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STUDIES AND RESEARCH
CONCERNING BNFP
COPROCESSING TESTS WITH URANIUM ON
THE ELECTROPULSE COLUMN
and
PILOT-SCALE PULSE COLUMNS SIMULATING
THE SECOND AND THIRD PLUTONIUM PROCESS CYCLES

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November 1979

Work Performed Under Contract DE-AC09-78ET-35900

ALLIED-GENERAL NUCLEAR SERVICES
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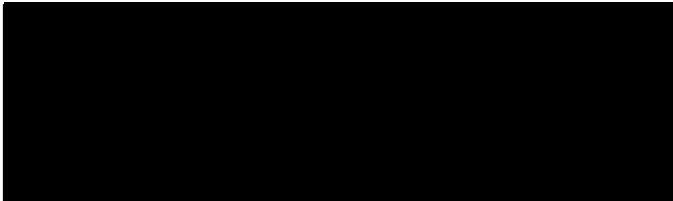


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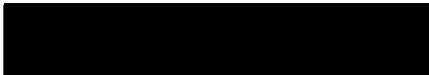
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PREPARED FOR THE
DEPARTMENT OF ENERGY
FUEL CYCLE PROJECT OFFICE
UNDER CONTRACT DE-AC09-78ET-35900

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ABSTRACT

Allied-General Nuclear Services (AGNS), under contract to the Department of Energy (DOE) during FY 1979, performed experimental studies and evaluations of the performance of the 1B Electropulse Column, the second plutonium cycle, and the third plutonium cycle under the proposed coprocessing flowsheet. Both pilot-plant and plant scale electropulse columns were tested for uranium electrolytic reduction efficiency. Both columns obtained the same efficiency which demonstrates that the design of the Electropulse Column is sufficient for the coprocessing flowsheet. Pilot plant scale tests demonstrated the proper functioning of the second and third plutonium cycles under coprocessing conditions. Comparisons of column uranium profile data with those predicted by the SEPHIS computer program show good agreement at the stream-end points, but the program does not accurately predict the column concentration profiles. The reason for this disagreement is believed to be continuous phase backmixing.

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1.0 INTRODUCTION

Allied-General Nuclear Services (AGNS), under contract to the Department of Energy during FY 1979, performed experimental studies and evaluations of the proposed coprocessing flowsheet. (1, 6) Coprocessing involves the retention of a portion of uranium with plutonium in the product stream of the plutonium cycles. During cold uranium runs in the plant during FY 1979, the coprocessing flowsheet conditions were tested on the 1B Electropulse Column and the second and third plutonium cycles. Supplemental testing was also performed on the pilot-scale equipment under the same conditions.

2.0 SUMMARY AND CONCLUSIONS

2.1 Electropulse Column Tests

Three runs were made on the plant 1B Electropulse Column to measure the uranium reduction efficiency under coprocessing flowsheet conditions. The reduction efficiency of these runs was determined to be in the range of 2.2% to 2.5%. This result was compared with similar measurements performed on the eight-inch diameter pilot-plant Electropulse Column. For the pilot-plant runs, the flow rate values were reduced seven times in line with the correspondingly smaller size of the pilot-plant equipment. The flow rate ratio (organic/aqueous) and the current density were the same as the plant column. The measured uranium reduction efficiency value from the pilot-plant runs was in the range of 2.3% to 2.5%, which is quite consistent with the data from the plant Electropulse Column tests (see Section 3.3).

A previously developed empirical relationship⁽⁵⁾ for estimating the uranium reduction efficiency was shown to yield good agreement with experimental values. Under conditions similar to the above experiments, the empirical relationship yielded a value of 2.5% (see Section 3.4).

The experimentally determined value of approximately 2.4% for the uranium reduction efficiency under coprocessing flowsheet conditions demonstrates the adequacy of the 1B Electropulse Column for reducing plutonium(IV) to the aqueous favoring plutonium(III) valence state. The results confirm the proper design of the column installed in the AGNS plant for operation under these conditions. Further, verification that the pilot-plant Electropulse equipment can reproduce results in the plant is quite valuable in any future pilot-plant experimental work aimed at studying the Electropulse Column behavior.

2.2 Second and Third Plutonium Cycle Tests

Four mass-transfer tests using uranium were performed on the pilot-plant equipment to simulate conditions in the second and third plutonium cycle under coprocessing conditions. These data were compared with results obtained from actual plant runs under the same conditions.

The pilot-plant experiments were designed to provide more flexibility and detailed information than that obtainable from the plant equipment. Whereas, data from the plant runs are restricted to the end-stream conditions, the pilot-plant column tests were used to obtain detailed column profile information. The scrub stream flow rates were varied to determine the effect on column profiles. The pilot-plant equipment consisted of two glass pulse columns (A and B). The A column was used to simulate the 2A or 3A columns for extraction and scrubbing. The B column was used to simulate the 2B and 3B columns for stripping.

In the pilot-plant runs, the second plutonium cycle was tested (i.e., 2A and 2B). In this case, the dual process A column was operated under

2A column conditions, and the B column under 2B column conditions. Two runs were made for different organic phase (2AX) and aqueous scrubbing stream (2AS) flow rates. Next, the third cycle (i.e., 3A and 3B) was tested on the pilot-plant equipment operating the A column under 3A column conditions, and the B column under 3B column conditions. Again, two runs were made under different flow conditions. In all cases, samples were taken along the A and B columns under steady-state conditions and analyzed for both the solutes content (uranium and HNO_3) and the phase volume ratio. From these data, the concentration profiles of solutes along each column were determined.

Results from the plant and pilot-plant testing were quite consistent with each other and demonstrated the adequacy of the second and third plutonium purification cycles. For example, the aqueous effluent stream from the pilot-plant A column (i.e., extraction column) contained a uranium concentration of less than 0.01 grams per liter in all cases.

Knowledge of the uranium concentration profiles from the four runs provided an opportunity to test the accuracy of the SEPHIS computer program (Richardson-Nance revised version). For process evaluations and inventory (accountability), it is often important to determine the solute concentration profile along the column under operational conditions. The usual analytical tool employed for these determinations is SEPHIS. Computer runs were made using SEPHIS with the same conditions (i.e., feed concentrations, flow conditions, etc.) as present in the pilot-plant tests. Comparison of results showed that whereas SEPHIS is capable of accurately predicting end-stream concentrations, the concentration profiles in the columns were not in agreement with the experimental results. This lack of accuracy with the SEPHIS column profile results is attributed to the fact that considerable backmixing of the continuous phase occurred within the columns, and the SEPHIS code is not designed to handle this type of phenomenon. On this basis, it is concluded that until a new (or improved) program for pulse columns is developed, concentration profile information along a column must be determined through pilot-plant experimental work.

3.0 ELECTROPULSE COLUMN TESTS

3.1 Summary

Uranium reduction efficiency under coprocessing flowsheet conditions was determined experimentally using both the plant 1B Electropulse Column and the eight-inch diameter pilot-scale Electropulse Column in the Engineering Laboratory. In the pilot-plant test, the actual organic phase from the plant (HSP and POR streams) was used as the organic feed. The aqueous feed for the run was prepared in the pilot plant by adding a concentrated uranyl nitrate solution to the actual 1BX stream obtained from the plant. The uranium was added to give a concentration, based on plant data from previous cold uranium runs, when the aqueous effluent stream leaving the 1B(X) column bottom section (feed stream to the Electropulse Column) was sampled and analyzed.^(2, 3)

Results from the plant Electropulse Column test and the pilot-scale test were compared with each other and with results derived from an empirical relationship. The adequacy of the Electropulse system under coprocessing flowsheet conditions was then confirmed.

3.2 Pilot-Plant Electropulse Column Description

The 8-inch (200-millimeter) diameter pilot-plant Electropulse Column is described in detail in Reference 6. It consists of a vented column head, column working section, and bottom disengaging section connected to a bellows-type pulser (see Figures 3-1 and 3-2). The column shell (body) is made of Plexiglas. A 3-inch (75-millimeter) diameter Plexiglas tube, centered within the column head, forms the upper part of the anode chamber. A 3-inch (75-millimeter) diameter and 60-inch (1500-millimeter) long porous ceramic tube (diaphragm), centered within the column working section, is attached to the bottom of the above Plexiglas tube and forms the lower part of the internal anode chamber. The top part of the anode chamber ends with an 8-inch (200-millimeter) diameter gas separator made of Plexiglas. A cylindrical platinum screen, 2.5 inches (62.5 millimeters) in diameter and 58 inches (1450 millimeters) long, is the anode located within the diaphragm. The external cathode chamber, i.e., the annular space between the central ceramic diaphragm and column wall, is provided with 44 titanium cathode sieve plates (of annular shape) at a plate spacing of 1.25 inches (31 millimeters). The pilot-scale Electropulse Column is seven times smaller than the plant-size 1B Electropulse Column with regard to the cathode chamber cross-sectional area and the number of cylindrical anode chambers.^(4, 5) The electroactive part (column working section) of both columns is of the same height.

3.3 Experimental Results

3.3.1 The Pilot-Plant Electropulse Column

The average flow rate of the organic feed to the plant Electropulse Column during coprocessing tests was approximately 2890 liters/hour, and the aqueous feed flow rate was approximately 538 liters/hour. These flow rate values were reduced seven times for the electrolytic test run in the pilot-plant Electropulse Column (see Tables 3-1 and 3-2). The flow rate ratio (O/A \approx 5.37), as well as the current density (0.0135 A/cm²), were the same as in the plant column. The pilot-plant unit operated with a pulse frequency of $f = 57$ cycles/minute and pulse amplitude of $A_0 = 1.2$ centimeters, i.e., about 20% below the upper flooding curve.⁽⁶⁾ Run duration was approximately 145 minutes. After reaching steady state (in about 100 minutes), samples of both the organic and aqueous effluent streams were taken about 120, 130, and 140 minutes after starting the electrolytic process (three sets of samples). The samples were analyzed for uranium(VI), uranium (IV), and nitric acid content. All data are summarized in Table 3-1. From analytical data and flow rates, the uranium reduction efficiency was calculated for each set of samples.

Example: The first set of samples.

- Uranium(IV) production rate:

Organic:	6.88 x 0.61	\approx 4.19 grams uranium(IV)/minute
Aqueous:	1.31 x 6.13	\approx 8.03 grams uranium(IV)/minute
Total:		<u>12.22 grams uranium(IV)/minute</u>

- Uranium(VI) feed rate:

Organic:	6.88 x 68.65	\approx 472.31 grams uranium(VI)/minute
Aqueous:	1.28 x 14.3	\approx 18.30 grams uranium(VI)/minute
Total:		<u>490.61 grams uranium(VI)/minute</u>

- Uranium reduction efficiency:

$$R(U) = \frac{\text{Total Uranium (IV)}}{\text{Total Uranium (VI)}} \times 100 = \frac{1222}{490.61} \approx \underline{\underline{2.5\%}}$$

In the same manner, the reduction efficiency was calculated for the second and third set of samples yielding values of 2.3 and 2.5, respectively. Results for three runs are shown in Table 3-1.

3.3.2 Plant 1B Electropulse Column

Three runs were made on the plant Electropulse Column (during cold uranium runs) under approximately the same conditions as in the test performed on the pilot-plant Electropulse Column. Plant data are shown in Table 3-2. During the last cold uranium run, the organic effluent stream (1BU) from the 1B(X) column was not analyzed for uranium(IV). However, it can be assumed from experience that uranium(IV)

concentration in the stream would be about the same as during previous cold uranium runs, i.e., approximately 0.25 to 0.3 grams uranium(IV)/liter. The uranium reduction efficiency was calculated for each electrolytic run shown in Table 3-2.

Example: Run No. 1.

- Uranium(IV) production rate:

Organic:	48.16 x 0.3	≈ 14.44	grams uranium(IV)/minute
Aqueous:	9.31 x 6.2	≈ 57.72	grams uranium(IV)/minute
Total:		<u>72.16</u>	grams uranium(IV)/minute

- Uranium(VI) feed rate:

Organic: 48.16 x 60 ≈ 2889.6 grams uranium(VI)/minute

- Uranium reduction efficiency:

$$R_{(U)} = \frac{72.16}{2889.6} \times 100 \approx \underline{\underline{2.5\%}}$$

The uranium reduction efficiency for the second and third runs was calculated in the same manner, yielding values of 2.5 and 2.2, respectively. These values are quite consistent with the values obtained on the pilot-scale equipment. Results from the plant tests are shown in Table 3-2.

3.4 Empirical Relationship

The uranium reduction efficiency obtained during test runs under coprocessing flowsheet⁽¹⁾ conditions on the plant-size, as well as on pilot-plant Electropulse Column, is about $R_{(U)} \approx 2.4\%$. This was expected because the plant-size column design is based on the pilot-plant column concept and experience.^(4, 5) Both the pilot-plant and plant-size column were designed and scaled up by use of the empirical equation developed during previous research work on Electropulse Columns.⁽⁵⁾ The equation, used for scale-up, has the form:

$$R_{(U)} = 0.000116 \frac{S_c \cdot f \cdot A_o}{L_{(o)}} (1 + P_a) \cdot (\log i + 2.545) \cdot C_{(o)} \exp \left[\frac{0.883}{C_{(a)}} \right] \quad (1)$$

where:

$R_{(U)}$ = Uranium reduction efficiency (%)

$C_{(o)}$ = Uranium(VI) concentration in the organic feed (grams/liter)

$C_{(a)}$ = Nitric acid concentration in the aqueous feed (M)

S_c = Cathode surface area (cm²)

$L(o)$ = Organic feed flow rate (milliliters/minute)

P_a = Flow rate ratio (= aqueous/organic)

f = Pulse frequency (cycles/minute)

A_o = Pulse amplitude (centimeters/cycle)

i = Cathode current density (A/cm^2)

By use of this equation and data from Tables 3-1 and 3-2, respectively, the uranium reduction efficiency can be calculated (predicted) within the correlation accuracy range of $\pm 6\%$.⁽⁵⁾

Example: Plant-size Electropulse Column (from Table 3-2, Run No. 2).

$C(o)$ \approx 69 grams uranium(VI)/liter

$C(a)$ \approx 2.15 M HNO_3

S_c \approx 190,000 cm^2 (from design)

f \approx 60 cycles/minute

$A(o)$ \approx 1.10 centimeters

$L(o)$ \approx 48,160 milliliters/minute

P_a \approx 0.186

i \approx 0.0135 A/cm^2

Substituting the above values in the empirical equation, the uranium reduction efficiency (predicted) will be:

$$R(U) = 0.000116 \times \frac{1.9 \times 10^5 \times 60 \times 1.1}{4.816 \times 10^4} \times 1.186 \times (\log 0.0135 + 2.545) \times 69 \times \exp \left[\frac{0.883}{2.15} \right] \approx \underline{\underline{2.5\%}}$$

which is very close to the values obtained experimentally on both columns.

3.5 Conclusions

The experimentally determined value of approximately 2.4% for the uranium reduction efficiency under coprocessing flowsheet conditions demonstrates the adequacy of the 1B Electropulse Column for reducing plutonium(IV) to the aqueous favoring plutonium(III) valence state. The results confirm the proper design of the Electropulse Column installed in the AGNS plant for operation under these conditions. Consistent results were obtained between the plant Electropulse Column and the pilot-plant equipment with regard to the uranium reduction efficiency.

Verification that the pilot-plant equipment can accurately reproduce the results in the plant column is quite valuable in any future experimental work aimed at studying the Electropulse Column behavior. Finally, it was shown that the uranium reduction efficiency can be accurately predicted using the empirical relationship of Equation (1) within the correlation accuracy range of $\pm 6\%$.

For further flowsheet studies, it is recommended to use the empirical equation⁽⁵⁾ in Section 3.3 for prediction of the uranium reduction, and the data from Reference (6) for prediction of the uranium to plutonium weight ratio in the LBP stream. This would then be followed by experimental verification of data on the pilot-plant equipment.

TABLE 3-1

PILOT-PLANT ELECTROPULSE COLUMN DATA

(Coproducting Test)

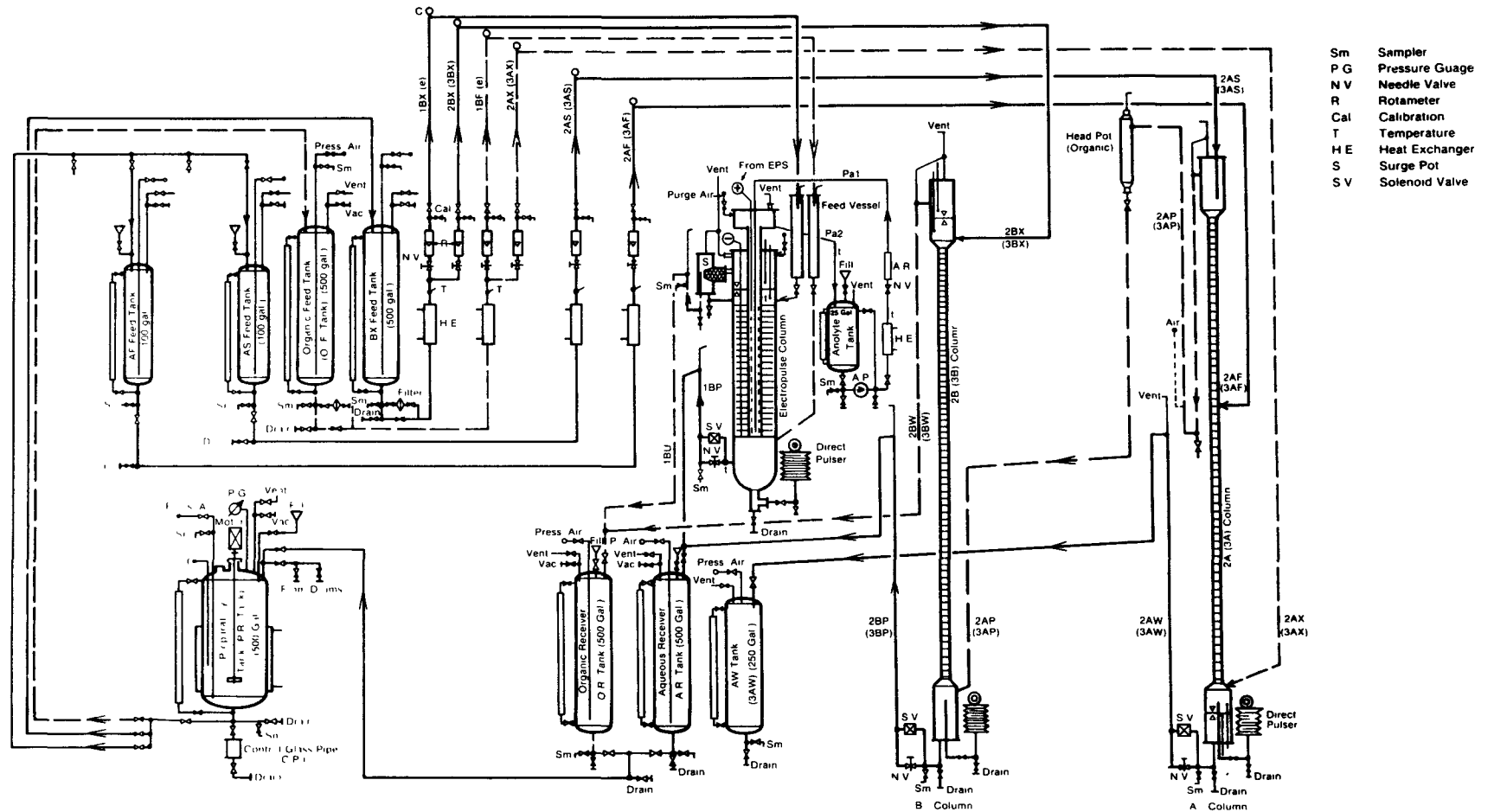
Organic Flow Rate:	(ℓ/min)	6.88		
Aqueous Flow Rate:	(ℓ/min)	1.28		
Pulse Frequency:	(c/min)	57		
Pulse Amplitude:	(cm)	1.2		
Current Input:	(A)	365		
Current Density:	(A/cm^2)	~ 0.0135		
Voltage Drop:	(V)	4.05		
Temperature:	($^{\circ}\text{C}$)	~ 30		
Run Duration:	(min)	144		
Organic Feed Concentration:				
Uranium(VI):	(g/ℓ)	68.65		
HNO_3 :	(M)	0.24		
Aqueous Feed Concentration:				
Uranium(VI):	(g/ℓ)	14.3		
HNO_3 :	(M)	2.15		
N_2H_4 :	(M)	0.14		
Sample Number:		No. 1	No. 2	No. 3
Sampling Time:	(min)	120	130	140
Organic Effluent Stream Concentration:				
Uranium(VI):	(g/ℓ)	66.97	66.12	67.44
Uranium(IV):	(g/ℓ)	0.61	0.59	0.65
HNO_3 :	(M)	0.22	0.25	0.26
Aqueous Effluent Stream Concentration:				
Uranium(VI):	(g/ℓ)	12.33	12.93	12.78
Uranium(IV):	(g/ℓ)	6.13	5.68	6.04
HNO_3 :	(M)	2.13	2.12	2.14
N_2H_4 :	(M)	0.14	0.14	0.14
Anolyte:				
Rate:	(ℓ/min)	6.0	6.0	6.0
HNO_3 :	(M)	2.05	2.05	2.05
Seepage:	(ℓ/min)	~ 0.03	0.03	0.03
Uranium Reduction Efficiency:				
$R_{(\text{U})}$:	(%)	2.5	2.3	2.5

TABLE 3-2

PLANT 1B ELECTROPULSE COLUMN DATA

(Coprocesing Test)

Organic Flow Rate:	(ℓ /min)	48.16		
Aqueous Flow Rate:	(ℓ /min)	8.97		
Pulse Frequency:	(c/min)	60		
Pulse Amplitude:	(cm)	~1.10		
Current Input:	(A)	2560		
Current Density:	(A/cm ²)	~0.0135		
Voltage Drop:	(V)	4.10		
Temperature:	(°C)	~30		
Run Number:		No. 1	No. 2	No. 3
Organic Feed Concentration:				
Uranium(VI):	(g/ ℓ)	~60	69	69
HNO ₃ :	(<u>M</u>)	0.24	0.24	0.24
Aqueous Feed Concentration:				
Uranium(VI):	(g/ ℓ)	0	0	0
HNO ₃ :	(<u>M</u>)	2.15	2.15	2.2
N ₂ H ₄ :	(<u>M</u>)	0.23	0.20	0.19
Organic Effluent Stream Concentration:				
Uranium(VI):	(g/ ℓ)	56.4	64	64
Uranium(IV):	(g/ ℓ)	~0.3	0.3	0.3
HNO ₃ :	(<u>M</u>)	0.21	0.22	0.22
Aqueous Effluent Stream Concentration:				
Uranium(VI):	(g/ ℓ)	17.9	25.2	25.8
Uranium(IV):	(g/ ℓ)	6.2	7.5	6.3
HNO ₃ :	(<u>M</u>)	2.0	1.9	2.1
N ₂ H ₄ :	(<u>M</u>)	0.21	0.19	0.17
Anolyte:				
Rate:	(ℓ /min)	~42	42	42
HNO ₃ :	(<u>M</u>)	2.0	2.0	2.1
Seepage:	(ℓ /min)	~0.34	0.34	0.34
Uranium Reduction Efficiency:				
R :	(%)	2.5	2.5	2.2



PILOT-PLANT FLOW DIAGRAM

FIGURE 3-1

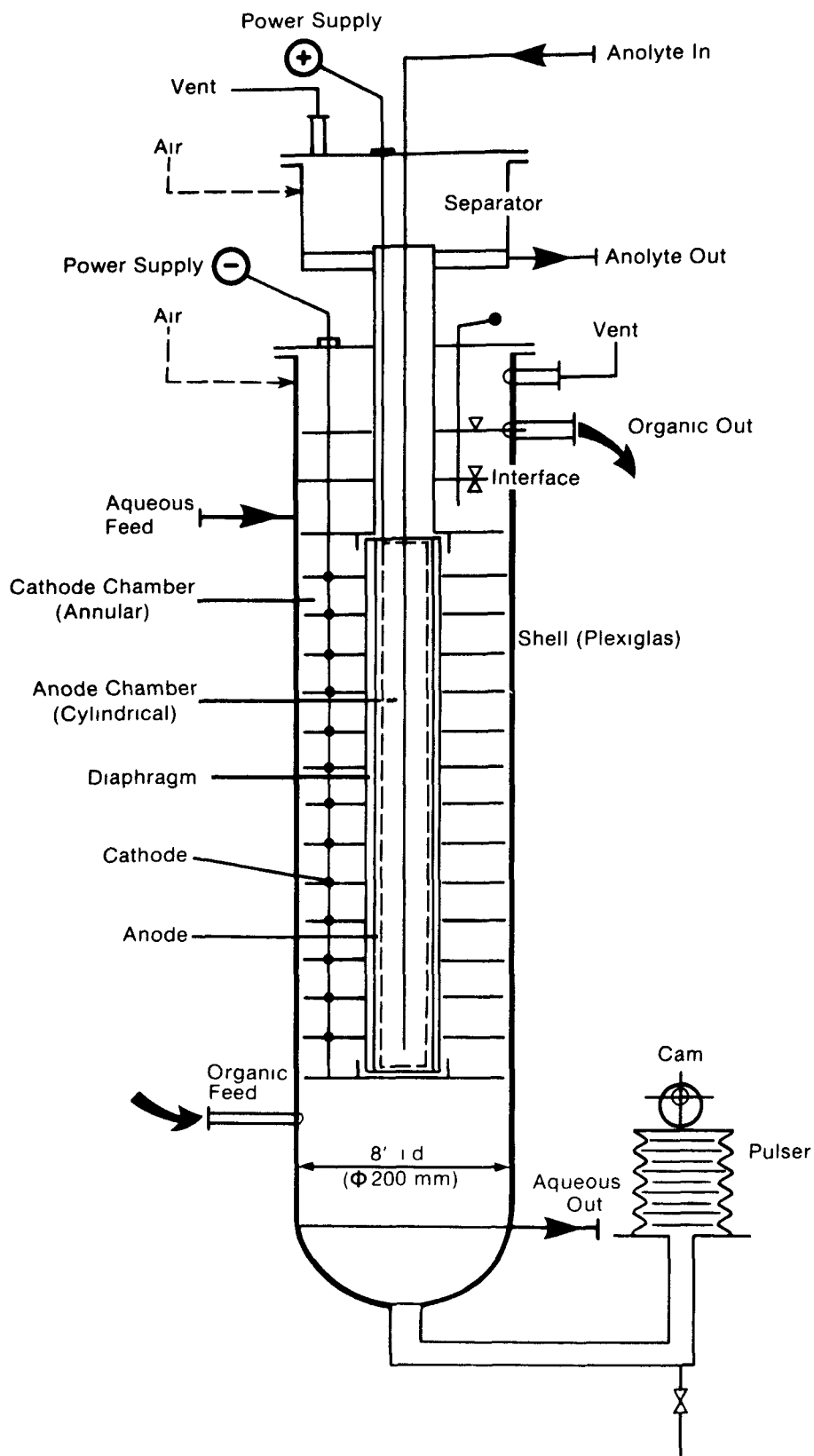


FIGURE 3-2

PILOT-PLANT ELECTROPULSE COLUMN

4.0 SECOND AND THIRD PLUTONIUM CYCLE TESTS (WITH URANIUM)

4.1 Summary

Four mass-transfer tests using uranium only were performed on the pilot-plant equipment (shown in Figure 3-1) to simulate conditions in the second and third plutonium cycles in the plant under coprocessing conditions. These tests supplement data obtained from plant tests on the second and third plutonium cycle. Runs on the pilot-plant equipment are particularly valuable in that they provide a convenient means to optimize flowsheet conditions for coprocessing and offer a better understanding of column behavior through detailed information on the processes within the columns.

Pilot-plant test results were compared to analytical predictions from the SEPHIS computer program. For process evaluations and inventory (accountability), it is essential to determine the solute concentration profile along the column under given operational conditions. To gain insight into the accuracy of SEPHIS for this purpose, comparisons were made between the uranium concentration column profiles from the pilot-plant tests and those predicted by the SEPHIS program. These results demonstrated that SEPHIS is capable of predicting endstream concentrations; however, concentration profiles in the columns were not in agreement with experimental results. This disagreement can be attributed to the considerable backmixing in the continuous phase which SEPHIS is not designed to handle.

4.2 Pilot-Plant Equipment Description

The experimental equipment (Figure 3-1) consisted of two glass pulse columns (A and B columns) and the supporting equipment such as stainless steel tanks, heat exchangers, and flowmeters. The coextraction column (A column) for extraction and scrubbing, and stripping column (B column) were 2 inches (50 millimeters) in diameter. The overall height of the A column (2A or 3A column) was 26.5 feet (8.15 meters) with a working extraction section of 13 feet (3.96 meters) (lower section) and scrubbing section of 9.3 feet (2.8 meters). The B column (2B or 3B column) for stripping was 24 feet (7.2 meters) high, with a working section of 19 feet (5.7 meters). Both the A column and B column were provided with stainless steel nozzle plates consisting of 23% free surface area, 1/8-inch (3.2 millimeter) orifice size, and 1/16-inch (1.6 millimeter) plate thickness. The plates were assembled on a central 1/4-inch (6 millimeter) diameter tie rod on a 2-inch (50-millimeter) plate spacing. Each column was provided with a 6-inch (150-millimeter) diameter top and bottom disengaging section made of glass. The top disengaging section was vented. The bottom disengaging section was connected to a bellows-type pulser. The interface in each column was controlled automatically by two titanium conductivity probes regulating a stainless steel (air operated) control valve at the aqueous phase outlet line. The interface was controlled on the A column in the

bottom disengaging section and on the B column in the top disengaging section.

The A column was provided with samplers at 14 different locations along the column, and the B column at 6 different locations. At each sampling point, both the continuous and dispersed phases were sampled. Both the aqueous and organic phase samplers were made of a stainless steel tube with an inside diameter of about 1/8 inch (3 millimeters). The organic sampler protruded inside the column with a tubular end piece made of teflon (preferably wetted by the organic phase); the aqueous phase sampler had a similar tube made of stainless steel (wetted by aqueous phase). See Figure 4-1 for details.

The feed preparation tank was a 500-gallon stainless steel cylindrical vessel provided with a mechanical agitator, cooling jacket, sight glass, temperature and pressure instrumentation, and connections for liquid inlet and outlet, pressurized air inlet, vacuum, vent, and sampling. All feed tanks and receiving tanks were equipped with sight glasses, liquid inlet and outlet piping, pressurized air inlets, vents, vacuum, and samplers. The tank volumes are shown in Figure 3-1. Feed flow rate was controlled by a calibrated flowmeter.

4.3 Experimental Results

Mass-transfer in both the second and third plutonium cycles, under coprocessing flowsheet(1) conditions, was simulated with uranium on the pilot-plant equipment shown in Figure 3-1. For the second plutonium cycle, the dual process A column operated under 2A column operating and flowsheet conditions, and B column under 2B column conditions. Two runs were performed (Runs U-1 and U-2) for different organic phase (2AX) and aqueous scrubbing stream (2AS) flow rates (see Tables 4-1 and 4-2). For the third plutonium cycle, the A column operated under 3A column conditions, and B column under 3B column conditions. Again, two runs were performed (Runs U-3 and U-4) for different organic phase (3AX) and aqueous scrubbing stream (3AS) flow rates (see Tables 4-3 and 4-4 for details). In each run, after establishing steady-state conditions (in about five to six hours), samples were taken along the A and B column (including effluent stream samples) and analyzed for both the solutes content (uranium, HNO_3) and phase volume ratio in the samples. From these data, the concentration profile of solutes in question along each column was determined by evaluating the actual solute concentration (in both phases) at each sampling point by use of the following method:

- (1) If the sample of the continuous phase does not contain dispersed phase, the following equation applies:

$$C_d = C_{d_s} + \frac{V_c}{V_d} \cdot (C_{c_s} - C_c) \quad (2)$$

where:

C_d is the solute's actual concentration in the dispersed phase (grams/liter), (M)

C_{d_s} is the solute's concentration in the dispersed phase of the sample (grams/liter), (M)

C_{c_s} is the solute's concentration in the continuous phase of the sample (grams/liter), (M)

C_c is the solute's actual concentration in the continuous phase (in continuous phase sample), (grams/liter), (M)

$\frac{V_c}{V_d}$ is the ratio of the continuous to the dispersed volume in the sample.

- (2) If both samples [(1) and (2)] contain the continuous as well as dispersed phase, the equations which were used are as follows:

$$C_c = \frac{C_{d_s}^{(1)} - C_{d_s}^{(2)} + \left(\frac{V_c}{V_d}\right)^{(1)} \cdot C_{c_s}^{(1)} - \left(\frac{V_c}{V_d}\right)^{(2)} \cdot C_{c_s}^{(2)}}{\left(\frac{V_c}{V_d}\right)^{(1)} - \left(\frac{V_c}{V_d}\right)^{(2)}} \quad (3)$$

and

$$C_d = C_{d_s}^{(1)} + \left(\frac{V_c}{V_d}\right)^{(1)} \cdot (C_{c_s}^{(1)} - C_c) \quad (4)$$

or

$$C_d = C_{d_s}^{(2)} + \left(\frac{V_c}{V_d}\right)^{(2)} \cdot (C_{c_s}^{(2)} - C_c) \quad (5)$$

Prior to using the equations, the solute concentration in the sample, determined analytically, must be rechecked and adjusted (if necessary) with respect to correct distribution coefficient values.

Example: 2A column, sampling point A-2.

$$C_{d_s}^{(1)} = 27.98 \text{ grams uranium/liter}$$

$$C_{d_s}^{(2)} = 26.44 \text{ grams uranium/liter}$$

$$C_{c_s}^{(1)} = 72.44 \text{ grams uranium/liter}$$

$$C_{c_s}^{(2)} = 72.46 \text{ grams uranium/liter}$$

$$\left(\frac{v_c}{v_d}\right)^{(1)} = 20.5$$

$$\left(\frac{v_c}{v_d}\right)^{(2)} = 17.4$$

$$C_c = C_o \approx \underline{72.8} \text{ grams uranium/liter}$$

$$C_d = C_a \approx 20.6 \text{ grams uranium/liter}$$

Using the above procedure, the uranium and nitric acid concentrations along the columns were determined. The results from the four runs are contained in Tables 4-5 through 4-8. Figure 4-2 shows the concentration profile of uranium in both the aqueous and organic phase along the 2A column (Runs U-1 and U-2) and 3A column (Runs U-3 and U-4).

Figures 4-3 through 4-10 show the column operating diagrams for the runs. There are a total of eight such diagrams, corresponding to the four runs and two columns per run. The flooding curves of both the dual process A column and stripping B column were determined previously.⁽⁷⁾ These flooding curves were verified (rechecked in three points).

As may be seen from Tables 4-1 through 4-4 and Figure 4-2, the aqueous effluent stream from A column (2AW or 3AW stream) during all four runs contained less than 0.01 grams per liter of uranium. This demonstrates that proper uranium extraction is taking place. Similarly, the results in these tables show that the B column provides the desired stripping function.

Results obtained on the second and third plutonium cycles of the plant during (coprocessing) cold uranium runs (Tables 4-9 and 4-10) can be compared with data determined experimentally on the pilot-plant equipment (Tables 4-1 through 4-4).

4.4 Comparison of Pilot-Plant and SEPHIS Results

Concentration profiles obtained experimentally for each column can be compared with that computed by the SEPHIS code. For the SEPHIS calculations, the number of stages for the program can be obtained from the column operating diagram. The same feed concentration and flow conditions are assumed as in the experimental runs.

Figures 4-11, 4-12, and 4-13 show the comparison of the A column concentration profiles predicted by the SEPHIS code (Richardson-Nance Revised Version) with the actual (experimental) profiles under the same run conditions (see Tables 4-1 through 4-3) and same number of stages. Figures 4-14, 4-15, and 4-16 represent the comparison of the experimental B column concentration profiles with the profiles predicted by SEPHIS program (code).

Resultant data and graphs indicate backmixing of the continuous phase in the dual process A column (organic phase), and in the stripping B column (aqueous phase). This can be seen by comparing the operating line for plug flow with the experimental operating curve in the corresponding operating diagram. In a pulse column, which is a differentially operating contactor, this result was expected.

Comparison of the A column experimental concentration profiles with those predicted by SEPHIS program, shown in Figures 4-11, 4-12 and 4-13, indicates that uranium concentrations obtained experimentally are lower in both phases along the column scrubbing section and higher in the organic phase along the extraction section than predicted by the SEPHIS program. However, the end stream concentrations are about the same.

Comparison of the actual (experimental) B column concentration profiles with profiles predicted by the SEPHIS program shows that, although the end-stream concentrations are about the same, SEPHIS predicts a higher uranium concentration along the stripping B column than determined experimentally (see Figures 4-14, 4-15, and 4-16).

During Run U-4, the 3A column was sampled under temporarily unstable operating conditions, most probably due to fluctuations in the feed streams flow rate. The experimental operating curve in the column scrubbing section (Figure 4-15) indicates a temporary decrease in the organic phase flow rate during sampling of the upper column section.

4.5 Conclusions

Resultant end-stream concentrations from uranium tests performed on pilot-plant equipment simulating the process in the second (2A and 2B columns) and third (3A and 3B columns) plutonium cycles under coprocessing flowsheet⁽¹⁾ conditions correlate well with those obtained on plant columns. Data from both sets of tests demonstrate the adequacy of the second and third plutonium cycles in the plant under coprocessing flowsheet conditions.

It was shown that the SEPHIS computer program is accurate in predicting end-stream concentrations, but the program does not correctly describe the concentration profile of the solutes in question due to backmixing effect on the process within the pulse column. Until a new (or improved) program for pulse columns is developed, concentration profiles along the column must be determined through pilot-plant experimental work.

TABLE 4-1

RUN U-1 CONDITIONS AND DATA

Stream:	2AS	2AF	2AX	2AP (2BF)	2AW	2BX	2BP	2BW
Phase:	A	A	O	O	A	A	A	O
Flow Rate: (ml/min)	113.5	650	218	~218	~763.5	235	~235	~218
Uranium: (g/l)	-----	21.31	-----	63.52	<0.01	-----	42.1	18.1
HNO ₃ : (M)	1.0	2.94	-----	0.11	2.62	0.29	0.36	0.3
Simulated Plant Pulse Column:	2A Column					2B Column		
Temperature: (°C)	~25					~25		
Pulse Frequency: (c/min)	85					76		
Pulse Amplitude: (cm)	2.0					2.0		
Run Duration: (hrs)	~11					~11		
Dispersed Phase Holdup: (8) (%)	Scrub: ~16% Extr: ~13% [From Equations in Reference (8)]					~15%		

TABLE 4-2

RUN U-2 CONDITIONS AND DATA

Stream:	2AS	2AF	2AX	2AP (2BF)	2AW	2BX	2BP	2BW
Phase:	A	A	O	O	A	A	A	O
Flow Rate: (ml/min)	67	650	257.8	~257.8	~717	244.6	244.6	257.8
Uranium: (g/l)	-----	21.31	-----	53.71	<0.01	-----	34.45	21.0
HNO ₃ : (M)	1.0	2.94	-----	0.11	2.66	0.29	0.393	0.035
Simulated Plant Pulse Column:	2A Column					2B Column		
Temperature: (°C)	~25					~25		
Pulse Frequency: (c/min)	86					76		
Pulse Amplitude: (cm)	2.0					2.0		
Run Duration: (hrs)	~8					~8		
Dispersed Phase Holdup: (8) (%)	Scrub: ~11% Extr: ~15% [From Equations in Reference (8)]					~15%		

TABLE 4-3

RUN U-3 CONDITIONS AND DATA

Stream:	3AS	3AF	3AX	3AP (3BF)	3AW	3BX	3BP	3BW
Phase:	A	A	O	O	A	A	A	O
Flow Rate: (ml/min)	112	510	301	~301	~622	340	~340	301
Uranium: (g/l)	-----	41.79	-----	70.72	<0.01	-----	49.82	14.44
HNO ₃ : (M)	1.02	3.08	-----	0.103	2.66	0.2	0.26	0.035
Simulated Plant Pulse Column:	3A Column					3B Column		
Temperature: (°C)	-25					-25		
Pulse Frequency: (c/min)	87					75		
Pulse Amplitude: (cm)	2.0					2.0		
Run Duration: (hrs)	~10					~10		
Dispersed Phase Holdup: (8) (%)	Scrub: ~ 14.0% Extr: ~ 17.5% [From Equations in Reference (8)]					~14.5%		

TABLE 4-4

RUN U-4 CONDITIONS AND DATA

Stream:	3AS	3AF	3AX	3AP (3BF)	3AW	3BX	3BP	3BW
Phase:	A	A	0	0	A	A	A	0
Flow Rate: (ml/min)	100	510	398	~398	~610	336	~336	~396
Uranium: (g/l)	-----	41.79	-----	53.3	<0.01	-----	38.1	21.1
HNO ₃ : (M)	1.02	3.02	-----	0.15	2.6	0.2	0.315	0.06
Simulated Plant Pulse Column:	3A Column					3B Column		
Temperature: (°C)	~25					~25		
Pulse Frequency: (c/min)	86					73		
Pulse Amplitude: (cm)	2.0					2.0		
Run Duration: (hrs)	~9					~9		
Dispersed Phase Holdup: (8) (%)	Scrub: ~11% Extr: ~19%					~13,5%		
	[From Equations in Reference (8)]							

TABLE 4-5

CONCENTRATION ALONG THE 2A AND 2B COLUMNS (RUN U-1)

Column:		2A Column:				2B Column:		2B Column:			
Sample:	Stream:	Aqueous		Organic		Sample:	Stream:	Aqueous		Organic	
		U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)			U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)
End	2AS	2AS	2AS	2AP	2AP	End	2BX	2BX	2BW	2BW	
Stream:	0	1.0	63.52	0.11	0.11	Stream:	0	0.29	18.1	0.03	
A-1	~4.03	~1.0	~65.33	~0.10	~0.10	B-1	~7.3	~0.3	~18.5	~0.03	
A-2	20.6	1.0	72.8	0.11	0.11	B-2	20.2	0.3	35.1	0.03	
A-3	25.0	1.0	74.39	0.11	0.11	B-3	29.7	0.3	48.5	0.03	
A-4	24.0	1.03	73.33	0.10	0.10	B-4	33.3	0.3	51.8	0.03	
A-5	19.6	1.07	72.93	0.137	0.137	B-5	40.7	0.3	62.0	0.04	
A-6	16.1	1.34	70.13	0.27	0.27	B-6	41.7	0.33	62.8	0.05	
A-7	15.1	2.75	67.57	0.44	0.44	End	2BP	2BP	2AP	2AP	
A-8	4.05	2.76	24.4	0.52	0.52	Stream:	42.1	0.36	63.52	0.11	
A-9	1.22	2.78	4.0	0.59	0.59						
A-10	0.33	2.78	2.12	0.61	0.61						
A-11	0.05	2.79	1.14	0.62	0.62						
A-12	0.02	2.73	0.8	0.61	0.61						
A-13	<0.01	2.73	0.07	0.6	0.6						
A-14	<0.01	2.64	0.02	0.52	0.52						
End	2AW	2AW	2AX	2AX	2AX						
Stream:	<0.01	2.62	0	0	0						

TABLE 4-6

CONCENTRATION ALONG THE 2A AND 2B COLUMNS (RUN U-2)

Column:		2A Column:				2B Column:			
Sample:	Aqueous		Organic		Sample:	Aqueous		Organic	
	U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)		U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)
End Stream:	2AS 0	2AS 1.0	2AP 53.71	2AP 0.11	End Stream:	2BX 0	2BX 0.29	2BW 21.2	2BW 0.035
A-1	~7.71	~0.93	~56.18	0.11	B-1	~9.6	~0.3	~23.8	~0.04
A-2	12.7	0.97	59.0	0.12	B-2	20.7	0.3	38.3	0.04
A-3	15.3	1.16	64.3	0.12	B-3	26.5	0.3	43.6	0.04
A-4	16.8	1.18	65.7	0.13	B-4	--	--	--	--
A-5	18.84	1.31	71.1	0.21	B-5	33.0	0.3	52.7	0.04
A-6	20.1	1.87	70.2	0.26	B-6	34.3	0.39	53.1	0.043
A-7	19.9	2.60	69.0	0.37	End Stream:	2BP 34.45	2BP 0.393	2AP 53.71	2AP 0.11
A-8	7.03	2.68	25.05	0.48					
A-9	1.4	2.80	4.0	0.52					
A-10	0.33	2.88	2.03	0.56					
A-11	0.05	2.8	1.20	0.56					
A-12	0.02	2.8	0.08	0.55					
A-13	0.01	2.78	0.07	0.51					
A-14	<0.01	2.70	0.02	0.4					
End Stream:	2AW <0.01	2AW 2.66	2AX 0	2AX 0					

TABLE 4-7

CONCENTRATION ALONG THE 3A AND 3B COLUMNS (RUN U-3)

Column:		3A Column:				3B Column:		3B Column:			
Sample:	End Stream:	Aqueous		Organic		Sample:	End Stream:	Aqueous		Organic	
		U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)			U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)
		3AS	3AS	3AP	3AP			3BX	3BX	3BW	3BW
		0	1.02	70.72	0.103			0	0.20	14.44	0.035
A-1		~9.41	~1.10	~73.5	~0.10	B-1		~3.9	~0.20	~15.02	~0.03
A-2		29.3	1.0	81.0	0.1	B-2		13.5	0.2	25.10	0.03
A-3		35.6	1.2	83.1	0.11	B-3		31.0	0.2	47.90	0.03
A-4		38.72	1.21	84.2	0.11	B-4		45.5	0.21	65.2	0.03
A-5		40.05	1.53	85.4	0.15	B-5		--	--	--	--
A-6		39.8	1.94	84.6	0.17	B-6		49.3	0.24	70.4	0.04
A-7		37.3	2.63	83.2	0.17	End Stream:		3BP	3BP	3AP	3AP
								49.82	0.26	70.72	0.103
A-8		17.62	2.71	44.51	0.2						
A-9		3.14	2.80	9.06	0.40						
A-10		0.61	2.82	1.90	0.50						
A-11		0.12	2.82	0.9	0.56						
A-12		0.02	2.80	0.1	0.51						
A-13		<0.01	2.78	0.05	0.46						
A-14		<0.01	2.71	0.01	0.38						
End Stream:		3AW	3AW	3AX	3AX						
		<0.01	2.66	0	0						

TABLE 4-8

CONCENTRATION ALONG THE 3A AND 3B COLUMNS (RUN U-4)

Column:	3A Column:				3B Column:				
Sample:	Aqueous		Organic		Sample:	Aqueous		Organic	
	U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)		U (g/l)	HNO ₃ (M)	U (g/l)	HNO ₃ (M)
End Stream:	3AS 0	3AS 1.02	3AP 53.30	3AP 0.10	End Stream:	3BX 0	3BX 0.20	3BW ~21.1	3BW ~0.06
A-1	~3.72	~1.10	~53.5	~0.105	B-1	~6.22	0.20	21.5	0.06
A-2	8.65	1.21	56.65	0.12	B-2	20.3	0.18	35.3	0.06
A-3	10.4	1.20	55.1	0.125	B-3	--	--	--	--
A-4	11.3	1.53	56.4	0.153	B-4	--	--	--	--
A-5	11.52	1.75	62.2	0.21	B-5	36.6	0.24	52.5	0.07
A-6	17.0	2.21	65.4	0.24	B-6	37.82	0.29	53.0	0.09
A-7	17.73	2.76	62.3	0.37	End Stream:	3BP 38.1	3BP 0.315	3AP 53.3	3AP 0.15
A-8	4.3	2.91	24.1	0.48					
A-9	1.34	2.9	4.15	0.56					
A-10	0.40	2.9	2.3	0.62					
A-11	0.06	2.83	1.10	0.62					
A-12	0.02	2.82	0.1	0.60					
A-13	<0.01	2.8	0.09	0.56					
A-14	<0.01	2.7	0.02	0.45					
Stream:	3AW <0.01	3AW 2.6	3AX 0	3AX 0					

TABLE 4-9

2A AND 2B PLANT COLUMN DATA

Stream:		2AS	2AF	2AX	2AP	2AW	2BX	2BP	2BW
Plant Actual Data	Flow Rate: (ℓ/hr)	~103.4	612.3	244.7	~244.7	~715.7	227.5	~227.5	~244.7
	Uranium: (g/ℓ)	--	23.2	--	52.4	(1.93)	--	39.7	(15.5)
	HNO ₃ : (M)	0.97	2.64	--	(0.1)	2.36	0.32	0.38	(0.02)
Expected (from SEPHIS)	Flow Rate: (ℓ/hr)	103	612.7	246	~251.9	~710.5	228	~247.6	~231.8
	Uranium: (g/ℓ)	--	23.4	--	56.9	Trace	--	45.3	15.4
	HNO ₃ : (M)	1.0	3.0	--	0.10	2.69	0.3	0.37	0.03
Note: Data in parentheses from material balance.									

TABLE 4-10

3A AND 3B PLANT COLUMN DATA

Stream:		3AS	3AF	3AX	3AP	3AW	3BX	3BP	3BW
Plant Actual Data	Flow Rate: (ℓ/hr)	~728	304.8	179.4	-179.4	-377.6	(169)	(~169)	179.4
	Uranium: (g/ℓ)	--	(31)	--	52.4	0.17	--	44.9	10.1
	HNO ₃ : (M)	1.0	(3.37)	--	(0.1)	2.86	0.21	0.31	(0.02)
Expected (from SEPHIS)	Flow Rate: (ℓ/hr)	76	309.4	175	-119	-381.6	203	-206.6	-175.2
	Uranium: (g/ℓ)	--	33.9	--	58.6	Trace	--	49.2	1.9
	HNO ₃ : (M)	1.0	3.29	--	0.1	2.82	0.2	0.27	0.02
Note: Data in parentheses from material balance.									

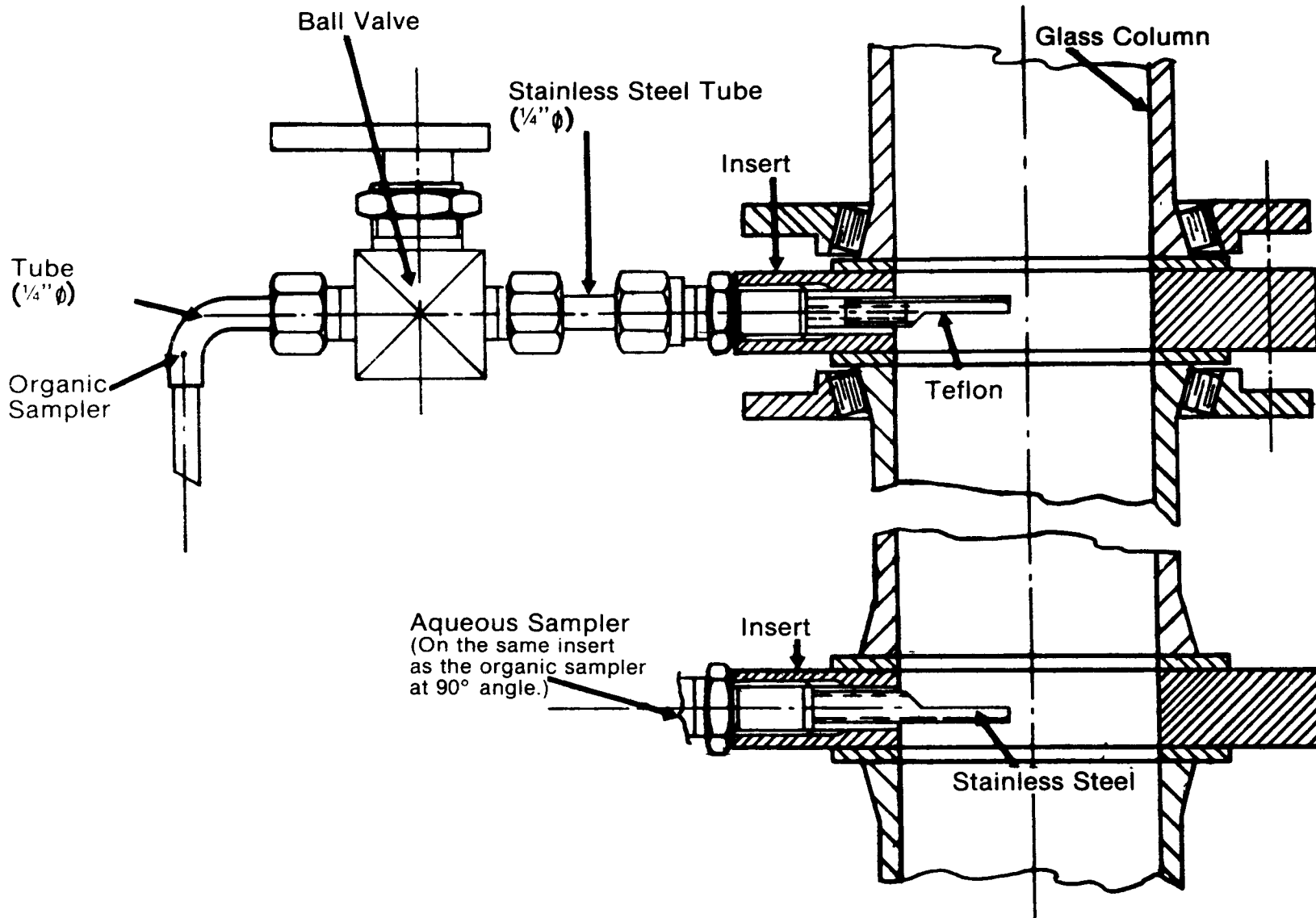
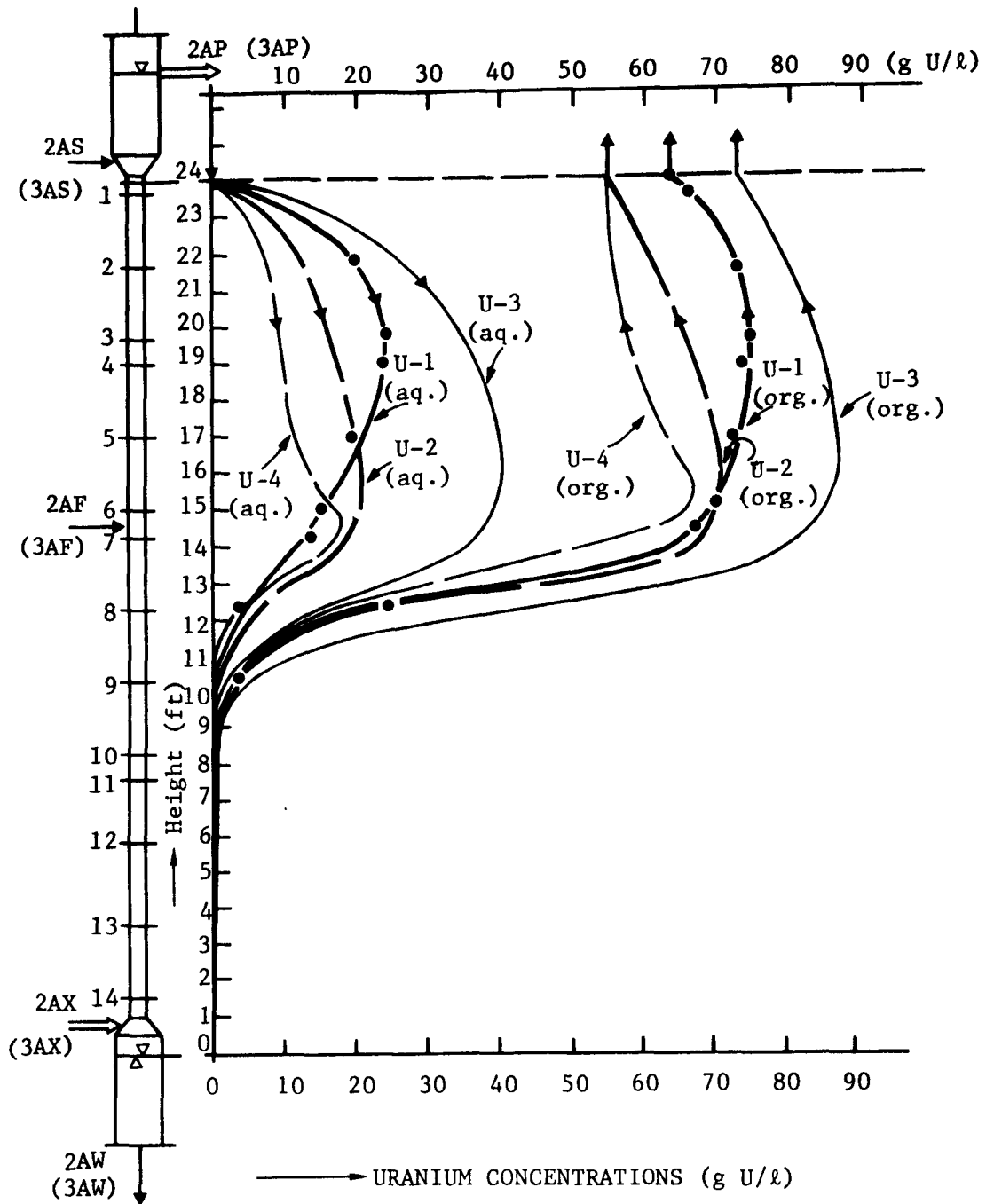


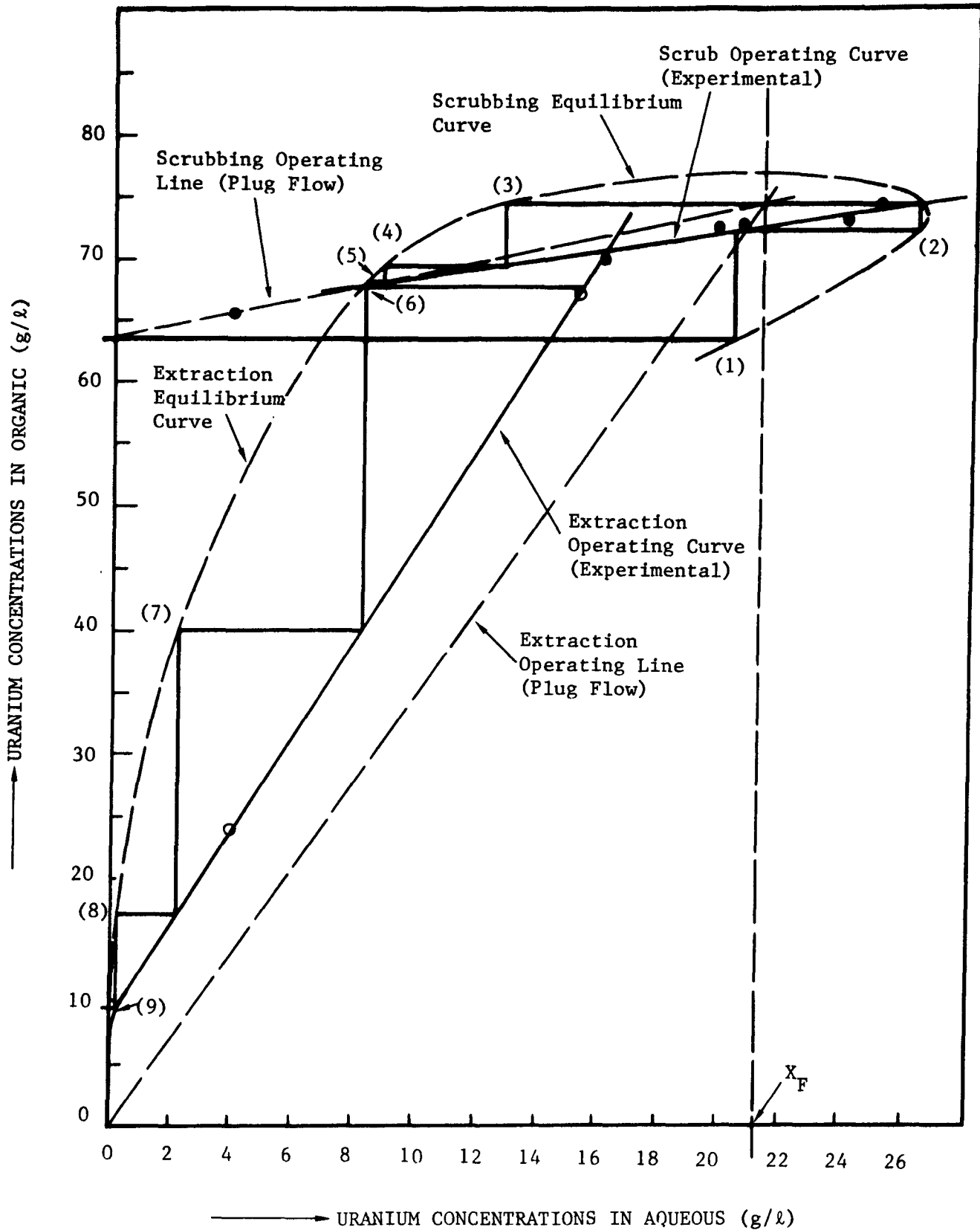
DIAGRAM SHOWING THE ORGANIC SAMPLER AND AQUEOUS SAMPLER SYSTEM

FIGURE 4-1



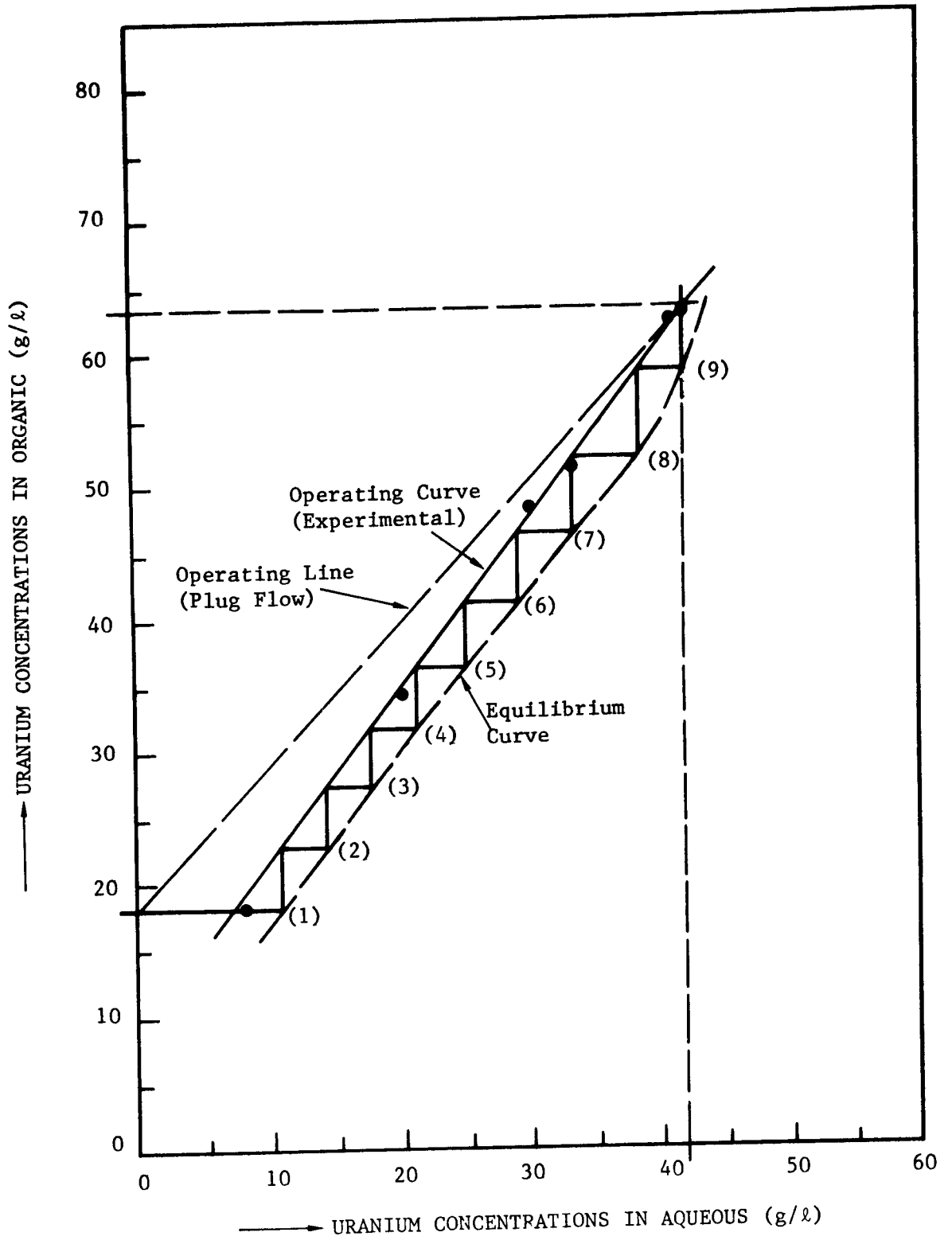
THE 2A COLUMN (RUNS U-1 AND U-2) AND 3A COLUMN (RUNS U-3 AND U-4)
EXPERIMENTAL CONCENTRATION PROFILES

FIGURE 4-2



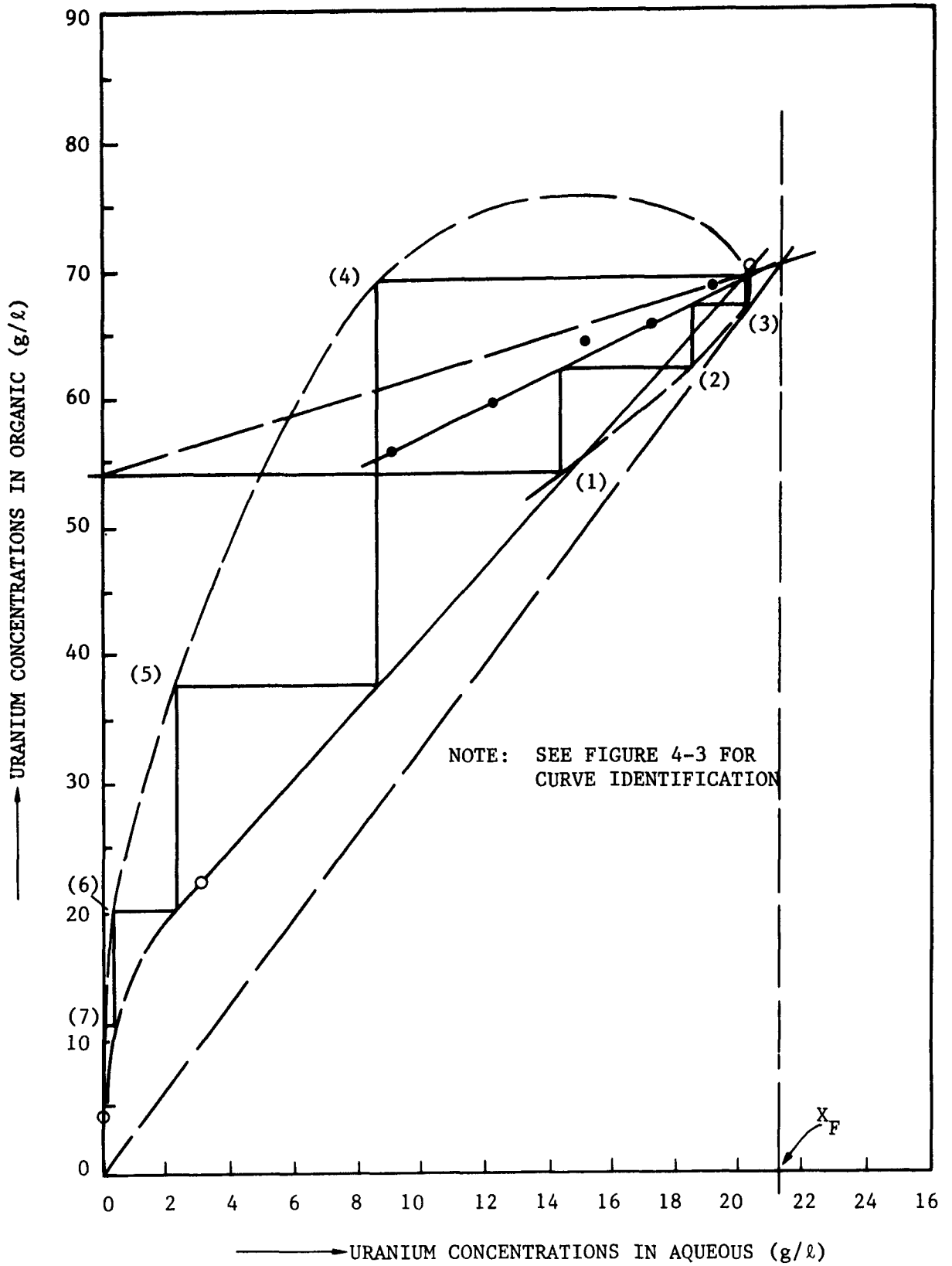
THE 2A COLUMN OPERATING DIAGRAM FOR RUN U-1

FIGURE 4-3



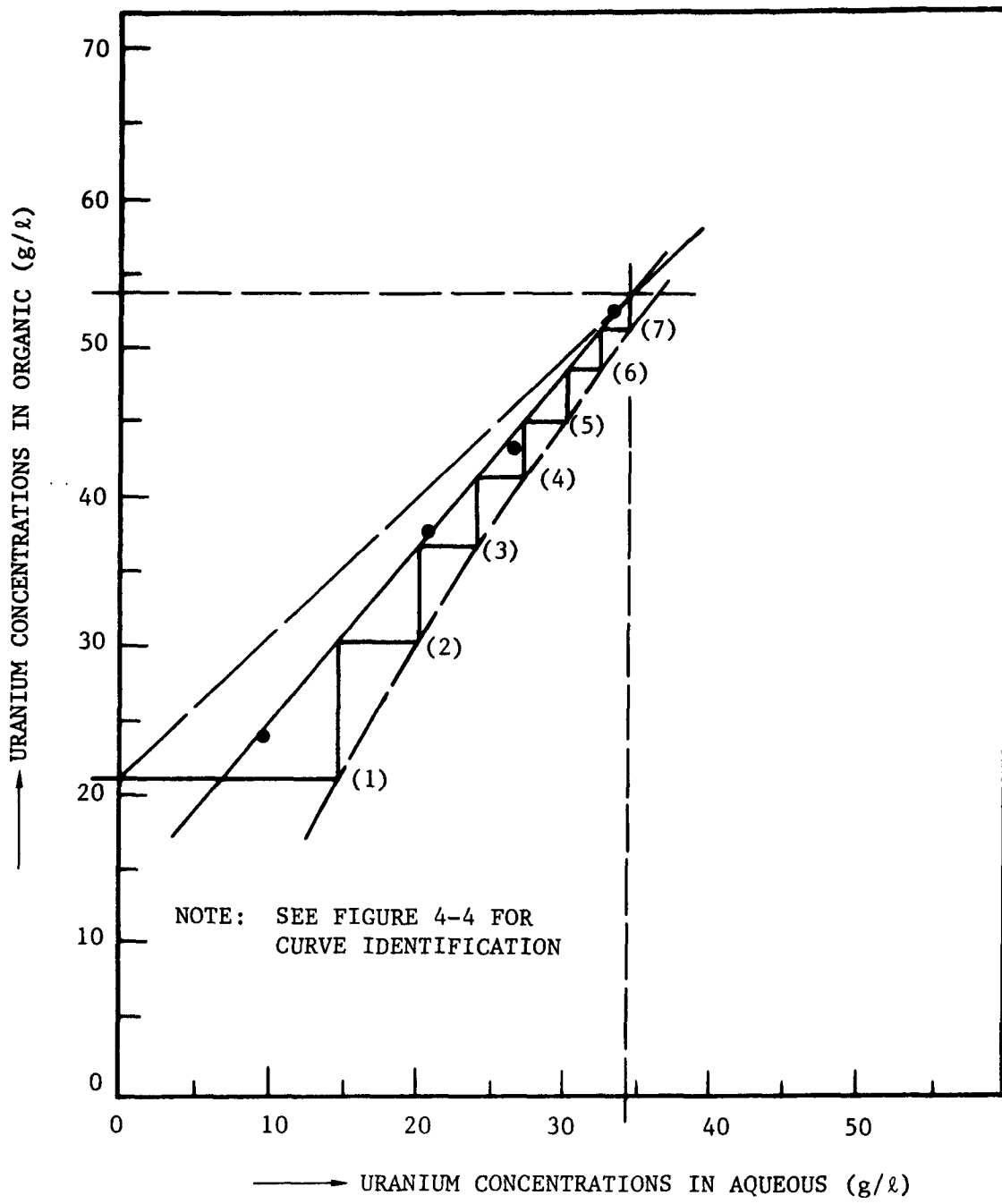
THE 2B COLUMN OPERATING DIAGRAM FOR RUN U-1

FIGURE 4-4



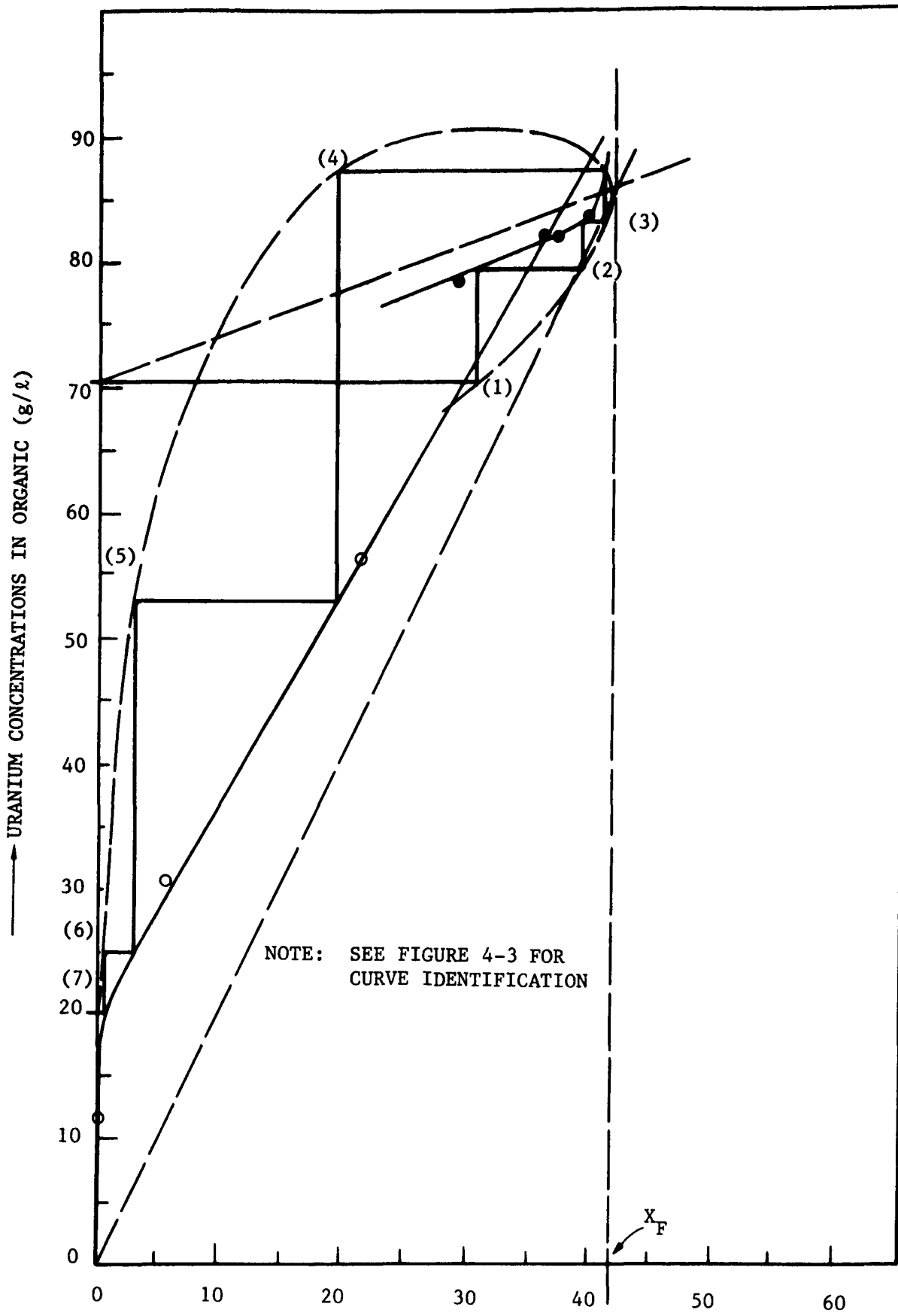
THE 2A COLUMN OPERATING DIAGRAM FOR RUN U-2

FIGURE 4-5



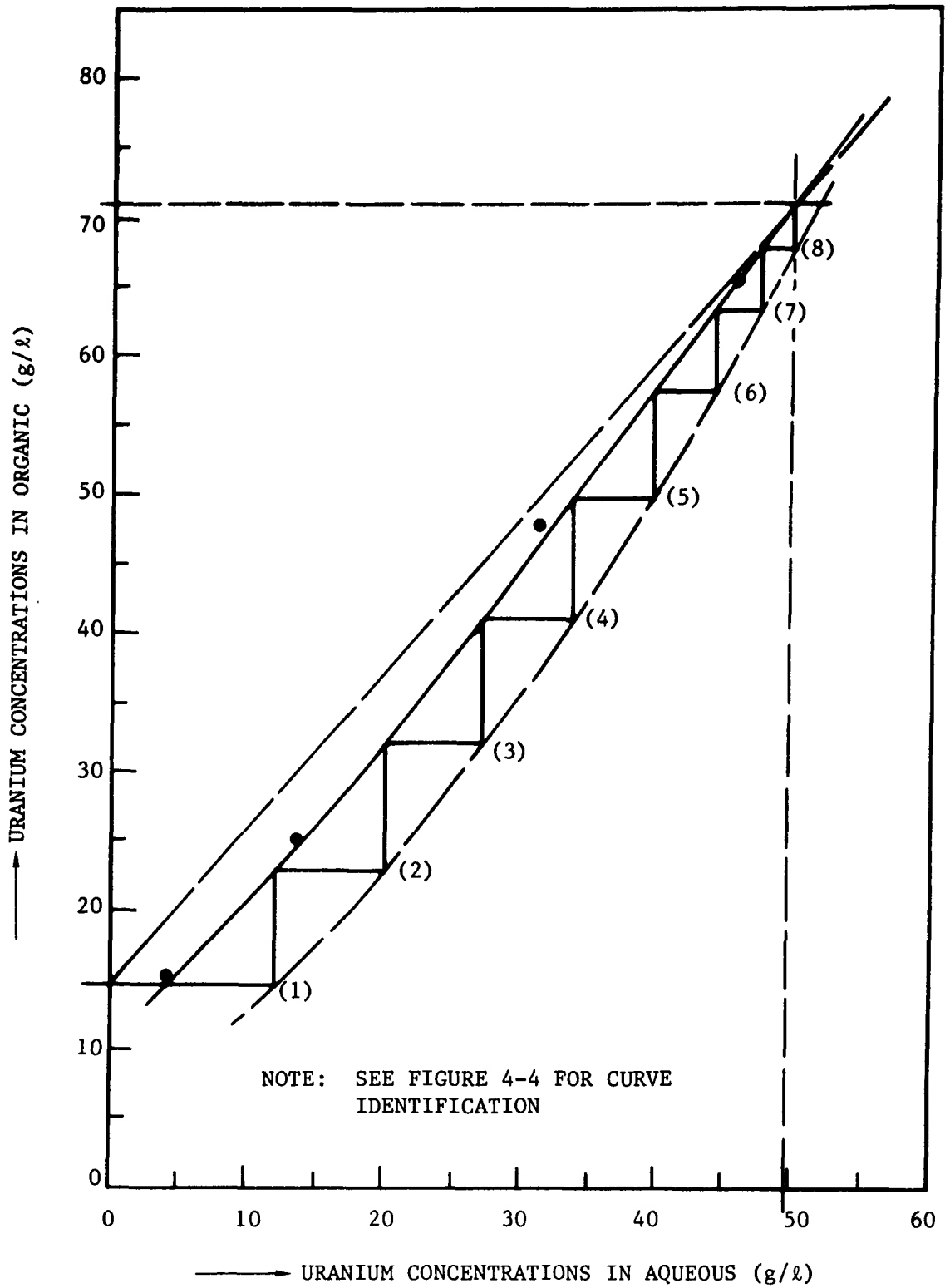
THE 2B COLUMN OPERATING DIAGRAM FOR RUN U-2

FIGURE 4-6



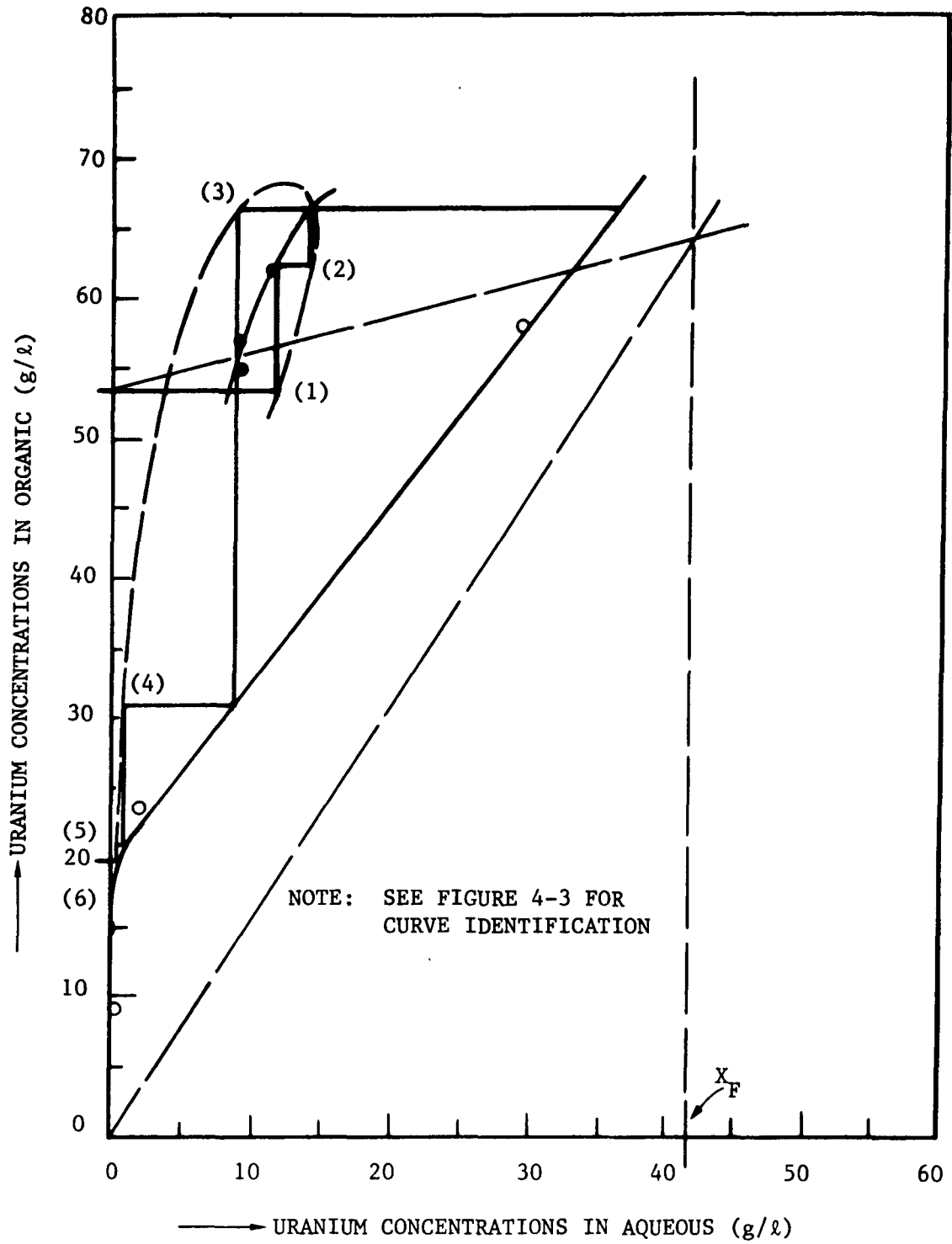
THE 3A COLUMN OPERATING DIAGRAM FOR RUN U-3

FIGURE 4-7



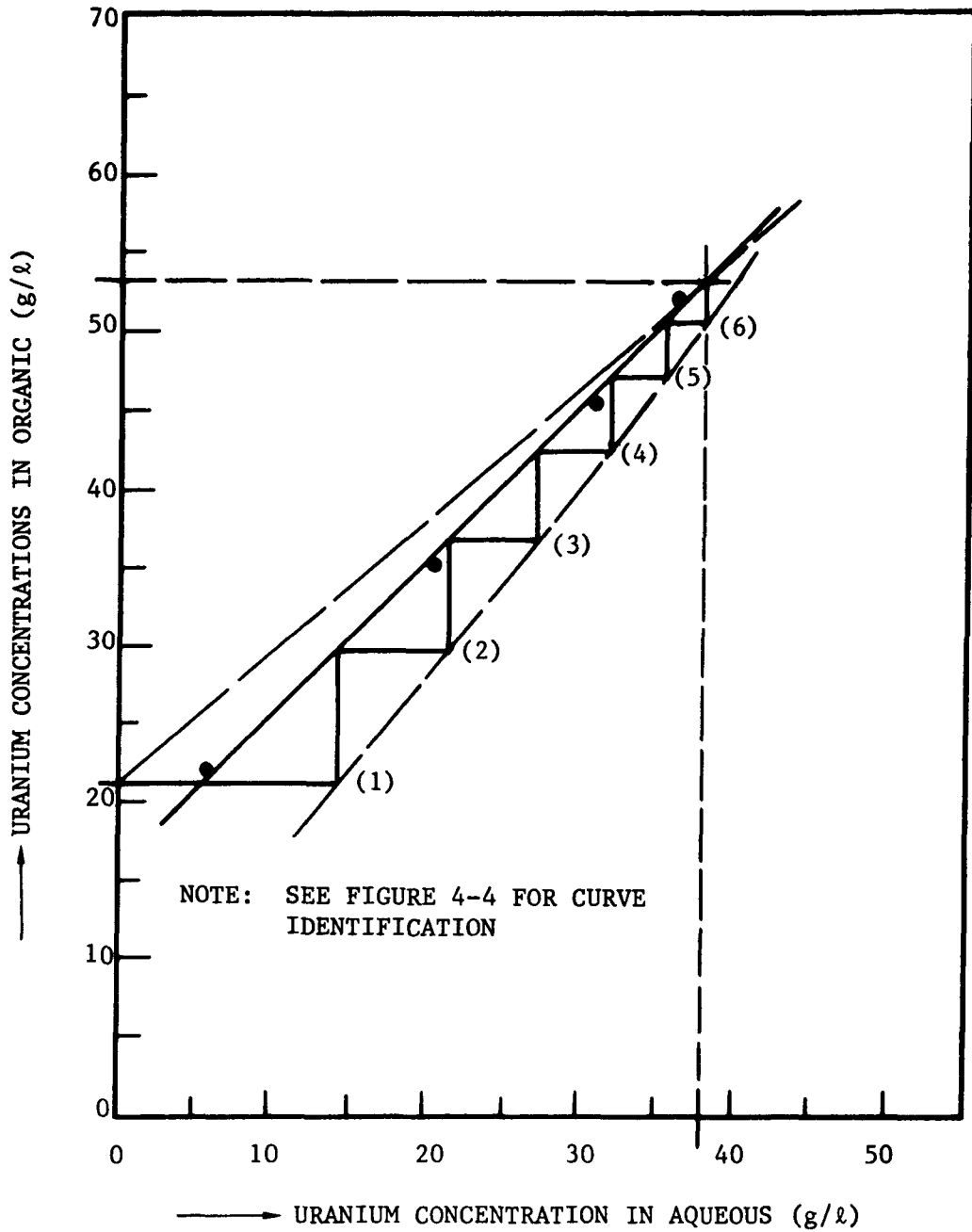
THE 3B COLUMN OPERATING DIAGRAM FOR RUN U-3

FIGURE 4-8



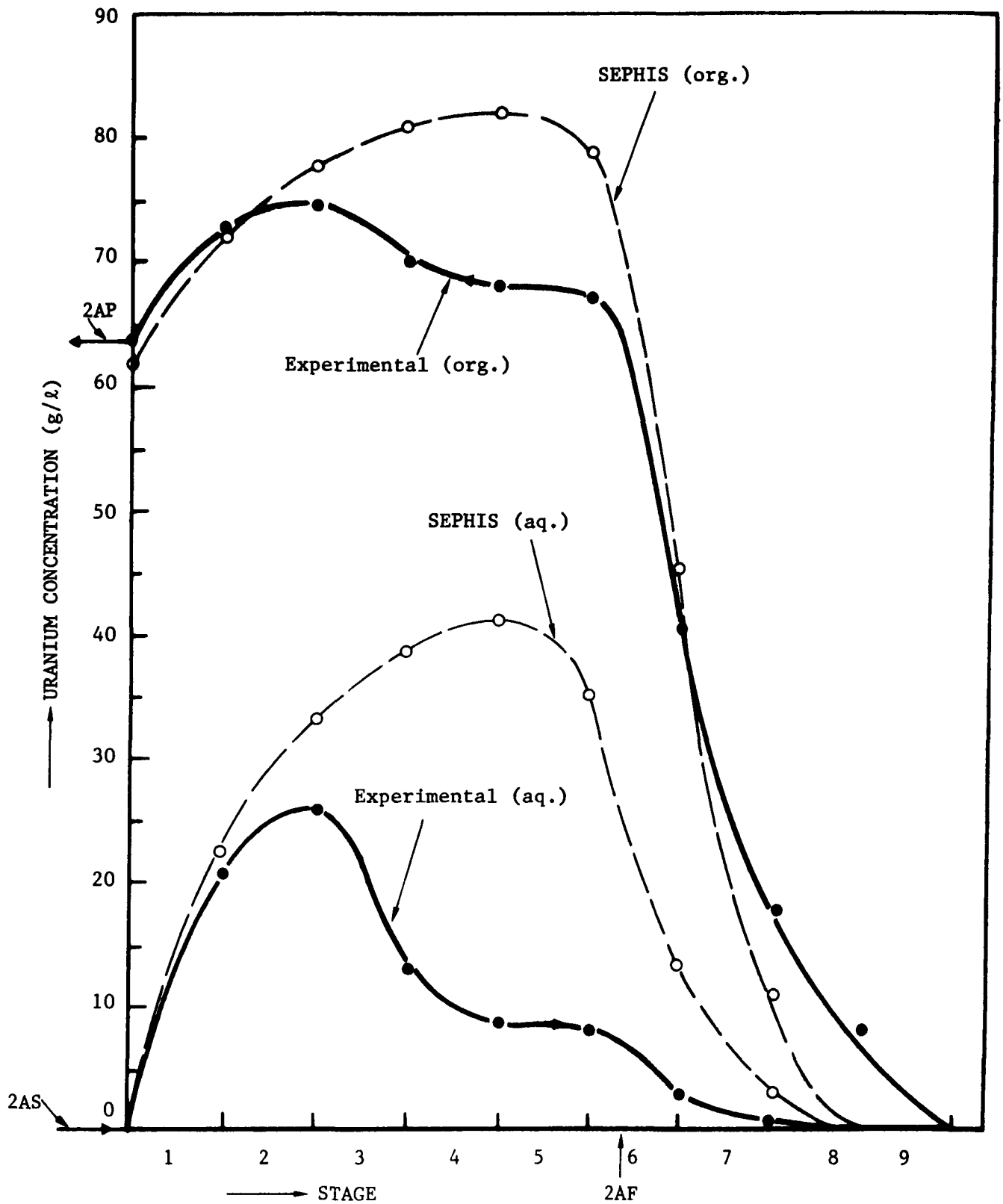
THE 3A COLUMN OPERATING DIAGRAM FOR RUN U-4

FIGURE 4-9



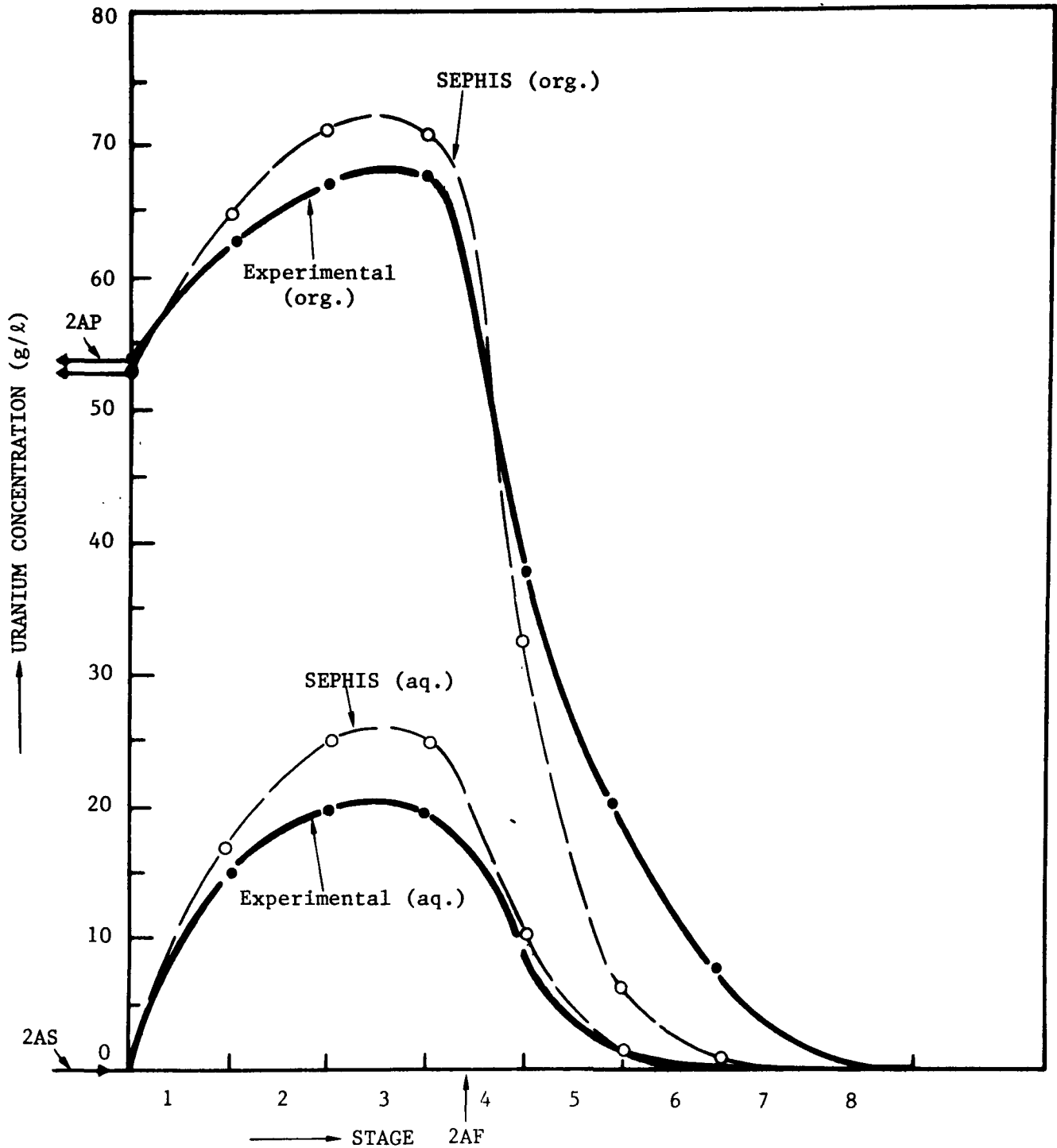
THE 3B COLUMN OPERATING DIAGRAM FOR RUN U-4

FIGURE 4-10



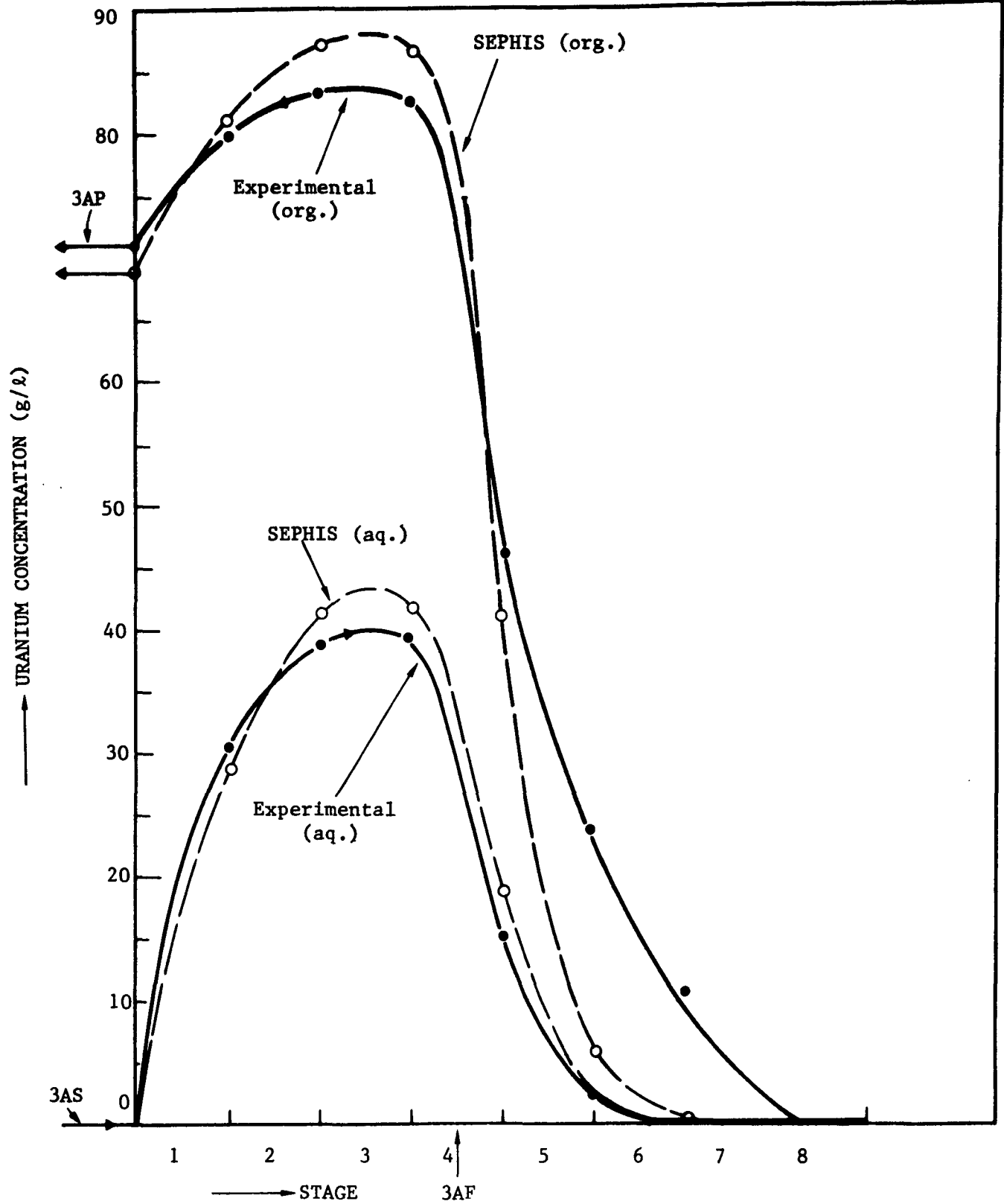
COMPARISON OF THE 2A COLUMN EXPERIMENTAL CONCENTRATION PROFILE WITH THAT OBTAINED FROM SEPHIS PROGRAM (RUN U-1)

FIGURE 4-11



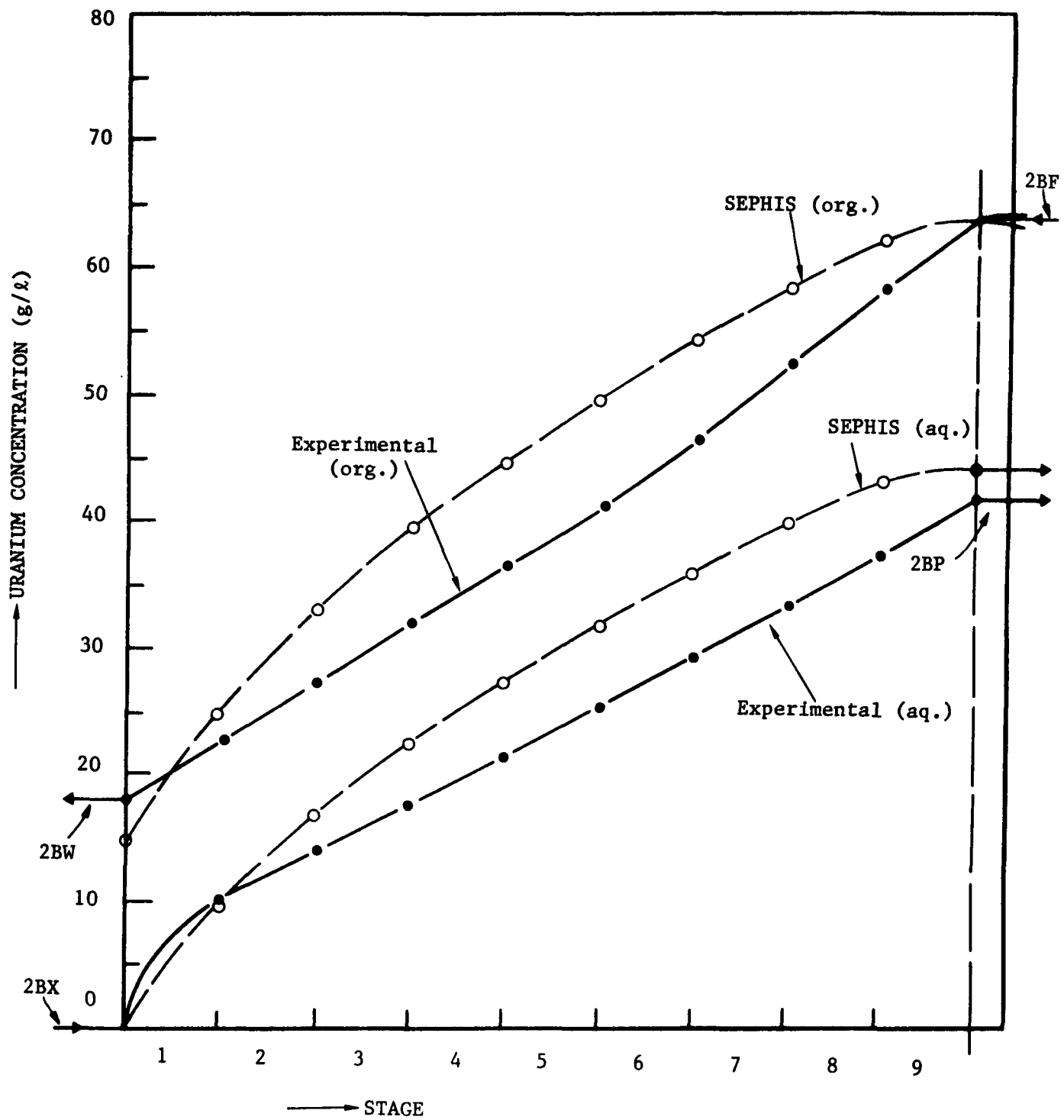
COMPARISON OF THE 2A COLUMN EXPERIMENTAL CONCENTRATION PROFILE
WITH THAT OBTAINED FROM SEPHIS PROGRAM (RUN U-2)

FIGURE 4-12



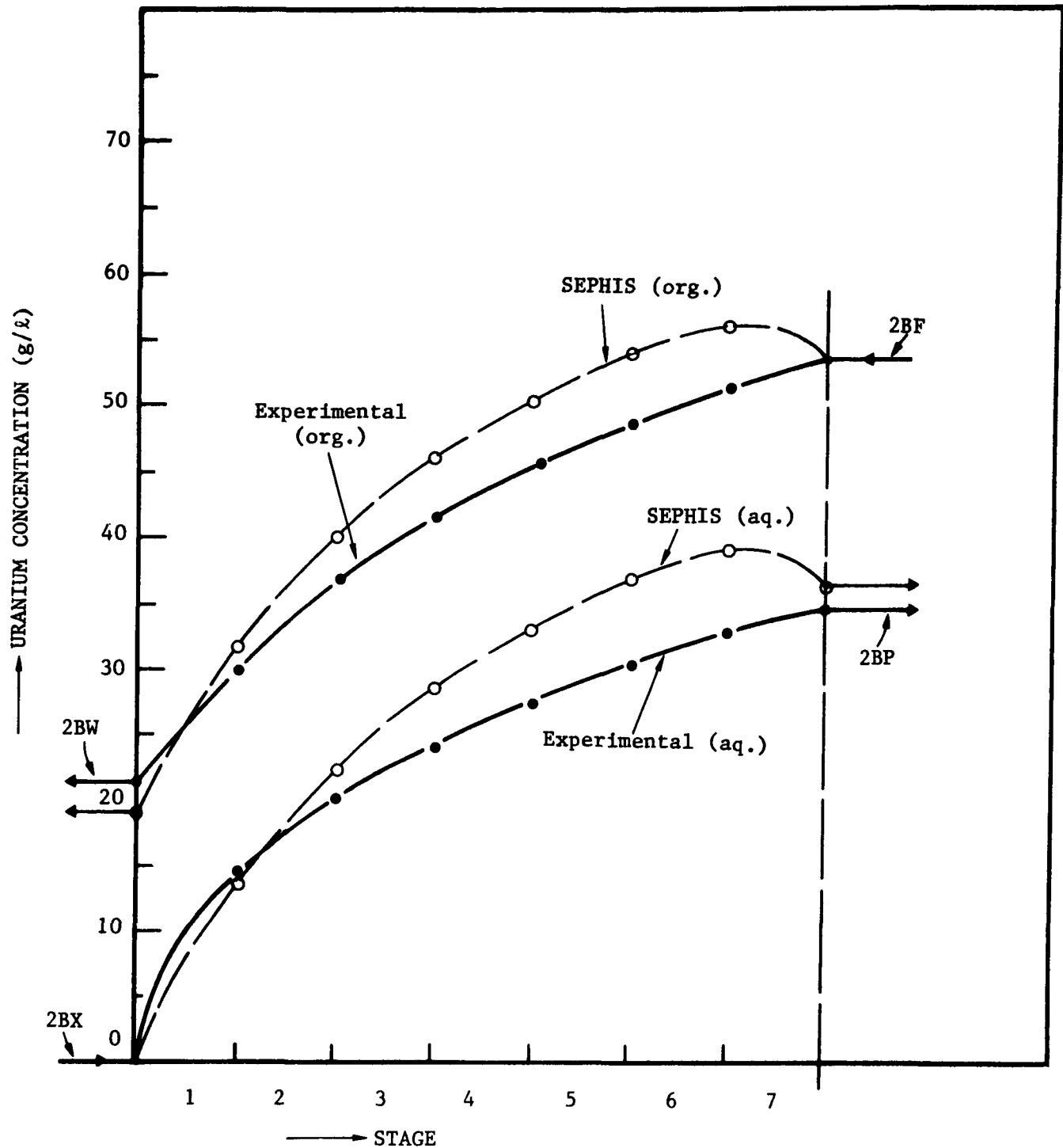
COMPARISON OF THE 3A COLUMN EXPERIMENTAL CONCENTRATION PROFILE WITH THAT OBTAINED FROM SEPHIS PROGRAM (RUN U-3)

FIGURE 4-13



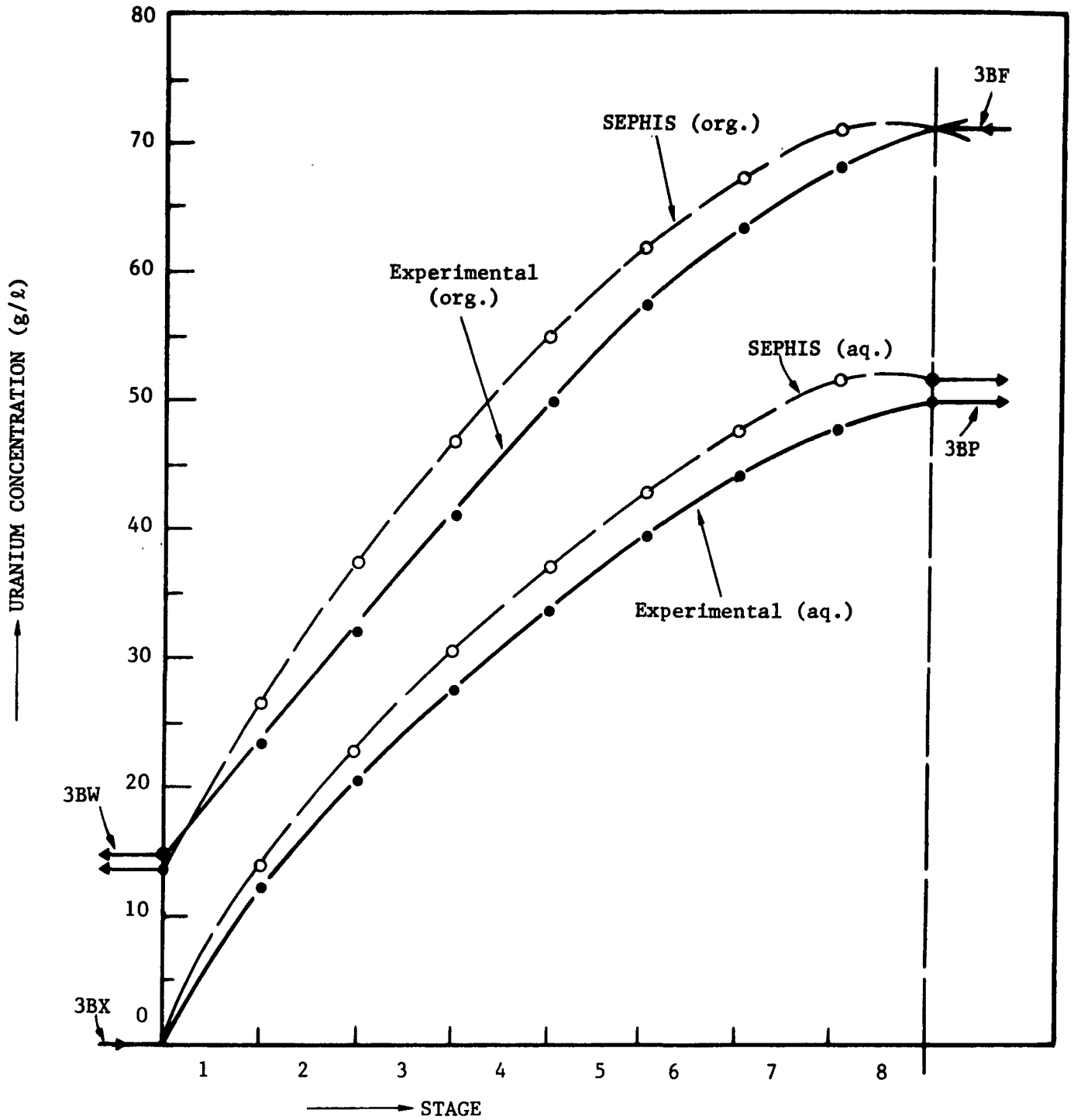
COMPARISON OF THE 2B COLUMN EXPERIMENTAL CONCENTRATION PROFILE WITH THAT OBTAINED FROM SEPHIS PROGRAM (RUN U-1)

FIGURE 4-14



COMPARISON OF THE 2B COLUMN EXPERIMENTAL CONCENTRATION PROFILE WITH THAT OBTAINED FROM SEPHIS PROGRAM (RUN U-2)

FIGURE 4-15



COMPARISON OF THE 3B COLUMN EXPERIMENTAL CONCENTRATION PROFILE WITH THAT OBTAINED FROM SEPHIS PROGRAM (RUN U-3)

FIGURE 4-16

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