10 | 0.067 | 0.012 | 0.031 | 0.001 | 0.097 | 0.012 | 0.048 | 0.0006 | 0.108 | 0.011 | 0.020 | 0.0002 |
| C-11 | | | | | | | | | 0.020 | 0.009 | 0.030 | 0.0002 |
| C-12 | 0.014 | 0.010 | 0.005 | 0.001 | 0.010 | 0.012 | 0.007 | 0.0008 | 0.013 | 0.011 | 0.026 | 0.0004 |
| C-13 | | | | | | | | | 0.010 | 0.011 | 0.015 | 0.0001 |
| C-14 | 0.001 | 0.011 | 0.001 | <0.001 | 0.0009 | 0.012 | 0.0009 | 0.0007 | 0.006 | 0.011 | 0.003 | 0.0001 |
| C-15 | | | | | | | | | 0.002 | 0.011 | 0.006 | 0.0001 |
| C-16 | 0.0006 | 0.011 | 0.005 | <0.001 | 0.003 | 0.012 | 0.0002 | 0.0004 | 0.010 | 0.011 | 0.004 | 0.0001 |

Table 3. Thorex Efficiency Runs. Run Conditions.

| | Run No. | |
|---|---|---|
| | Thorex-2 | Thorex-3 |
| Percent of Standard Flowrates | 100 | 200 |
| Length of Run, hours | 8.3 | 7.5 |
| Impeller Speed, A-bank, rpm | ~700 | ~700 |
| Impeller Speed, B-bank, rpm | ~700 | ~700 |
| Impeller Speed, C-bank, rpm | ~700 | ~700 |
| Temperature, °C | 26 | 26 |
| Flowrates: | | |
| AX, ml/min | 7.0 | 14.0 |
| Salting Agent, ml/min | 0.3 | 0.6 |
| AF, ml/min | 1.0 | 2.0 |
| AS, ml/min | 1.0 | 2.0 |
| BS, ml/min | 1.4 | 2.8 |
| BX, ml/min | 4.2 | 8.4 |
| CX, ml/min | 8.4 | 16.8 |
| AX and BS, v/o, TBP | 30.8 | 30.8 |
| AF, g/l Th | 245.2 | 245.2 |
| AF, g/l U | 20.9 | 20.9 |
| AF, acid deficiency, N | 0.20 | 0.20 |

## 4.3 Efficiencies

The overall stage efficiency for the Purex flowsheet was determined from McCabe-Thiele diagrams. The diagrams for Purex run BMS-7 are included. Figure 6 shows the diagram for extraction-scrubbing, Fig. 7 for stripping. The overall efficiencies have only been evaluated for the "concentrated ends" of the banks where the analyses are more accurate and the results consistent. The efficiencies are given in Table 6. For the extraction, it was 65-67%, regardless of flowrate. The stripping efficiency showed some flowrate dependency, although not entirely consistent with 77%, 70%, and 56% for 100%, 75%, and 150%, respectively, of standard conditions.

Table 4. Thorex Efficiency Runs. Samples.

| Stage No. | Run No. Thorex-2 Aqueous Th, g/l | Thorex-2 Aqueous U, g/l | Thorex-2 Aqueous $H^+$, M | Thorex-2 Organic Th, g/l | Thorex-2 Organic U, g/l | Thorex-2 Organic $H^+$, M | Thorex-3 Aqueous Th, g/l | Thorex-3 Aqueous U, g/l | Thorex-3 Organic Th, g/l | Thorex-3 Organic U, g/l |
|---|---|---|---|---|---|---|---|---|---|---|
| A-1 | 0.061 | 0.0001 | 1.53 | 0.042 | 0.004 | | 0.078 | 0.00007 | 0.043 | 0.0041 |
| A-2 | 0.185 | 0.0007 | 2.50 | 0.289 | 0.002 | | | | | |
| A-3 | 0.589 | 0.0008 | 3.09 | 0.758 | 0.004 | | 0.773 | 0.00003 | 1.07 | 0.00031 |
| A-4 | | | | 4.4 | 0.003 | | | | | |
| A-5 | 18.8 | 0.002 | 1.85 | 15.0 | 0.088 | | 22.6 | 0.0012 | 24.5 | 0.0352 |
| A-6 | | 0.017 | 1.25 | 25.5 | 0.75 | | | | | |
| A-7 | | 0.358 | 1.04 | 39.6 | 2.62 | | 105 | 0.420 | 45.3 | 1.99 |
| A-8 | 92.8 | 2.76 | | 40.1 | 2.51 | | | | | |
| A-9 | 72.4 | 0.31 | | 40.6 | 2.58 | | 96.6 | 0.185 | 45.7 | 2.08 |
| A-10 | 41.3 | 2.45 | | 40.7 | 2.63 | | | | | |
| A-11 | | | | 40.9 | 2.64 | | 94.2 | 0.216 | 45.8 | 2.08 |
| A-12 | 75.5 | 0.294 | | 40.8 | 2.40 | | | | | |
| A-13 | 75.5 | 0.297 | | 40.5 | 2.55 | | 94.0 | 0.215 | 45.0 | 2.62 |
| A-14 | 74.2 | 0.300 | | 39.5 | 2.58 | | | | | |
| A-15 | 64.6 | 0.303 | | 38.5 | 2.49 | | 73.2 | 0.215 | 40.9 | 2.65 |
| A-16 | 50.7 | 0.338 | 0.92 | 35.5 | 2.60 | 0.15 | 108 | 0.205 | 35.9 | 2.64 |
| B-1 | 56.4 | 0.009 | | 22.8 | 0.034 | | 56.9 | 0.0034 | 22.9 | 0.00018 |
| B-2 | 61.8 | 0.020 | | 21.8 | 0.086 | | | | | |
| B-3 | 65.3 | 0.031 | | | | | 65.6 | 0.0185 | 29.8 | 0.0022 |
| B-4 | 66.4 | 0.064 | | 31.5 | 0.167 | | | | | |
| B-5 | 67.0 | 0.080 | | 31.2 | 0.251 | | 66.8 | 0.041 | 30.5 | 0.0061 |
| B-6 | 66.9 | 0.122 | | 31.8 | 0.411 | | | | | |
| B-7 | 67.8 | 0.172 | | 31.7 | 1.25 | | 67.2 | 0.134 | 30.5 | 0.432 |
| B-8 | 68.3 | 0.324 | | 30.3 | 2.12 | | | | | |
| B-9 | 66.4 | 0.381 | | 27.4 | 2.25 | | 67.0 | 0.37 | 29.0 | 1.36 |
| B-10 | 49.7 | 0.413 | | 22.4 | 2.74 | | | | | |
| B-11 | 48.0 | 0.810 | | | | | 52.8 | 0.66 | 18.8 | 2.84 |
| B-12 | | | | 8.11 | 4.16 | | | | | |
| B-13 | 15.2 | 4.83 | | 4.03 | 5.47 | | 24.7 | 2.75 | 5.48 | 4.61 |
| B-14 | 6.39 | 7.23 | | 1.14 | 5.71 | | | | | |
| B-15 | 0.83 | 7.28 | | 0.369 | 5.00 | | 3.38 | 5.48 | 0.64 | 4.17 |
| B-16 | 0.24 | 4.08 | | 0.081 | 3.15 | | 0.92 | 3.45 | 0.14 | 2.36 |
| C-1 | | 1.95 | | | 0.33 | | 0.159 | 2.03 | | |
| C-2 | | 0.355 | | | | | | | | |
| C-3 | | | | | | | 0.031 | 0.27 | | |
| C-4 | | 0.015 | | | | | | | | |
| C-5 | | | | | | | 0.006 | 0.0083 | | |
| C-6 | | 0.0009 | | | 0.0013 | | | | | |
| C-7 | | | | | | | 0.002 | 0.0009 | | |
| C-8 | | 0.0008 | | | | | | | | |
| C-9 | | | | | 0.0012 | | 0.001 | 0.00009 | | |
| C-10 | | | | | | | | | | |
| C-11 | | | | | | | 0.001 | 0.00003 | | |
| C-12 | | 0.0002 | | | 0.0005 | | | | | |
| C-13 | | | | | | | 0.0004 | 0.00003 | | |
| C-14 | | 0.0001 | | | 0.0006 | | | | | |
| C-15 | | | | | | | 0.0001 | 0.00002 | | |
| C-16 | | 0.00005 | | 0.003 | 0.0003 | | 0.0006 | 0.00001 | | |

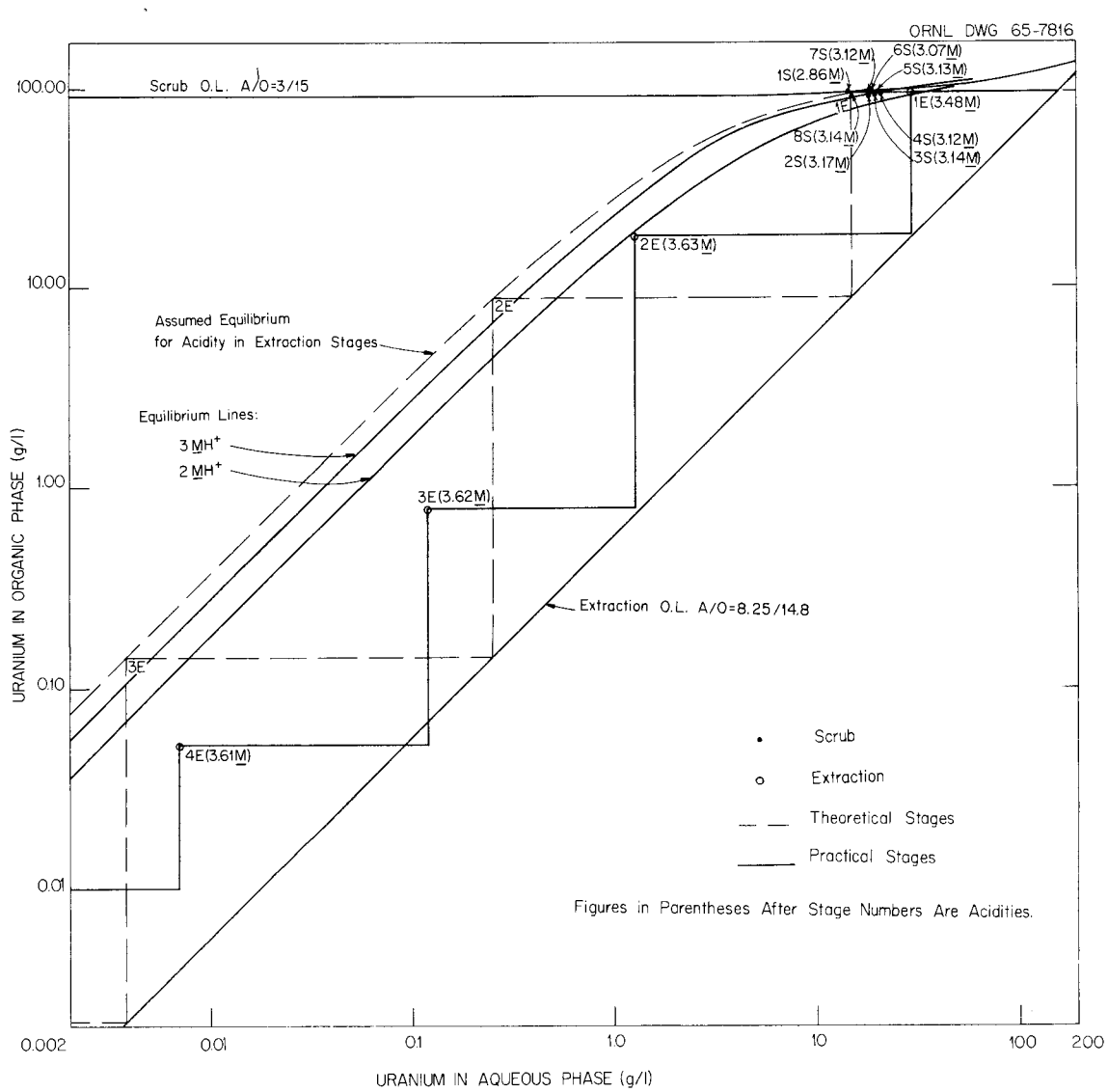


**Fig. 6. McCabe-Thiele Diagram. Purex Run BMS-7. A-Bank. Extraction and Scrub.**

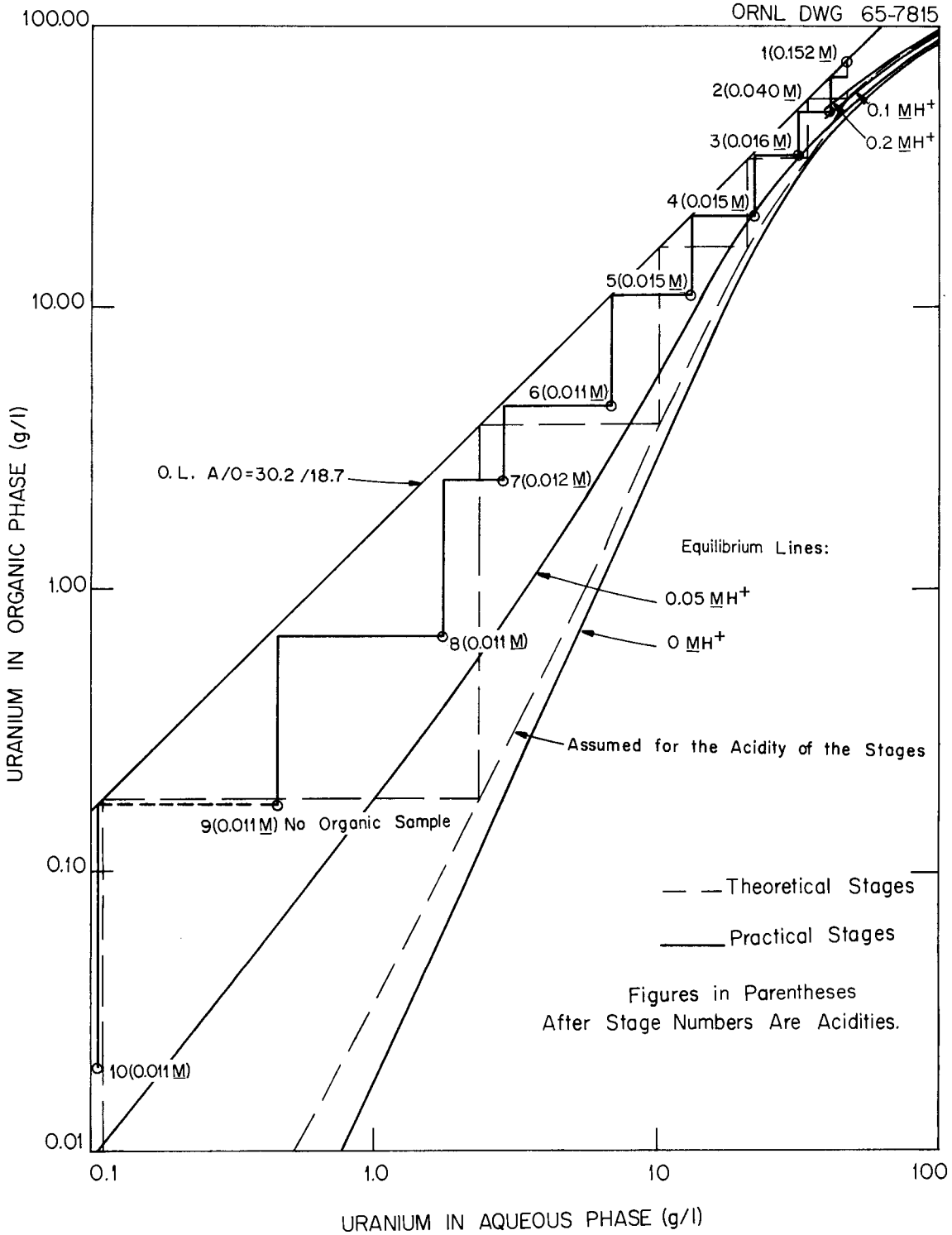


Fig. 7. McCabe-Thiele Diagram. Purex Run BMS-7. C-Bank. Stripping.

Table 5. Efficiency Runs. Losses and Separation Factors.

| | Run No. | | | | |
|---|---|---|---|---|---|
| | BMS-5 | BMS-6 | BMS-7 | Thorex-2 | Thorex-3 |
| Percent of Standard Flowrates | 100 | 75 | 150 | 100 | 200 |
| U-loss, AW, % | 0.0006 | 0.0029 | 0.0012 | 0.0011 | 0.0008 |
| U-loss, BT, % | | | | 0.181 | 0.068 |
| U-loss, CW, % | 0.0068 | 0.0003 | 0.0055 | 0.012 | |
| U-loss, total, % | 0.007 | 0.003 | 0.007 | 0.19 | |
| Th-loss, AW, % | | | | 0.057 | 0.073 |
| Th-loss, BU, % | | | | 0.277 | 0.480 |
| Th-loss, total, % | | | | 0.33 | 0.55 |
| SF, Th from U, B-bank | | | | 360 | 210 |
| SF, U from Th, B-bank | | | | 555 | 1460 |

Table 6. Purex Efficiency Runs. Overall Efficiencies.

| Run No. | Extraction | | | Stripping | | |
|---|---|---|---|---|---|---|
| | No. of Stages | | Efficiency | No. of Stages | | Efficiency |
| | Theo. | Actual | % | Theo. | Actual | % |
| BMS-5 | 3 | 4.65 | 65 | 6 | 7.8 | 77 |
| BMS-6 | 3 | 4.58 | 65 | 5 | 7.1 | 70 |
| BMS-7 | 3 | 4.49 | 67 | 5 | 9.0 | 56 |

## 5. CONCLUSIONS

The hydraulics of the modified CEN mixer-settlers could be adjusted for proper operation by changes in the diameter, speed, and height of the impellers. Further readjustments could be made with manipulators during hot cell operation.

Operating characteristics of the mixer-settler units appeared to be entirely satisfactory for use in hot cell tests. The capacity of the mixer-settlers was more than adequate. Throughputs of about 85 g of uranium and 30 g of thorium per hour were realized using the Purex and Acid Thorex flowsheets, respectively. The limiting total flows in the stripping (C) units were about 3 liters and 2 liters per hour for Purex and Acid Thorex. Eight extraction and eight partitioning stages, at about 67% efficiency, gave very low losses to the aqueous raffinates (AW) and good separation of uranium and thorium in the case of the Acid Thorex flowsheet. Although the ORNL Curium Recovery Facility has abandoned Homalite as a material of construction because of leaks caused by operation at elevated temperatures and degradation of the plastic by the concentrated (~11 M) chloride solutions used,[7] the Homalite has been satisfactory for dilute (<3 M) nitric acid solutions at ambient temperature used in the current studies. The cascade of mixer-settlers was therefore installed in the High Level Chemical Development Facility for studies on solvent degradation and for other experiments.

## 6. ACKNOWLEDGEMENTS

We wish to thank L. A. Byrd for his help during the Purex tests, and R. C. Shipwash and G. E. Woodall for their help during the Thorex runs. Also, the help of G. R. Wilson and his group of the Analytical Chemistry Division in analyzing the many samples is appreciated.

## 7. REFERENCES


1. H. W. Alter, et al., Miniature Mixer-Settler for Continuous Countercurrent Solvent Extraction, Anal. Chem., 26, 1357 (1954).

2. F. L. Culler, et al., Chem. Technol. Div. Annl. Rept. for Period Ending August 31, 1960, ORNL-2993 (Sept. 26, 1960).

3. F. L. Culler, et al., Chem. Technol. Div. Annl. Rept. for Period Ending May 31, 1961, ORNL-3153 (Sept. 11, 1961).

4. R. DeWitte, et al., The CEN Mixer-Settler for Liquid-Liquid Extraction, CEN-R-718 (1959), Centre d'Energie Nucleaire, Mol, Belgium.

5. F. L. Culler, et al., Chem. Technol. Div. Annl. Rept. for Period Ending June 30, 1962, ORNL-3314 (Sept. 14, 1962).

6. R. H. Rainey and J. G. Moore, Laboratory Development of the Acid Thorex Process for Recovery of Consolidated Edison Thorium Reactor Fuels, ORNL-3155 (April 27, 1962).

7. W. D. Burch (comp.), Transuranium Quarterly Progress Report for Period Ending August 31, 1964, ORNL-3739 (March 1965).

## DISTRIBUTION

### Internal

1. R. E. Blanco
2. R. E. Brooksbank
3. K. B. Brown
4. D. J. Crouse
5. F. L. Culler
6. F. L. Daley
7. D. E. Ferguson
8. L. M. Ferris
9. J. R. Flanary
10. H. E. Goeller
11-15. J. H. Goode
16. W. S. Groenier
17. R. E. Leuze
18. E. L. Nicholson
19. R. H. Rainey
20. J. T. Roberts
21. A. D. Ryon
22. V. C. A. Vaughen
23. M. E. Whatley
24. R. G. Wymer
25-26. C. R. Library
27. Doc. Ref. Sec.
28-29. Lab. Records
30. Lab. Records-RC

### External

31-33. Jorgen Klitgaard, Eurochemic, Mol, Belgium
34-35. E. M. Shank, Eurochemic, Mol, Belgium
36-50. DTIE
51. Research and Development Div, ORO