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STUDIES AND RESEARCH
CONCERNING BNFP

PILOT-SCALE PULSED COLUMNS, AQUEOUS CONTINUOUS DUAL PROCESS AND HOLDUP STUDIES

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ABSTRACT

Experimental studies included in this report were conducted for the purpose of obtaining data supporting safeguards programs related to determination of near real-time solutes inventory in pulsed columns.

Holdup tests on pulsed columns resulted in an empirical equation suitable for evaluation of the average dispersed phase holdup in a column. Mass-transfer tests on pilot-scale columns, simulating the HA and 1C column coprocessing flowsheet conditions, were performed operating both columns with the aqueous phase continuous. The aqueous continuous HS column is efficient, has a lower uranium inventory, and considerably shorter phase contact time than the organic continuous column. Concentration profile and uranium inventory data were determined during each run. The newly developed "mass rate measurement" technique has been tested and proven suitable for determination of the solute's inventory in a pulsed column.

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1.0 INTRODUCTION

Considerable interest has been shown in recent years in developing reliable nuclear materials accounting systems for enriched uranium and plutonium in spent reactor fuel reprocessing plants.

With regard to this, the Los Alamos Scientific Laboratory (LASL), Barnwell Nuclear Fuel Plant (BNFP), and other facilities are working intensively in developing advanced methods for nuclear materials measurement in reprocessing plant separation equipment.[1, 2, 7, 8, 9]

The pilot-scale work at the BNFP, performed under the Department of Energy Contract (DE-AC09-78ET-35900, Sub-Task 2.5), comprises experimental studies supporting the development of techniques for solutes inventory determination in pulsed columns used in the first process cycle (HS and 1C columns), when operated with the aqueous phase continuous. The studies include experimental determination of uranium concentration profile, dispersed phase holdup, and uranium inventory data in tested columns. The newly developed "mass rate measurement" technique for solute's inventory determination in separation equipment was also employed successfully.

2.0 SUMMARY

Fifty-two hydraulic tests performed on the pilot-scale pulsed columns verified the effect of basic hydrodynamic variables on the dispersed phase holdup (X_d) as given in Reference 1. In addition, tests have shown that the dispersed phase superficial velocity (U_d) affects more distinctively the phase holdup in the column at its lower values (U_d <5 centimeters/second) than at higher, even at a flow rate ratio of one. Evaluation of experimental holdup data resulted in a modified empirical equation which takes into consideration the above effect. Experimental conditions and all resultant holdup data are summarized in Tables 4-1 and 4-2.

Three mass-transfer tests were performed on the pilot-plant equipment (using natural uranium) simulating conditions in the first process cycle (HS and 1C column run conditions). The pilot-scale 1A (≡HS) column, used for extraction and scrubbing, was operated with the aqueous phase continuous instead of the organic phase continuous. All three experiments have resulted in an efficient extraction showing an undetectable amount of uranium in the 1AW aqueous waste stream. The total uranium inventory in the aqueous continuous 1A column has been determined to be about 35 to 40% lower than in the organic continuous column tested previously.[1] Run conditions are shown in Table 4-3. Results are discussed in Section 4.0.

The pilot-plant mass-transfer tests were designed to obtain detailed concentration profiles, operational dispersed phase holdup, and uranium inventory in each tested column. Concentration profile data are shown in Tables 4-4 through 4-6 and in Figures 4-6 through 4-11. They are discussed in Section 4.3. Operational holdup data are included in Tables 4-2 and 4-3. Uranium inventory experimental data are summarized in Table 4-9 and discussed in Section 4.4.

Particular attention was paid to the experimental determination of the uranium inventory in pulsed columns, which is comprehensively discussed in Sections 3.2.3 and 4.4. The uranium inventory in tested pulsed columns was determined through:

(1) volume and concentration measurement at the end of the run,

(2) calculations from experimental concentration profile and holdup data, and

(3) uranium mass rate measurement in feed and effluent streams in short-time intervals, which is a new method.

Uranium inventory resultant data from all three techniques are in good agreement as shown in Table 4-9. The mass rate measurement technique for solute's inventory determination in a pulsed column is described in Section 3.2.3. Resultant data are discussed in Sections 4.4 and 5.0.

3.0 EXPERIMENTAL

3.1 Equipment Description

The pilot-scale experimental equipment, consisting of three glass pulsed columns and ancillary equipment such as tanks and pumps, is shown in Figure 3-1. The 2-inch (50-millimeter) diameter 1A dual column, used for extraction and scrubbing, had a working extraction section of 13 feet (3.96 meters) and (upper) scrubbing section of 9.3 feet (2.8 meters). The 3-inch (75-millimeter) diameter 1BX column, used for stripping had a working section of 17 feet (5.2 meters). Both of the above columns operated with the aqueous phase continuous during mass-transfer tests. The 2-inch (50-millimeter) diameter 1BS column, with a working section height of 18 feet (approximately 5.5 meters) and operating with the aqueous phase continuous, was used for hold-up tests.

All three columns were provided with stainless steel nozzle plates of a thickness of 1/16-inch (1.6 millimeters), having 23% relative free surface area, and 1/8-inch (3.2-millimeter) orifice diameter. In each column, the nozzle plates were assembled on a central 1/4-inch (6-millimeter) diameter stainless steel 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. A bellows-type direct pulser was attached to the bottom disengaging section. To minimize the effect of interface fluctuation on resultant data, the interface in each column was controlled manually during tests.

Columns were equipped with several samplers at different locations along the columns' working section as shown in Figure 3-2.

All feed and receiving tanks, as well as preparatory tanks, are made of stainless steel. Their outfit and size are shown in Figure 3-1. Feed flow rate was controlled by calibrated displacement pumps.

3.2 Procedure

Experimental work on pilot-plant pulsed columns included hydrodynamic and mass-transfer tests. Hydrodynamic tests were made to define the effect of flow conditions on the dispersed phase holdup within the column. Mass-transfer tests were essentially made to determine the efficiency of the 1A dual process column (extraction and scrubbing) operating with the aqueous phase continuous, which includes the uranium concentration profile measurement along the column, as well as determination of the uranium inventory (mass holdup) within the column. Both the uranium concentration profile and inventory were also measured in the 1BX single process column (stripping).

3.2.1 Dispersed Phase Holdup

The dispersed phase holdup was determined through liquid volume measurements in the column at the end of each test. This technique includes

determination of the interface location prior to shutdown, then simultaneously shutting off feed and effluent lines, separation of both phases and measurement of liquid volumes within the column. The dispersed phase holdup was determined by dividing the measured volume of the dispersed phase by the total liquid volume of the column active section.[1] Holdup values were measured in the same manner also during the actual mass-transfer tests.

For verification of selected resultant data, the holdup determination technique based on weight recorder readings, shown in Reference 1, was also used. Holdup tests were performed at room temperature with the liquid system 30% TBP - 0.1 M HNO_3.

Test conditions and resultant holdup data are shown in Tables 4-1 and 4-2, and in Figures 4-1 through 4-5.

3.2.2 Mass-Transfer Tests with Aqueous Phase Continuous

Pilot-plant cold uranium runs (utilizing natural uranium only) were performed with the aqueous phase continuous in both tested pilot-scale columns simulating the BNFP HS and 1C column process conditions when the HS column is operated as an extraction-scrub column. The experimental work objectives were to determine (a) the efficiency of the HS dual process column operating with the aqueous phase continuous, (b) uranium concentration profile in each column, and (c) uranium inventory within tested columns.

The mass-transfer efficiency, concentration profile, and uranium inventory are discussed in Section 4.0. Concentration profiles were based on chemical analyses of samples drawn at each sampler along the column during steady-state operation. The method for calculation of concentration profiles is described in Reference 2. The methods for chemical analyses are shown in Reference 1 (Appendix A).

Data for the three runs, simulating the HS and 1C column process conditions, are shown in Table 4-3. Operating conditions and resultant data are discussed in Section 4.0. All tests were conducted at room temperature (about 23°C).

3.2.3 Solutes Inventory (Accountability)

Inventories for accountability of solutes (U, Pu, HNO_3, etc.) in a pulsed column are discussed in Reference 1. The solutes inventory can be basically determined through

(a) volume-concentration measurement at the end of the run,

(b) concentration profiles in conjunction with holdup data within the column, and

(c) empirical equations.

In addition, a new method described below, which is based on feed and effluent streams flow rate and solute's concentration measurement in short time intervals, can be used effectively for the column inventory determination. Basically, the theoretical background of the method is as follows:

(A) Solute's Inventory in a Column

The single and dual process column are shown schematically in Figures 3-3 and 3-4, respectively. The uranium (or plutonium) inventory is considered in both columns.

(1) Assume conditions as shown in Figure 3-5, i.e,

$$m_f = v_f \cdot C_f = \text{constant},$$

$$m_A + m_O = v_A \cdot C_A + v_o \cdot C_o = \Phi(t),$$

and the combined effluent mass-rate $(m_A + m_o)$ equals zero at starting time $t_{(o)}$.

The total solute's amount fed to the column per time $t_{(n)}$ is given by the surface area below the feed mass-rate curve (m_f = constant) from time $t_{(o)}$ to time $t_{(n)}$. The total amount of the solute carried away from the column is given by the surface area below the combined effluent mass-rate curve from time $t_{(o)}$ to time $t_{(n)}$. The solute's inventory in the column [ΔM (col) $t_{(n)}$] is then given by the difference between the above two surface areas. This can be expressed mathematically as:

$$\Delta M_{(col)_{t_{(n)}}} = m_f \cdot t_{(n)} - \int_{t_{(o)}}^{t_{(n)}} (m_A + m_O) \cdot dt$$

$$\approx m_f \cdot t_{(n)} - \frac{\Delta t}{2} \cdot \sum_{i=(1)}^{(n)} \left[\left(m_A + m_O\right)_{i-1} + \left(m_A + m_O\right)_i \right]$$

$$\approx m_f \cdot t_{(n)} - \frac{\Delta t}{2} \cdot \left[\left(m_A + m_O\right)_{(n)} + 2 \sum_{i=(1)}^{(n-1)} \left(m_A + m_O\right)_i \right] \quad \ldots \underline{1}.$$

(2) Assume conditions as shown in Figure 3-6, i.e.,

$$m_f = \Psi(t),$$

$$m_A + m_o = \Phi(t),$$

and the combined effluent mass-rate $(m_A + m_0)$ equals zero at starting time $t_{(o)}$.

Analogously, the mathematical equation expressing the solute's inventory in the column as the difference between surface areas below both curves in Figure 3-6 has the form:

$$\Delta M_{(col)}{}_{t_{(u)}} = \int_{t_{(o)}}^{t_{(n)}} m_f \cdot d_t - \int_{t_{(o)}}^{t_{(n)}} (m_A + m_0) \cdot d_t$$

$$\approx \frac{\Delta t}{2} \cdot \sum_{i=(1)}^{(n)} \left[\left(m_f - m_A - m_o \right)_{i-1} + \left(m_f - m_A - m_o \right)_i \right]$$

$$= \frac{\Delta t}{2} \left[(m_f)_{(o)} + (m_f - m_A - m_o)_{(n)} \right]$$

$$+ 2 \sum_{i=(1)}^{(n-1)} \left[\left(m_f - m_A - m_0 \right)_i \right] \qquad \ldots \underline{2}.$$

(3) Assume general conditions as shown in Figure 3-7, i.e.,

$$m_f = \Psi(t),$$

$$m_A + m_0 = \theta(t),$$

and the combined effluent mass-rate $m_A + m_0 \neq 0$ at starting time $t_{(o)}$.

The equation expressing the solute's inventory change in a column in a time period of $t_{(n,o)} = t_{(n)} - t_{(o)}$ is then

$$\Delta M_{(col)_{t_{(n)}}} = \int_{t_{(o)}}^{t_{(n)}} m_f \cdot d_t - \int_{t_{(o)}}^{t_{(n)}} (m_A = m_0) \cdot dt$$

$$\cong \frac{\Delta t}{2} \sum_{i=(1)}^{(n)} \left[\left(m_f - m_A - m_0 \right)_{i-1} + \left(m_f - m_A - m_0 \right)_i \right]$$

$$= \frac{\Delta t}{2} \cdot \left[\left(m_f - m_A - m_0 \right)_{(o)} + \left(m_f - m_A - m_0 \right)_{(n)} \right]$$

$$+ 2 \cdot \sum_{i=(1)}^{(n-1)} \left[\left(m_f - m_A - m_0 \right)_i \right] \qquad \ldots \underline{3}.$$

Substituting

$$\Delta m = m_f - (m_A + m_o)$$

$$= v_f \cdot C_f - (v_A \cdot C_A + v_O \cdot C_O)$$

the above equation obtains the form

$$\Delta M_{(col)_{t_{(n)}}} = \frac{\Delta t}{2} \cdot \sum_{i=(1)}^{(n)} (\Delta m_{i-1} + \Delta m_i)$$

$$= \frac{\Delta t}{2} \cdot \left[\Delta m_{(o)} + \Delta m_{(n)} + 2 \sum_{i=(1)}^{(n-1)} \Delta m_i \right] \qquad \ldots \underline{4}.$$

This equation can be generally used for calculations of either (a) the solute's inventory change within the column due to changes in streams, flow rate, or concentration, or both, or (b) the total solute's inventory in the column if solute mass-rate of each stream is followed from the process startup.

(B) Total Solute's Inventory From Both the Dual and Single Process Column

Generally, the process cycle consists of a dual process column for extraction and scrubbing, and a single process column for stripping, as shown in Figure 3-8.

It can be proven mathematically that the equation for evaluation of the total solute's inventory from both columns will have the form:

$$\Delta M_{(d,\,St)_{t_{(n)}}} = \frac{\Delta t}{2} \cdot \sum_{i=(1)}^{(n)} \left\{ \left[v_f \cdot c_f - \left(v_A \cdot c_A\right)^{(d)} - \left(v_A \cdot c_A\right)^{(St)} - v_0 \cdot c_0 \right]_{i-1} \right.$$

$$\left. + \left[v_f \cdot c_f - \left(v_A \cdot c_A\right)^{(d)} - \left(v_A \cdot c_A\right)^{(St)} - v_0 \cdot c_0 \right]_i \right\} \quad \ldots \underline{5}.$$

Substituting

$$\Delta m^* = v_f \cdot c_f - \left[\left(v_A \cdot c_A\right)^{(d)} + \left(v_A \cdot c_A\right)^{(St)} + v_0 \cdot c_0 \right]$$

the equation is then

$$\Delta M_{(d,\,St)_{t_{(n)}}} = \frac{\Delta t}{2} \cdot \sum_{i=(1)}^{(n)} \left(\Delta m^*_{i-1} + \Delta m^*_i \right)$$

$$= \frac{\Delta t}{2} \cdot \left[\Delta m^*_{(o)} + \Delta m^*_{(n)} + 2 \cdot \sum_{i=(1)}^{(n-1)} \Delta m^*_i \right] \quad \ldots \underline{6}.$$

(C) Total Solute's Inventory in Separation Equipment Consisting of Several Process Cycles

Data which are required for the solute's inventory determination in a separation equipment arrangement consisting of several process cycles are shown in Figure 3-9.

It can be proven analogously that the equation for evaluation of the solute's inventory $[M_{(E)}]$ in the entire separation equipment arrangement will have the form

$$\Delta M_{(E)}{}_{t_{(n)}} = \frac{\Delta t}{2} \sum_{i=(1)}^{(n)} \left[\left(v_f \cdot C_f - \sum^{AR} v_A \cdot C_A - \sum^{OR} v_O \cdot C_O \right)_{i-1} + \left(v_f \cdot C_f - \sum^{AR} v_A \cdot C_A - \sum^{OR} v_O \cdot C_O \right)_i \right] \quad \cdots 7.$$

Substituting

$$\Delta m(E) = v_f C_f - \left(\sum^{AR} v_A C_A + \sum^{OR} v_O C_O \right)$$

$$= m_f - \left(\sum^{AR} m_A + \sum^{OR} m_O \right)$$

The above equation will be then

$$\Delta M(E)_{t_{(n)}} = \frac{\Delta t}{2} \sum_{i=(1)}^{(n)} \left[\Delta m(E)_{i-1} + \Delta m(E)_i \right]$$

$$= \frac{\Delta t}{2} \left[\Delta m(E)_{(o)} + \Delta m(E)_{(n)} + 2 \sum_{i=(1)}^{(n-1)} \Delta m(E)_i \right] \quad \cdots 8.$$

Following the solute's mass rate in the streams indicated in Figure 3-9 from the process startup [during time period $t_{(n)} - t_{(o)}$], the solute's inventory in entire separation equipment could be determined by one of the above equations.

In equation 7,

$\sum^{AR} m_A = \sum^{AR} v_A \cdot C_A$ = The sum of solute's mass-rate in all final aqueous receivers at sampling (measuring) time (grams/minute).

$\sum^{OR} m_O = \sum^{OR} v_O \cdot C_O$ = The sum of solute's mass-rate in all final organic receivers at sampling (measuring) time (grams/minute).

During experimental runs shown in this report, the uranium inventory in pilot-scale pulsed columns was determined

- by use of the volume-concentration measurement method,
- from concentration profiles and measured hold-up data, and
- through mass rate measurement method using the above equations 4 and 6.

Resultant data are summarized in Tables 4-7 through 4-9.

In the above equations:

v = Flow rate (liters/minute)

C = Solute's concentration (grams/liter)

m, m^* = Solute's mass rate (grams/minute)

Δm, Δm^* = Solute's mass rate difference (grams/minute)

t = Time (minute)

Δt = Time interval (minute).

Indexes:

f = Feed

A = Aqueous phase

O = Organic phase

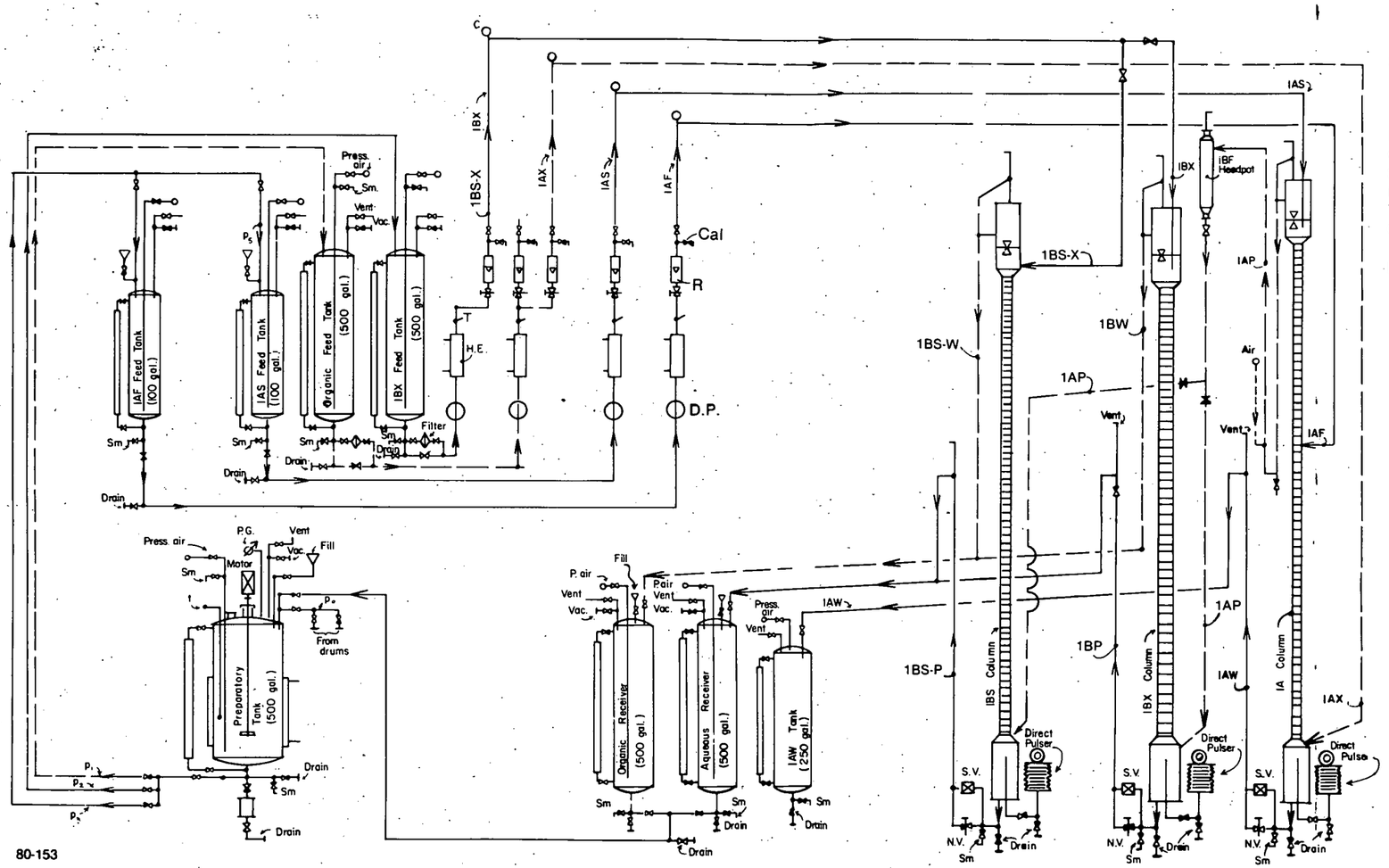
d = Dual column

St = Stripping column

E = Separation equipment

AR = Aqueous receivers

OR = Organic receivers.

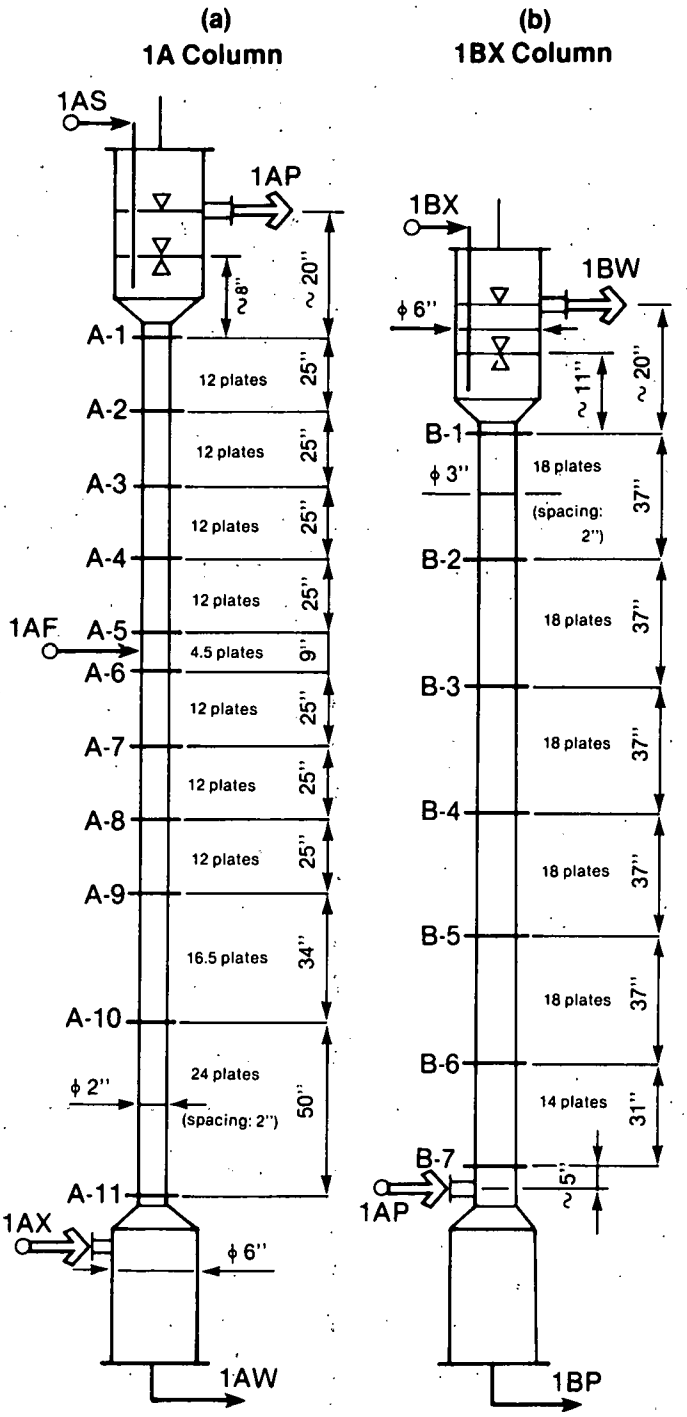


LEGEND

SM	- Sampling
P.G.	- Pressure Gage
P. Air	- Pressurized Air
H.E.	- Heat Exchanger
R	- Rotameter
Cal	- Calibration
S.V.	- Solenoid Valve
N.V.	- Needle Valve
T	- Temperature
C	- Surge Pot
⧖	- Interface Control
D.P.	- **Displacement Pump**

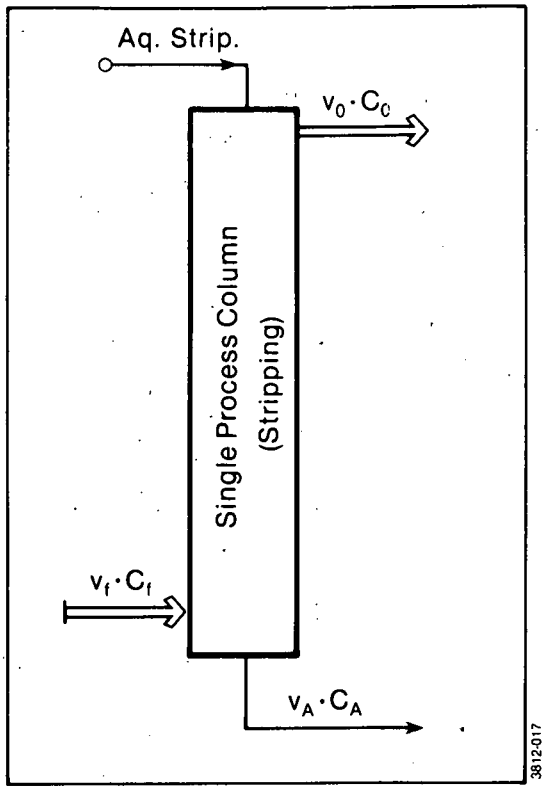
FLOW DIAGRAM

FIGURE 3-1



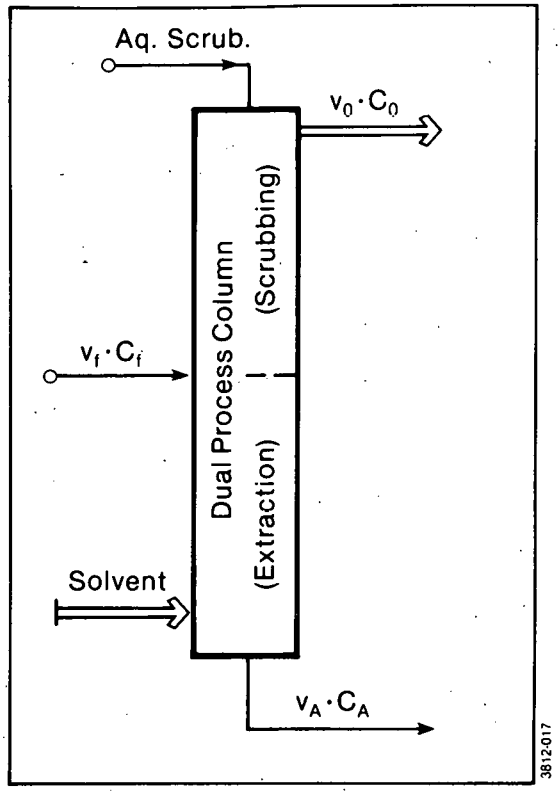
SAMPLERS LOCATION OF THE 1A AND 1BX COLUMNS

FIGURE 3-2



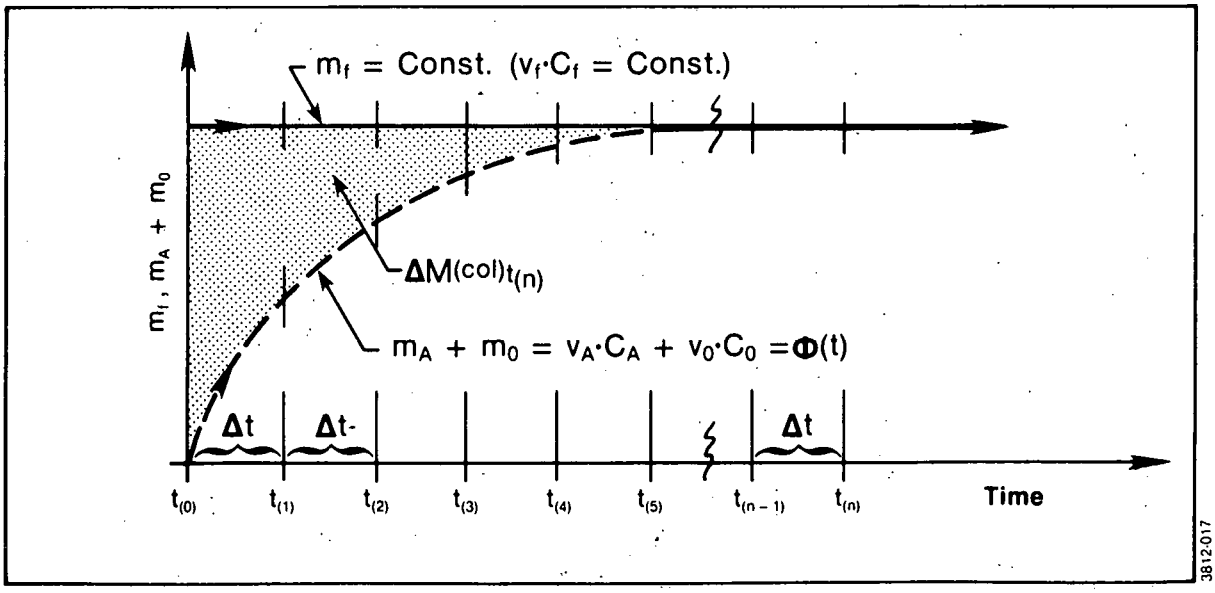
SINGLE PROCESS COLUMN
(for stripping)

FIGURE 3-3



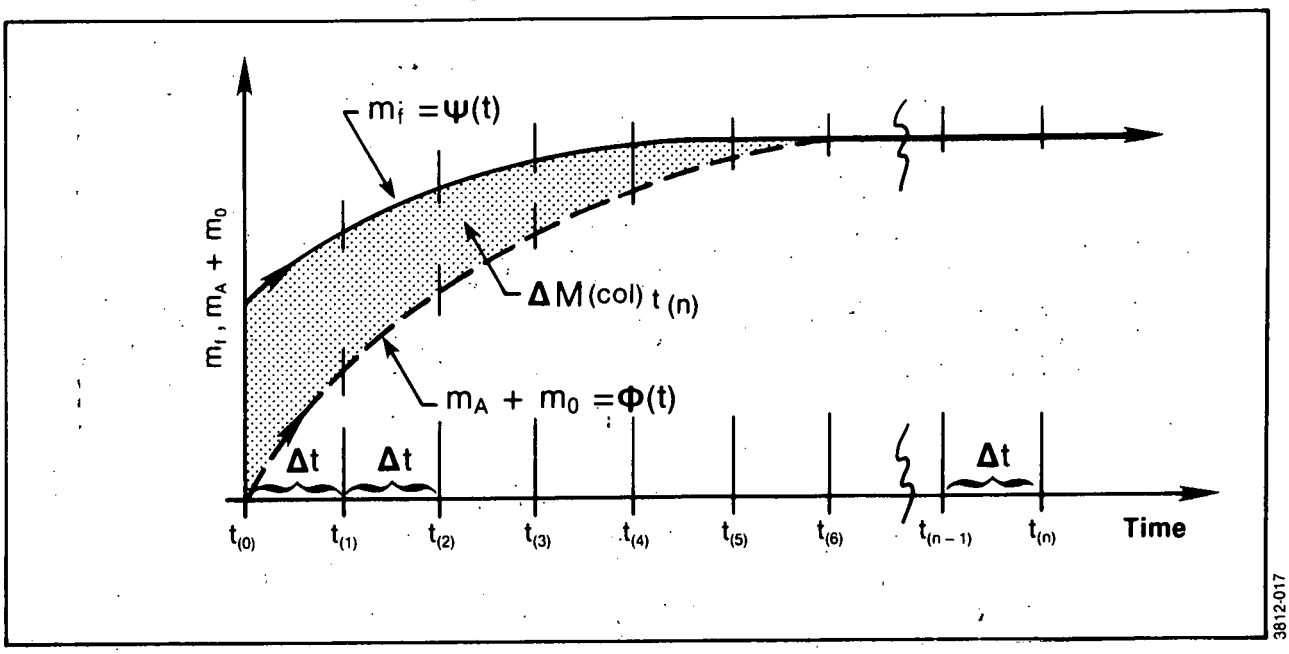
DUAL PROCESS COLUMN
(for extraction and scrubbing)

FIGURE 3-4



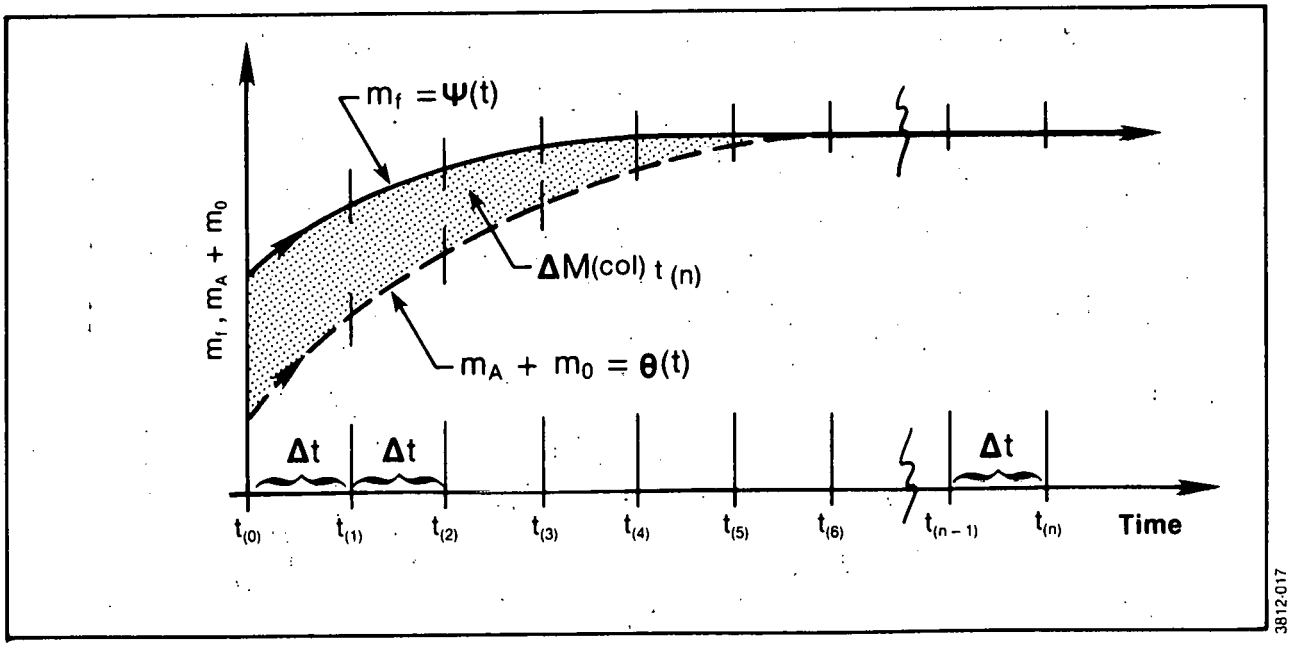
SOLUTE'S MASS RATE VERSUS TIME

FIGURE 3-5



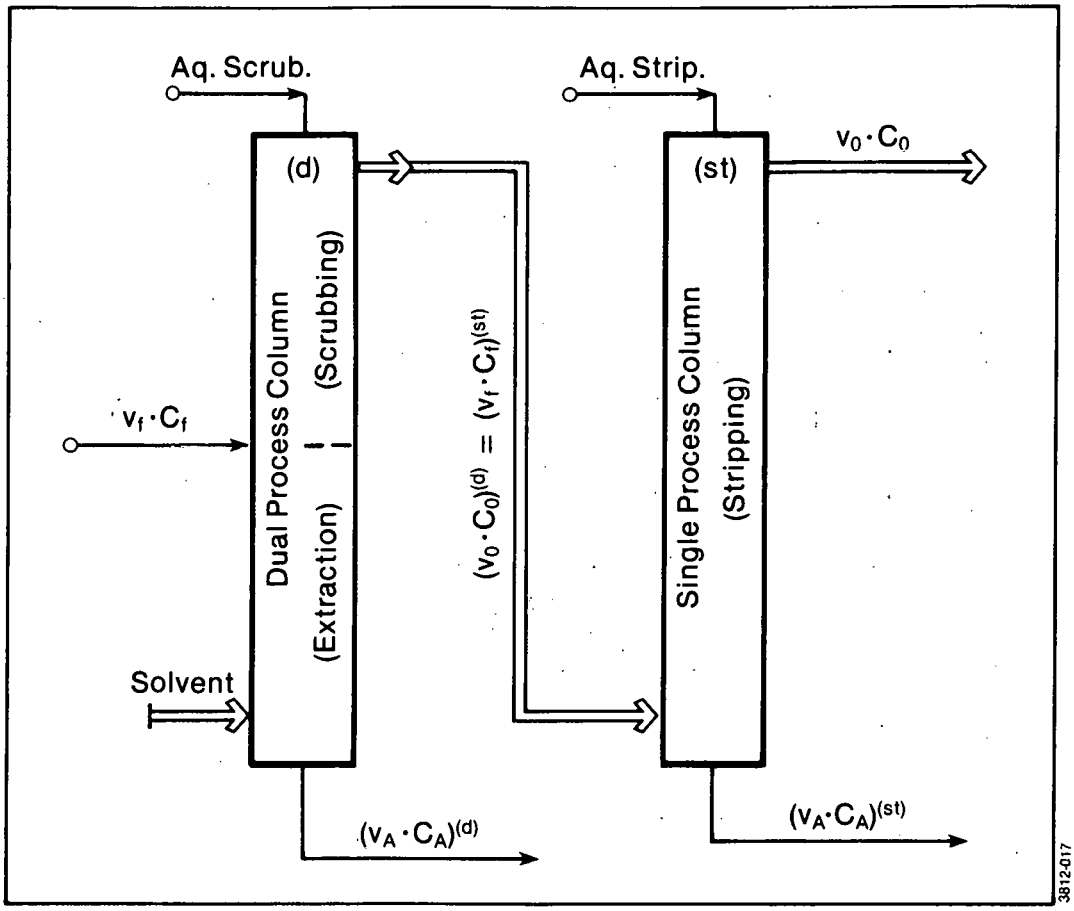
SOLUTE'S MASS RATE VERSUS TIME

FIGURE 3-6



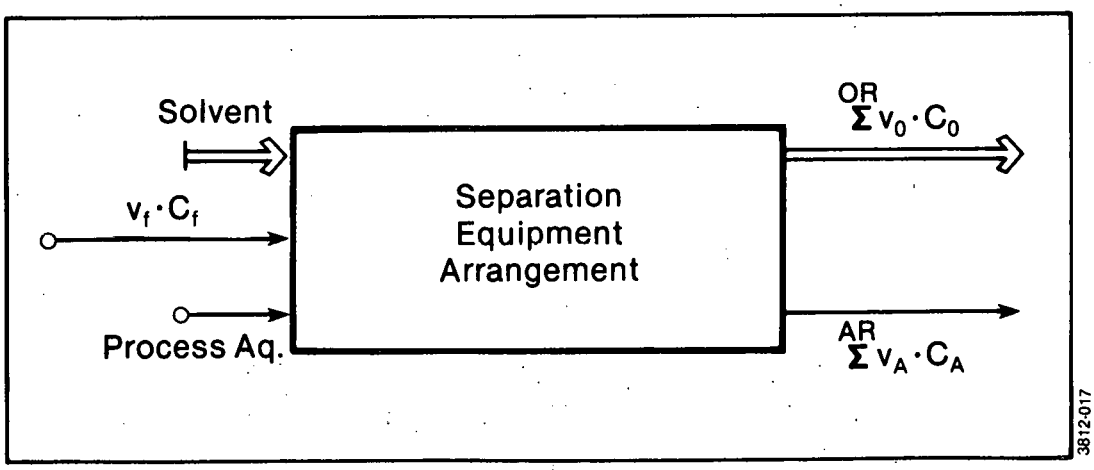
SOLUTE'S MASS RATE VERSUS TIME

FIGURE 3-7



PROCESS CYCLE CONSISTING OF A DUAL AND SINGLE PROCESS COLUMN

FIGURE 3-8



SEPARATION EQUIPMENT ARRANGEMENT DATA REQUIRED FOR SOLUTE'S INVENTORY

FIGURE 3-9

4.0 RESULTS AND DISCUSSION

4.1 Dispersed Phase Holdup

Run conditions and experimental holdup data obtained from actual holdup tests, described in Section 3.2.1, and also measured during mass-transfer tests are summarized in Tables 4-1 and 4-2.

Fifty-two dispersed phase holdup (X_d) values in the tables were determined by volumetric measurements at the end of each run. Thirteen of these values were verified by calculations based on weight recorder charts.

A previous study,[1] which resulted in an empirical equation for X calculation of the form

$$X_d \approx 0.185\ (2fA_o + \Delta U_{d-c})^{2.8} \qquad \ldots\ 9.$$

has shown that the X_d value increases with an increase in the pulse frequency (f), pulse amplitude (A_o), ratio of the dispersed to continuous phase flow, and flow rate of the dispersed phase entering the pulsed column. The effect of the above variables on X_d was demonstrated again through tests included in this report.

Experimental data correlated well with data calculated from Equation 9, within the tested range, shown in Tables 4-1 and 4-2, and Figure 4-4, although the equation does not describe the effect of the dispersed phase flow rate on X_d at a flow ratio of $U_c/U_d = 1$. For that ratio, it takes into account an average X_d value of the tested flow rate range.

Experiments have shown a quite recognizable effect of the dispersed phase flow rate (or superficial velocity) on the holdup at a ratio of $U_c/U_d = 1$, as shown in Figure 4-3. This effect is more distinctive at lower dispersed phase superficial velocities ($U_d < 0.5$ centimeters/second) than at its higher values. The figure indicates also an increase in the X_d value with any increase in pulse frequency and/or pulse amplitude.

Figure 4-1 shows the measured dispersed phase holdup as a function of the dispersed phase superficial velocity at ratios of $U_c/U_d = 0.33$ and 3.0, operating the column with the organic phase continuous. Figure 4-2 shows the relationship between the holdup (X_d) and superficial velocity (U_d) at a flow rate of $U_c/U_d = 0.33$, operating the tested column with the aqueous phase continuous.

Experimental holdup data in Tables 4-1 and 4-2 were mathematically analyzed and evaluated. With respect to the effect of both the expression $2fA_o + \Delta U_{d-c}$, which can be considered as twice the pulse velocity in the direction of the dispersed phase flow in the column[1] (see Figure 4-4), and of the superficial velocity U_d on the dispersed

phase holdup (see Figures 4-1 through 4-3), an empirical equation was derived in the form

$$X_d \approx 0.20\ U_d^{0.11}(2fA_o + \Delta U_{d-c})^{2.8} \qquad \ldots\ 10.$$

In this equation

X_d = dispersed phase holdup (%),

U_d = dispersed phase superficial velocity (cm/second),

f = pulse velocity (1/second),

A_o = Pulse amplitude (cm), and

ΔU_{d-c} = $U_d - U_c$, i.e., the difference between the dispersed and continuous phase superficial velocities (cm/second).

Figure 4-5 describes graphically the measured dispersed phase holdup X_d as a function of the expression $U_d^{0.11}(2fA_o + \Delta U_{d-c})^{2.8}$. Statistical analysis indicates an average uncertainty of ±9% around the fixed line in this figure.

The equation (10) takes into account the effect of the dispersed phase flow rate on the X_d value (also) at a flow ratio of one ($U_c/U_d = 1.0$). This cannot be neglected, especially at low dispersed phase flows (U_d <0.5 centimeters/second).

Both equations (9 and 10) can be used for estimation of the average dispersed phase holdup within the pulsed column operating under conditions shown in Tables 4-1 and 4-2. However, at small flow rates and ratio $U_c/U_d = 1$, the equation 10 is more descriptive and precise than equation 9. The holdup value, required for evaluation of the uranium inventory in a column from the uranium concentration profile in this report, was calculated from equation 10.

4.2 Dual Process Column Operating with the Aqueous Phase Continuous

During PUREX nuclear fuel reprocessing, the solvent (TBP) quality will be degraded if it remains in contact with the highly radioactive solution of the first process cycle for an extensive period of time.[3] The dual process pulsed column used for the first uranium-plutonium coextraction and scrubbing can operate either with the organic or aqueous phase continuous.

The plant-size 1A (≡HS) column operating with the organic phase continuous has a total solvent (organic phase) residence time of approximately 23 to 25 minutes, including the time in the column working section above the organic phase inlet and in the top disengaging section. The solvent exposure to high radiation on the interface in the bottom disengaging section, separating the solvent (TBP) from the highly radioactive aqueous waste containing beta emitters, will last for hours, which may

result in a high TBP radiation damage. The solvent could contain unacceptable quantities of radiolysis products (MBP, DBP) resulting in higher lossess of uranium, plutonium and solvent, and in a decrease of the fission products decontamination factor.[3] As cited in the literature,[4, 5] the distribution of uranium and plutonium is significantly affected when the TBP radiation dosage exceeds about 2.0 watt-hour per liter. The radiation dosage at the bottom interface of the HS column could be expected to be about the same order of magnitude, when reprocessing highly irradiated and shortly cooled nuclear fuel.

To minimize solvent degradation due to the above long lasting exposure to high radiation, it would require either to redesign the HS dual column bottom disengaging section, or to operate the HS column with the aqueous phase continuous. The second alternative was experimentally tested and is described in this report.

The plant-size HS (≡1A) dual process column operating with the aqueous phase continuous would have an organic residence time of about 8 to 9 minutes, including the time in the top disengaging section.[3] The TBP exposure to radiation on the top interface in the top disengaging section will be negligible in comparison to the exposure in the bottom disengaging section when the fission products are concentrated.

Three experimental runs, simulating the plant-size column performance when operated with the aqueous phase continuous, were performed on the two-inch diameter 1A dual process column in the pilot plant. The 1A column was used for uranium extraction and scrubbing. Subsequent uranium stripping was performed on the three-inch diameter 1BX column, simulating the 1C plant column process. Both the 1A and 1BX columns operated with the interface located in the top disengaging section. Operating conditions and run data are presented in Table 4-3. Resultant data are summarized in Tables 4-4 through 4-9 and Figures 4-6 through 4-17.

All three runs (HSA-1, HSA-2, and HSA-3) resulted in an efficient extraction in the aqueous continuous 1A dual process column (see Table 4-4 through 4-6), showing an undetectable uranium concentration (<0.01 gram uranium/liter) in the raffinate (1AW) stream.

Comparing experimental results of columns operating under similar conditions (Figure 4-6), uranium concentration in the mid-section of the aqueous continuous column is about 17 to 20% higher than that of the organic continuous column.[1] The above extraction performance and higher concentration in the column mid-section are mainly caused by back mixing (axial mixing) of the continuous aqueous phase, which lowers (somewhat) the solute's concentration in the column bottom section.

Comparing data in Table 4-9 and Reference 1, the uranium inventory in the aqueous continuous 1A dual process column is about 35 to 40% lower than in the organic continuous column, due essentially to the low organic (dispersed) phase holdup within the column (about 15 to 25%).

The organic phase (30 v/o TBP) residence time in the column section from the organic bottom inlet to the top outlet (overflow) is about 6.5 minutes (Run HSA-1) operating the 1A column with the aqueous phase continuous. The organic phase residence time in the same section of the organic continuous column[1] is approximately 2.5 times longer than the above value, not taking into consideration the relatively stagnant organic layer between the bottom interface and organic inlet.

4.3 Concentration Profile

Solutes (uranium and nitric acid) concentration along the pulsed column was determined through sampling as shown in Section 3.2.2 and Reference 1. Resultant concentration profile data for each run are summarized in Tables 4-4 through 4-6 and presented in Figures 4-6 through 4-11.

During experimental runs, HSA-1 through HSA-3, the flow rate of all feed streams was changed as shown in Table 4-3. The intention was to maintain the same flow rate ratio in each process section during the above runs simulating the HS and 1C column conditions.[6] The experimental flow rate ratio in the 1A column, however, varied within the extraction section from O/A = 2.3 to 2.7, and in the scrubbing section from O/A = 6.9 to 8.0. The flow ratio in the 1BX stripping column varied within a range of O/A = 0.66 to 0.90.

Figure 4-6 shows the experimental uranium concentration profile along the 1A dual process column, in both the aqueous and organic phases, obtained during Run HSA-1. In this figure, concentration profiles obtained along the aqueous continuous column are compared with those determined along the organic continuous column[1] operating approximately under the same conditions. As shown in the diagram, uranium concentration in the organic phase around the aqueous solution (1AF) central inlet is about 20% higher when the 1A column operates with the aqueous phase continuous. About the same percentage increase in uranium concentration is shown also in the aqueous phase.

Figures 4-8 and 4-10 present uranium concentration profiles along the 1A column during Runs HSA-2 and HSA-3, respectively, simulating the HS column conditions.[6] Data from similar, comparative runs on the column, operating with the organic phase continuous and under the same flow conditions, are not available.

Figures 4-7, 4-9, and 4-11 show uranium concentration profiles along the 1BX column during Runs HSA-1, HSA-2, and HSA-3, respectively, simulating the 1C column conditions.[6] The concentration profiles are comparable with those obtained previously during Runs HS-4 through HS-6 in Reference 1.

All the above concentration profiles were used for determination of the uranium inventory in the corresponding column, as indicated in Section 3.2.3 and described in Reference 1.

4.4 Uranium Inventory in Pulsed Columns

The uranium inventory, i.e., its holdup within the pulsed column was determined through methods shown in Section 3.2.3. Both the "Volume-Concentration Measurement" and "Concentration Profile-Holdup" methods, described also in Reference 1, were applied for each test. The new "Mass Rate Measurement" method for evaluation of the solute's column inventory was used during Runs HSA-1 and HSA-3. Resultant data from all three methods are comparable as shown in Table 4-9.

For solute's inventory determination through the "Mass Rate Measurement" method, diagrams showing the "mass rate versus time" relationship in tested columns were determined experimentally (see Figures 4-12 through 4-15). Uranium feed rate (m_f) is constant in each diagram. Uranium mass rate in the combined effluent streams ($m_0 + m_A$) increases with time (t) up to the point when the operational steady state is reached. After that point, its value is constant. This curve was determined by taking periodically the effluent stream samples (in 20-minute intervals), starting from the moment when the aqueous feed (1AF) to the 1A column was turned on, and ending when the steady state was reached (in about 100 to 120 minutes). Samples were analyzed for uranium and nitric acid concentration and the corresponding uranium effluent mass-rate values, calculated from flow rate and concentration data, were then plotted in the diagram in Figures 4-12 through 4-15. Using Lagrange (semigraphical) interpolation method, curves expressing the change in the uranium effluent mass rate ($m_A + m_0$) with time were determined and plotted in the above figures.

Figures 4-12 and 4-14 show the uranium feed mass rate curve (m_f = constant) and effluent mass rate curve [$m_A + m_0 = \Psi(t)$] determined in the 1A dual process column during Runs HSA-1 and HSA-3, respectively. Using equation 4, the uranium inventory was calculated starting from time zero (0), when the 1AF aqueous feed was turned on, up to the time when the 1A column operated under steady-state conditions. The total uranium inventory in the 1A column is given in the above figures by the size of the surface area enclosed between both curves [m_f = constant and $m_A + m_0 = \Psi(t)$] and ordinate (vertical axis). Its value in the 1A column was at about $\Delta M_{(1A)}$ = 640 grams of uranium during Run HSA-1, and $\Delta M_{(1A)}$ = 670 grams of uranium during Run HSA-3.

Figures 4-13 and 4-15 present both experimental uranium mass rate curves [i.e., m_f = constant and $m_A + m_0 = \rho(t)$] required for calculations of the uranium inventory in the process cycle consisting of the 1A and 1BX columns during Runs HSA-1 and HSA-3, respectively. Using equation 6, the uranium inventory was calculated starting from time zero (0), when the 1AF aqueous feed to the 1A column was turned on, up to the operational steady-state time in the entire cycle. The total uranium inventory in both columns of the process cycle is expressed graphically by the surface area enclosed between curves m_f = constant and $m_A + m_0 = \rho(t)$, and ordinate. The value of the total uranium inventory in both columns was about $\Delta M_{(1A\ \&\ 1B)}$ = 1670 grams of uranium during Run HSA-1 and about 1580 grams of uranium during Run HSA-3.

The uranium inventory in the 1BX stripping column was determined by subtracting the 1A column inventory value from the total inventory in the process cycle.

All calculated inventory data for Runs HSA-1 and HSA-3 are shown in Tables 4-7 and 4-8, respectively. By plotting these data in the diagram in Figures 4-16 and 4-17, curves were obtained describing graphically the increase of the uranium inventory with time in both columns (separately), as well as in the entire cycle during Runs HSA-1 and HSA-3, respectively. The above figures indicate that the 1A column operated under steady-state conditions in about 95 minutes during Run HSA-1, and in about 80 minutes during Run HSA-3. In both cases, the operational steady state of the 1BX column (and entire process cycle) was achieved in about 20 minutes after the 1A column started to operate steadily.

All measured and calculated total uranium inventory data in tested columns are summarized in Table 4-9. The uranium inventory values in the 1A (≡HS) column, determined by using the above three methods, correlate well with data obtained from the derived equation.

$$\Delta M_{(1A)} \equiv \Delta M_{(HS)} \cong 0.28\ V_{col} \cdot \frac{v_f}{v_{St}} \cdot C_f \pm 5\% \qquad \ldots \underline{11}.$$

The equation is similar to that presented in Reference 1 which applies to the HS column operating with the organic phase continuous, except of the lower coefficient value (of 0.28). The above uncertainties of equation 9 indicate the difference between the calculated and measured value of the uranium inventory in the HS column operated with the aqueous phase continuous.

The 1A (≡HS) column uranium inventory data in Table 4-9 are about 35 to 40% lower than data shown in Reference 1 for the column operating with the organic phase continuous. The 1C stripping column (1BX column) inventory data in the table are about the same as in Reference 1. They correlate well with data calculated by use of equation:[1]

$$\Delta M_{(1BX)} \equiv \Delta M_{(1C)} \cong 0.45\ V_{col} \cdot \frac{v_f}{v_{St}} \cdot C_f \qquad \ldots \underline{12}.$$

TABLE 4-1

DISPERSED PHASE HOLDUP TEST DATA, ORGANIC PHASE CONTINUOUS

Superficial Velocity		Flow Ratio	Velocity Difference	Pulse Frequency	Pulse Amplitude	Pulse Velocity	Dispersed Phase Holdup: X_d (%)			
U_c ($=U_O$) (cm/sec)	U_d ($=U_A$) (cm/sec)	U_c/U_d	ΔU_{d-c} ($=U_d - U_c$) (cm/sec)	f (1/min)	A_O (cm)	f x A_O (cm/sec)	Experimental	From W. R. Readings	Calculated (Equation 8)	Calculated (Equation 7)
0.30	0.30	1.0	0.0	65	1.8	1.95	~7.0	--	7.9	8.3
				75		2.25	11.0	13.0	11.8	12.4
				85		2.55	15.9	--	16.8	17.7
0.43	0.43	1.0	0.0	55	1.8	1.65	~6.0	--	5.1	5.2
				65		1.95	8.0	--	8.2	8.3
				75		2.25	12.0	13.0	12.3	12.4
				85		2.25	18.3	--	17.5	17.7
0.56	0.56	1.0	0.0	55	1.8	1.65	~6.6	--	5.3	5.2
				65		1.95	9.4	--	8.5	8.3
				75		2.25	13.0	13.5	12.7	12.4
				85		2.55	19.0	--	18.0	17.7
0.22	0.65	0.33	0.43	55	2.0	1.83	~10.4	--	9.9	9.6
				65		2.17	14.2	--	15.2	14.7
				75		2.50	20.7	--	21.8	21.1
0.28	0.85	0.33	0.57	55	2.0	1.83	~10.9	--	11.1	10.5
				65		2.17	16.0	--	16.8	15.8
				75		2.50	22.0	--	24.0	22.6
0.35	1.04	0.33	0.69	55	2.0	1.83	~13.0	--	12.3	11.4
				65		2.17	18.0	--	18.5	17.0
0.66	0.22	3.0	-0.43	55	2.0	1.83	~5.8	--	4.5	5.0
				65		2.17	7.2	8.5	7.7	8.3
				75		2.50	10.6	--	11.9	13.0
0.85	0.28	3.0	-0.57	55	2.0	1.83	~6.0	--	4.1	4.5
				65		2.17	8.0	8.5	7.2	7.6
				75		2.50	12.5	--	11.3	11.9
1.05	0.35	3.0	-0.70	55	2.0	1.83	~5.1	--	3.7	4.0
				65		2.17	8.2	8.0	6.7	6.9
				75		2.50	12.0	--	10.6	11.0

TABLE 4-2

DISPERSED PHASE HOLDUP TEST DATA, AQUEOUS PHASE CONTINUOUS

Superficial Velocity		Flow Ratio	Velocity Difference	Pulse Frequency	Pulse Amplitude	Pulse Velocity	Dispersed Phase Holdup: X_d (%)			
U_c ($=U_A$) (cm/sec)	U_d ($=U_O$) (cm/sec)	U_c/U_d	ΔU_{d-c} ($=U_d - U_c$) (cm/sec)	f (1/min)	A_O (cm)	f x A_O (cm/sec)	Experimental	From W. R. Readings	Calculated (Equation 10)	Calculated (Equation 9)
0.52	0.52	1.0	0.0	55	2.2	2.02	~10.0	--	9.2	9.2
				65		2.38	14.0	13.5	14.7	14.7
				75		2.75	21.1	--	22.0	21.9
				85		3.12	32.0	--	31.2	31.0
0.78	0.78	1.0	0.0	55	2.2	2.02	~10.3	--	9.6	9.2
				65		2.38	15.2	14.0	15.4	14.7
				75		2.75	22.8	--	23.0	21.9
1.04	1.04	1.0	0.0	55	2.2	2.02	~10.9	--	9.9	9.2
				65		2.38	16.5	16.0	15.8	14.7
0.26	0.78	0.33	0.52	45	2.4	1.80	~10.5	--	10.3	9.8
				55		2.20	17.0	--	16.9	16.0
				65		2.60	25.6	--	25.7	24.4
0.39	1.18	0.33	0.79	45	2.4	1.80	~13.0	--	12.7	11.6
				55		2.20	21.0	--	20.3	18.5
0.52	1.56	0.33	1.04	45	2.4	1.80	~14.0	--	15.4	13.6
				55		2.20	23.0	--	24.1	21.2
0.78	0.26	3.0	-0.52	55	2.2	2.02	~5.6	--	5.8	6.3
				65		2.38	9.0	9.5	9.9	10.6
				75		2.75	17.0	--	15.4	16.1
1.17	0.39	3.0	-0.78	55	2.4	2.20	~7.5	--	6.6	6.8
				65		2.60	10.2	9.5	11.6	11.9
*0.526	0.426	1.23	-0.1	60	2.3	2.3	~13.3	~13.0	12.4	11.5
*0.426	0.386	1.10	-0.04	60	2.3	2.3	14.6	14.0	14.2	13.3
*0.357	0.237	1.50	-0.12	60	2.3	2.3	12.8	--	11.4	12.3

*Holdup measured during mass-transfer tests in the stripping column.

TABLE 4-3

RUN CONDITIONS AND DATA
(Runs: HSA-1, HSA-2, and HSA-3)

Column	Stream	Phase	Concentration U (g/l)	Concentration HNO_3 (M)	Flow Rate: (ml/min) Run HSA-1	Run HSA-2	Run HSA-3
HS Column (1A Column)	HSS (1AS)	A	0.0	3.04	150	125	90
	HSF (1AF)	A	271.4	2.66	290	240	175
	HSX (1AX)	O	0.0	0.0	1110	1000	620
1C Column (1BX Column)	1CX (1BX)	A	0.0	0.0	1380	1100	940
HS Column (1A Column)	Pulse Frequency: (c/min)				56	70	80
	Pulse Amplitude: (cm)				~2.1	2.1	2.1
	Dispersed Phase Holdup: (%)	Scrubbing			(15.8)	(25.5)	(29.1)
		Extraction			(13.6)	(22.9)	(27.1)
		Average (Scrub & Extr)			~15.6	~23.5	~28.2
	Pulse Frequency: (c/min)				60	60	60
	Pulse Amplitude: (cm)				~2.3	2.3	2.3
	Dispersed Phase Holdup: (%)				~13.3	~14.6	~12.8
HS & 1C Columns	Temperature: (°C)				~23	~23	~23

NOTE: Holdup values in parentheses were calculated from equation 10.

TABLE 4-4

CONCENTRATION PROFILE DATA
(Run: HSA-1)

HS Column					1C Column				
	Aqueous		Organic			Aqueous		Organic	
Sample	U (g/l)	HNO_3 (M)	U (g/l)	HNO_3 (M)	Sample	U (g/l)	HNO_3 (M)	U (g/l)	HNO_3 (M)
End Stream	(HSS) 0.0	(HSS) 3.04	(HSP) ~69.04	(HSP) ~0.22	End Stream	(1CX) 0.0	(1CX) 0.0	(1CW) <0.01	(1CW) <0.001
A-1	~8.0	2.99	69.6	0.21	B-1	<0.01	<0.01	<0.01	<0.001
A-2	--	--	--	--	B-2	--	--	--	--
A-3	20.6	3.06	77.9 78.7	0.23	B-3	--	--	--	--
A-4	--	--	--	--	B-4	16.7 17.3	0.03	14.9 15.6	0.003
A-5	23.9 24.3	3.3	91.5 91.9	0.26	B-5	36.3 36.9	0.06	42.8 43.6	0.02
A-6	22.3 22.7	3.4	91.1 91.6	0.27	B-6	50.1 50.3	0.11	59.7 60.4	0.07
A-7	19.6 20.5	3.44	75.4 72.3	0.4	B-7	56.0	0.20	69.0	0.17
A-8	--	--	--	--	End Stream	(1CU) ~56.2	(1CU) 0.20	(HSP) 69.04	(HSP) 0.22
A-9	1.84	3.46	8.6	0.60					
A-10	--	--	--	--					
A-11	0.014	2.60	0.08	0.37					
End Stream	(HSW) <0.01	(HSW) 2.20	(HSX) 0.0	(HSX) 0.0					

TABLE 4-5

CONCENTRATION PROFILE DATA
(Run: HSA-2)

HS Column					1C Column				
	Aqueous		Organic			Aqueous		Organic	
Sample	U (g/l)	HNO_3 (M)	U (g/l)	HNO_3 (M)	Sample	U (g/l)	HNO_3 (M)	U (g/l)	HNO_3 (M)
End Stream	(HSS) 0.0	(HSS) 3.04	(HSP) ~63.0	(HSP) 0.23	End Stream	(1CX) 0.0	(1CX) 0.0	(1CW) <0.01	(1CW) <0.001
A-1	~7.6	2.99	65.1	0.22	B-1	<0.01	<0.01	<0.01	<0.001
A-2	--	--	--	--	B-2	--	--	--	--
A-3	18.2 19.1	3.1	75.2 76.8	0.21	B-3	--	--	--	--
A-4	--	--	--	--	B-4	19.2 19.8	0.01	20.1 20.4	0.016
A-5	19.4 20.2	3.28	79.2 79.9	0.23	B-5	37.5 37.9	0.02	43.1 43.8	0.023
A-6	19.1 19.9	3.30	79.0 79.8	0.25	B-6	50.1 50.7	0.09	58.6 58.3	0.04
A-7	13.5	3.45	50.4 54.7	0.36	B-7	57.0 57.3	0.19	62.8	0.12
A-8	--	--	--	--	End Stream	(1CU) 57.4	(1CU) 0.208	(HSP) 63.0	(HSP) 0.23
A-9	0.4	3.4	2.1	0.53					
A-10	--	--	--	--					
A-11	0.012	2.53	0.02	0.34					
End Stream	(HSW) <0.01	(HSW) 2.14	(HSX) 0.0	(HSX) 0.0					

TABLE 4-6

CONCENTRATION PROFILE DATA
(Run: HSA-3)

	HS Column					1C Column				
	Aqueous		Organic			Aqueous		Organic		
Sample	U (g/l)	HNO_3 (M)	U (g/l)	HNO_3 (M)	Sample	U (g/l)	HNO_3 (M)	U (g/l)	HNO_3 (M)	
End Stream	(HSS) 0.0	(HSS) 3.04	(HSP) ~75.0	(HSP) ~0.24	End Stream	(1CX) 0.0	(1CX) 0.0	(1CW) <0.01	(1CW) <0.001	
A-1	~8.4	2.94	76.2	0.23	B-1	<0.01	<0.01	<0.01	<0.001	
A-2	--	--	--	--	B-2	--	--	--	--	
A-3	18.1	3.12	79.2 79.8	0.26	B-3	--	--	--	--	
A-4	--	--	--	--	B-4	16.3 16.9	0.015	15.1 15.3	0.02	
A-5	19.5 20.4	3.29	82.0 82.4	0.28	B-5	34.1 34.8	0.021	43.7 44.0	0.023	
A-6	19.4 18.3	3.31	80.7 81.4	0.28	B-6	46.4 46.8	0.04	63.3 65.0	0.07	
A-7	12.2	3.50	52.1 59.3	0.35	B-7	49.7 50.1	0.15	74.4 74.6	0.17	
A-8	--	--	--	--	End Stream	(1CU) ~50.2	(1CU) ~0.16	(HSP) 75.0	(HSP) 0.24	
A-9	0.30	3.51	1.14	0.50						
A-10	--	--	--	--						
A-11	<0.01	2.61	<0.01	0.37						
End Stream	(HSW) <0.01	(HSW) 2.21	(HSX) 0.0	(HSX) 0.0						

TABLE 4-7

URANIUM INVENTORY DATA (RUN HSA-1)

		1A (≡HS) Column			1A and 1BX Columns			1BX (≡1C) Column
Time (min) (Δt = 5 min)	(i)	Δm_{i-1} (gU/min)	Δm_i (gU/min)	$\Delta M_{(1A)}$ (Equation 4) (grams U)	Δm^*_{i-1} (gU/min)	Δm^*_i (gU/min)	$\Delta M_{(1A \& 1BX)}$ (Equation 6) (grams U)	$\Delta M_{(1BX)}$ (grams U)
5	(1)	~78.7	~28.8	~268.8	~78.7	~58.7	~343.5	~74.7
10	(2)	28.8	16.7	382.5	58.7	46.6	606.8	224.3
15	(3)	16.7	11.7	453.5	46.6	37.2	816.3	362.8
20	(4)	11.7	8.2	503.2	37.2	29.6	983.3	480.1
25	(5)	8.2	6.2	539.2	29.6	23.7	1116.5	577.3
30	(6)	6.2	4.7	566.5	23.7	19.1	1223.5	657.0
35	(7)	4.7	3.7	587.5	19.1	15.7	1310.5	723.0
40	(8)	3.7	2.8	603.8	15.7	12.6	1381.3	777.5
45	(9)	2.8	1.7	615.0	12.6	10.7	1439.5	824.5
50	(10)	1.7	1.1	622.0	10.7	8.6	1487.8	865.8
55	(11)	1.1	0.7	626.5	8.6	6.9	1526.5	900.0
60	(12)	0.7	0.55	629.6	6.9	5.6	1557.8	928.2
65	(13)	0.55	0.40	632.0	5.6	4.7	1583.5	951.5
70	(14)	0.40	0.35	633.9	4.7	3.6	1604.3	970.4
75	(15)	0.35	0.30	635.5	3.6	3.1	1621.3	985.8
80	(16)	0.30	0.25	636.9	3.1	2.5	1635.5	998.6
85	(17)	0.25	0.20	638.0	2.5	1.9	1646.5	1008.5
90	(18)	0.20	0.15	638.9	1.9	1.3	1654.5	1015.6
95	(19)	0.15	0.10	639.5	1.3	0.7	1659.5	1020.0
100	(20)	0.10	0.05	639.9	0.7	0.4	1662.3	1022.4
105	(21)	0.05	0.0	640.0	0.4	0.2	1664.3	1024.3
110	(22)	0.0	0.0	640.0	0.2	0.1	1665.0	1025.0
115	(23)				0.1	0.05	1665.3	1025.3
120	(24)				0.05	0.0	1665.5	1025.5
125	(25)				0.0	0.0	1665.5	1025.5

TABLE 4-8

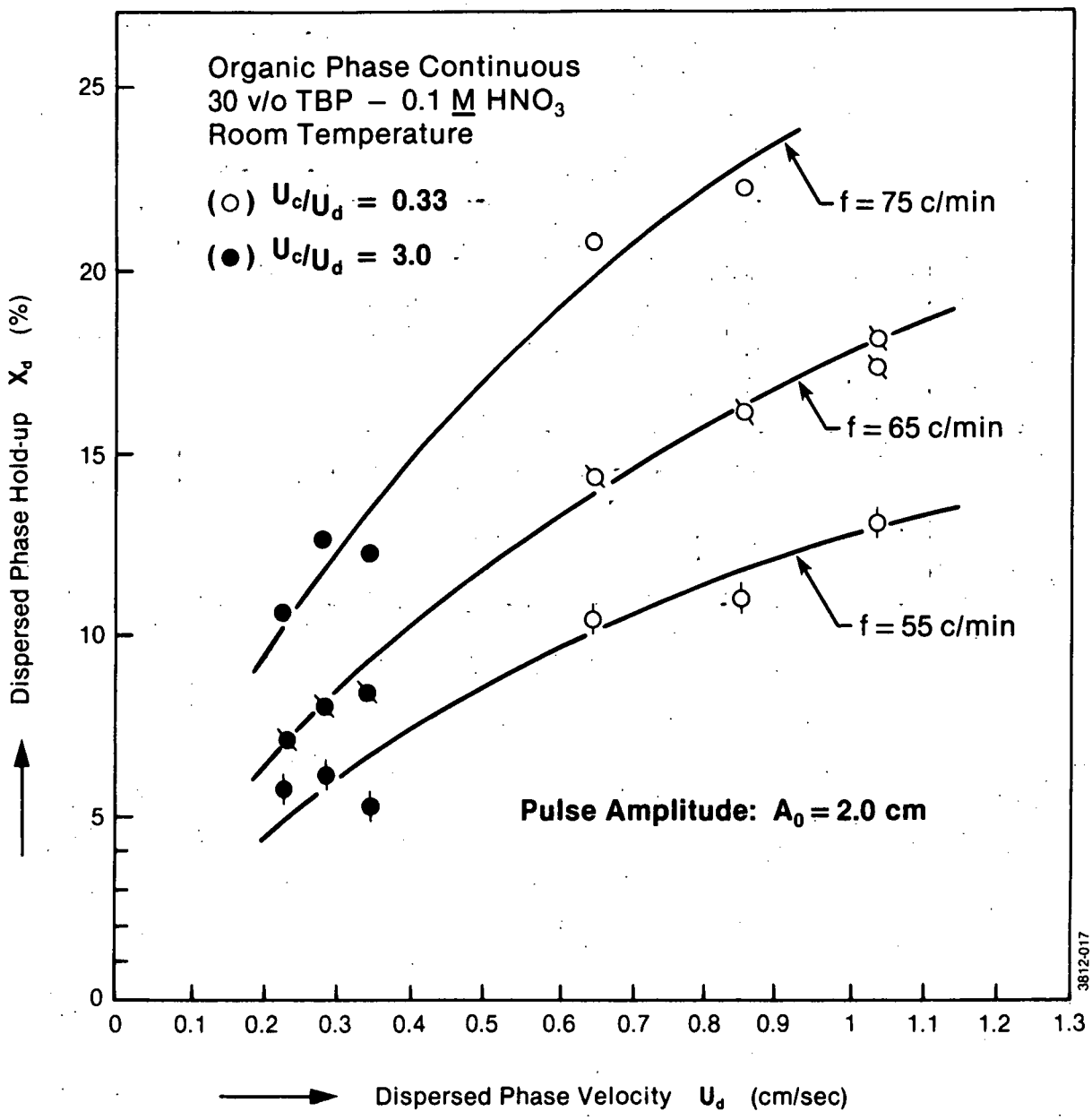
URANIUM INVENTORY DATA (RUN HSA-3)

		1A (≡HS) Column			1A and 1BX Columns			1BX (≡1C) Column
Time (min) (Δt = 5 min)	(i)	Δm_{i-1} (gU/min)	Δm_i (gU/min)	$\Delta M_{(1A)}$ (Equation 4) (grams U)	Δm^*_{i-1} (gU/min)	Δm^*_i (gU/min)	$\Delta M_{(1A\ \&\ 1BX)}$ (Equation 6) (grams U)	$\Delta M_{(1BX)}$ (grams U)
5	(1)	~47.5	~28.0	~188.8	~47.5	~42.5	~225.0	~36.2
10	(2)	28.0	20.0	308.7	42.5	37.9	426.0	117.3
15	(3)	20.0	14.5	395.0	37.9	33.5	604.5	209.5
20	(4)	14.5	11.2	459.3	33.5	29.9	763.0	303.7
25	(5)	11.2	8.8	509.2	29.9	26.3	903.5	394.3
30	(6)	8.8	6.9	548.5	26.3	22.9	1026.5	478.0
35	(7)	6.9	5.5	579.5	22.9	19.8	1133.3	553.8
40	(8)	5.5	3.9	603.0	19.8	16.9	1225.0	622.0
45	(9)	3.9	3.0	620.3	16.9	14.3	1303.0	682.7
50	(10)	3.0	2.5	634.0	14.3	11.9	1368.5	734.5
55	(11)	2.5	2.0	645.3	11.9	9.8	1422.8	777.5
60	(12)	2.0	1.6	654.3	9.8	7.9	1467.0	812.7
65	(13)	1.6	1.2	661.3	7.9	6.0	1501.8	840.5
70	(14)	1.2	0.8	666.2	6.0	4.4	1527.8	861.6
75	(15)	0.8	0.4	669.3	4.4	3.1	1546.5	877.2
80	(16)	0.4	0.1	670.5	3.1	1.9	1559.0	888.5
85	(17)	0.1	0.0	670.7	1.9	1.3	1567.0	896.3
90	(18)	0.0	0.0	670.7	1.3	0.6	1571.8	901.1
95	(19)				0.6	0.3	1574.0	903.3
100	(20)				0.3	0.1	1575.0	904.3
105	(21)				0.1	0.0	1575.2	904.5
110	(22)				0.0	0.0	1575.2	904.5

TABLE 4-9

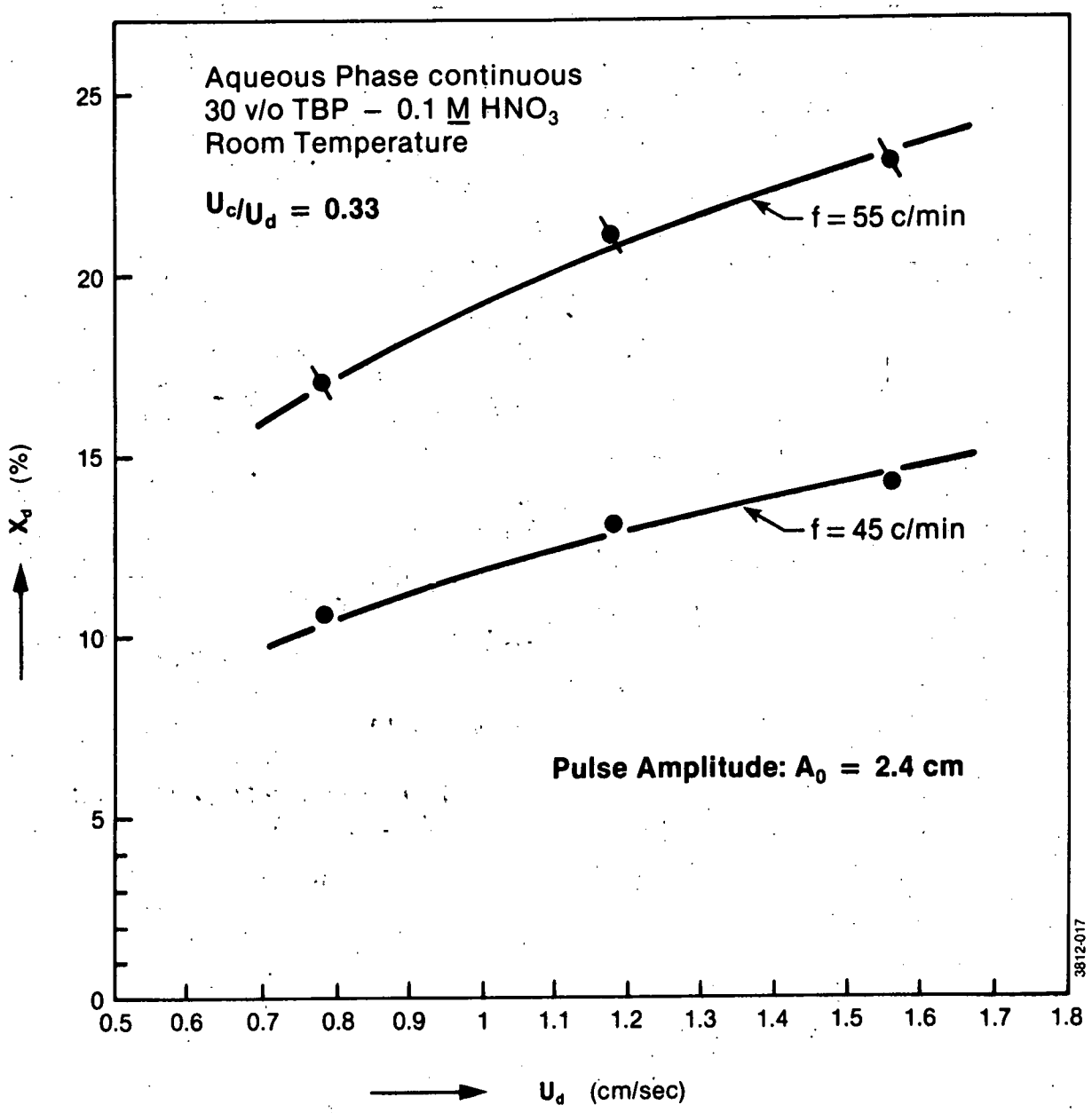
URANIUM INVENTORY IN TESTED COLUMNS (SUMMARY)

Column	Method	Uranium Inventory (grams) Run HSA-1	Run HSA-2	Run HSA-3
1A Column (≡HS Column)	1. Volume-Concentration Measurement	~605	~560	~650
	2. Concentration Profile-Holdup Data	590	550	635
	3. Mass Rate Measurement	640	--	670
	4. Equation 11 (Calculated)	(610)	(560)	(660)
1BX Column (≡1C Column)	1. Volume-Concentration Measurement	~1020	~1070	~860
	2. Concentration Profile-Holdup Data	990	1020	915
	3. Mass Rate Measurement	1025	--	905
	4. Equation 12 (Calculated)	(1000)	(1010)	(890)



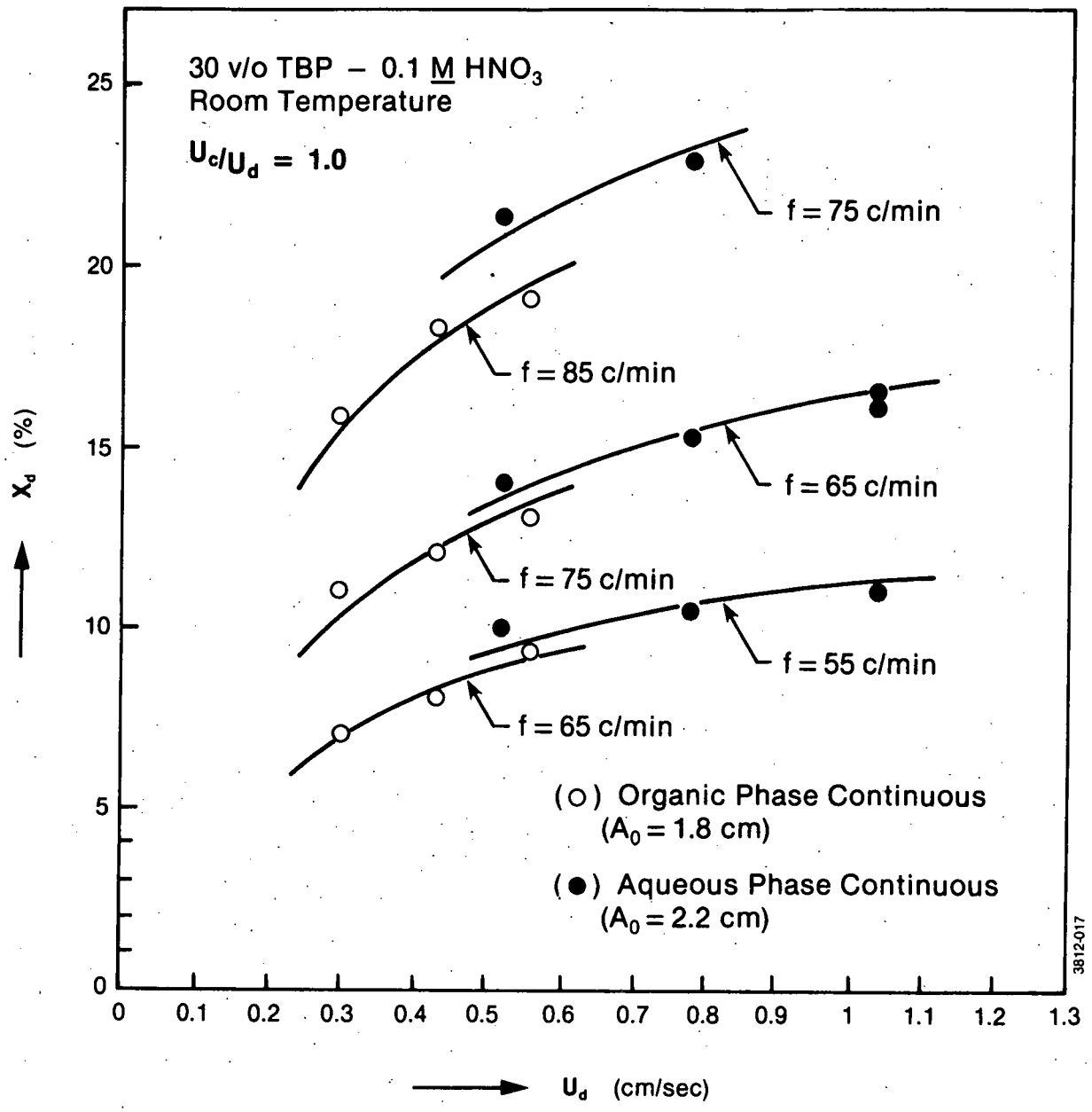
DISPERSED PHASE HOLDUP X_d VERSUS
THE DISPERSED PHASE SUPERFICIAL VELOCITY
(ORGANIC CONTINUOUS)

FIGURE 4-1



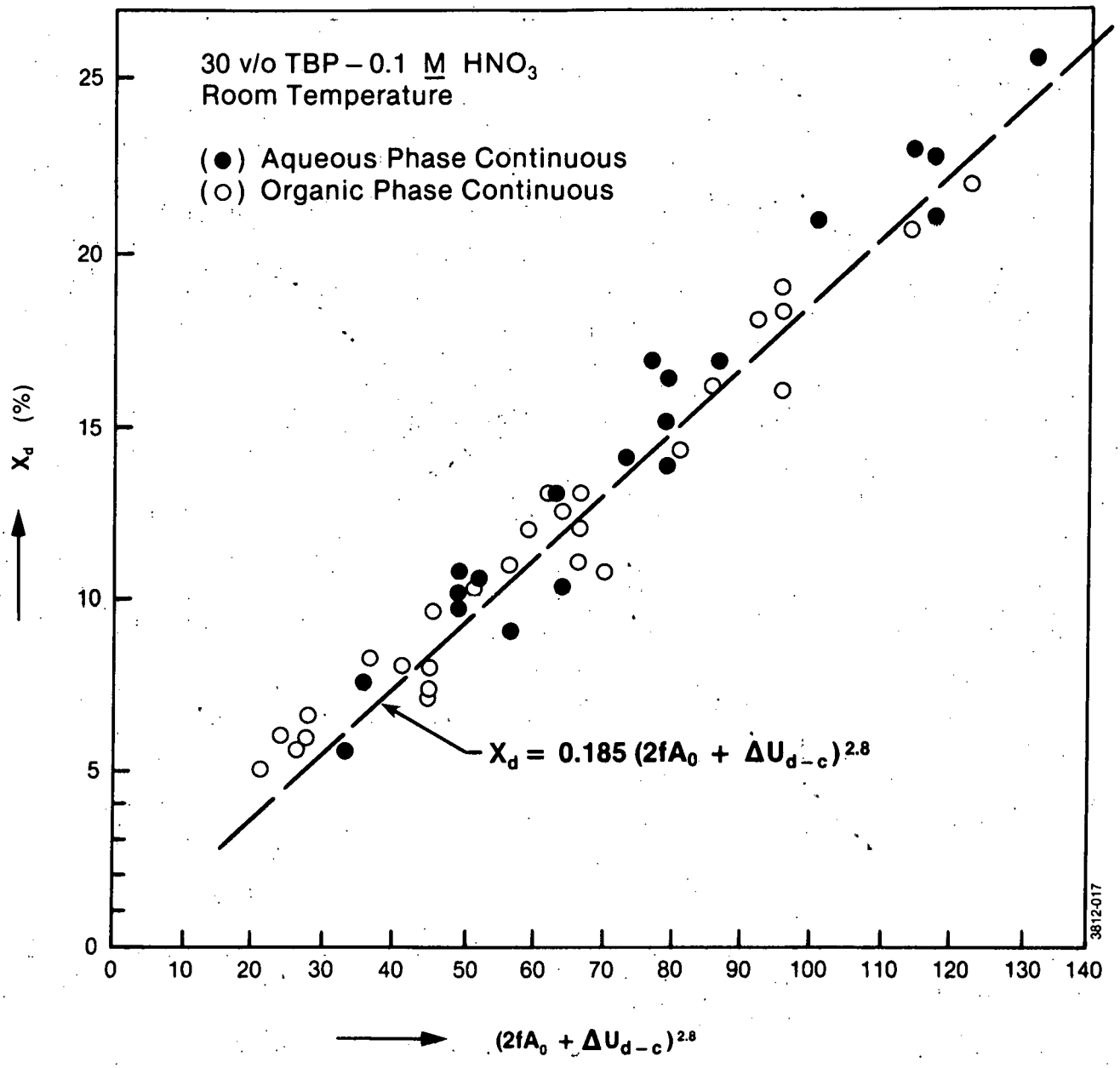
DISPERSED PHASE HOLDUP X_d VERSUS
THE DISPERSED PHASE SUPERFICIAL VELOCITY
(AQUEOUS CONTINUOUS)

FIGURE 4-2



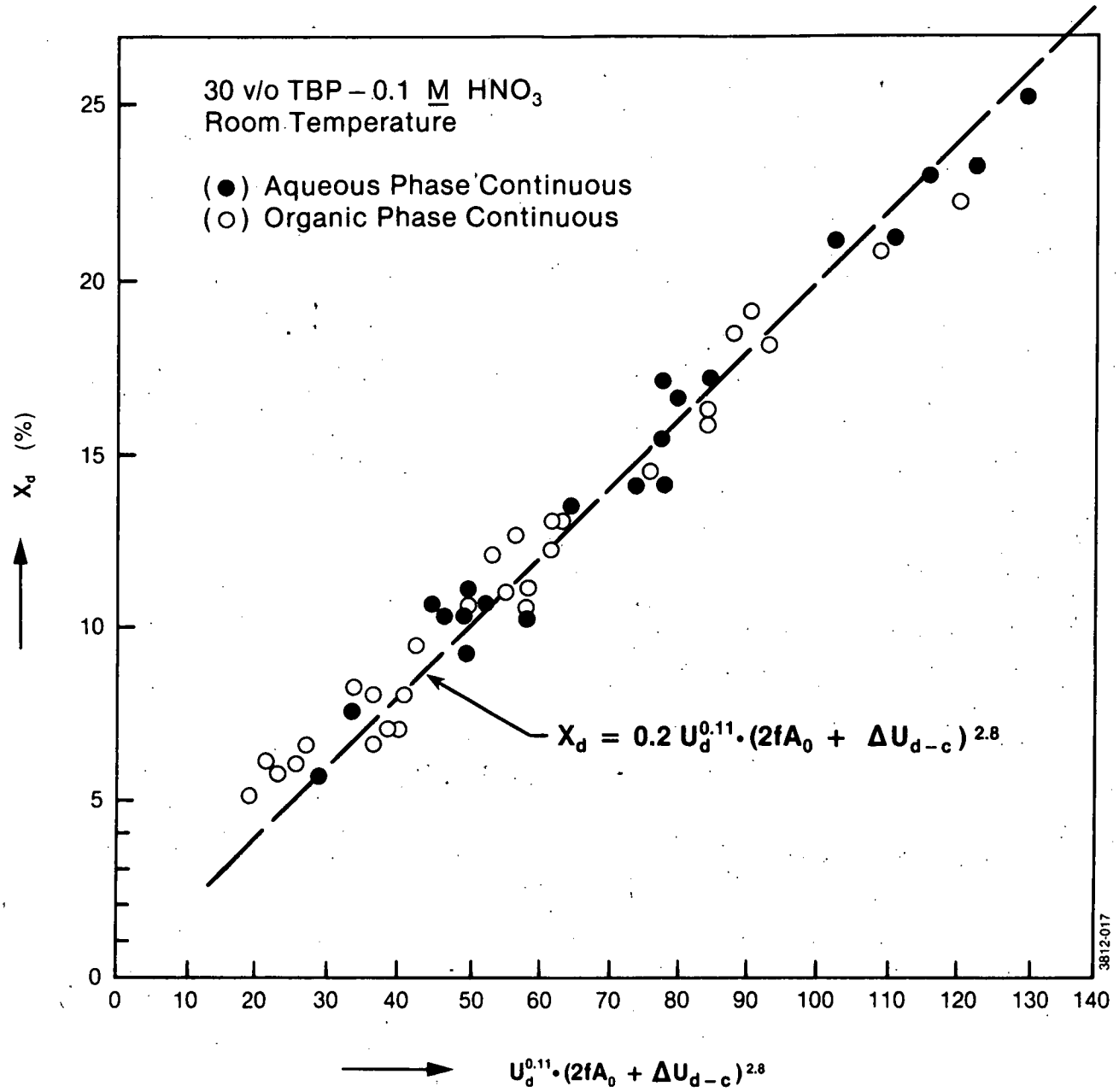
DISPERSED PHASE HOLDUP X_d VERSUS
THE DISPERSED PHASE SUPERFICIAL VELOCITY
(FLOW RATE U_c/U_d=1)

FIGURE 4-3



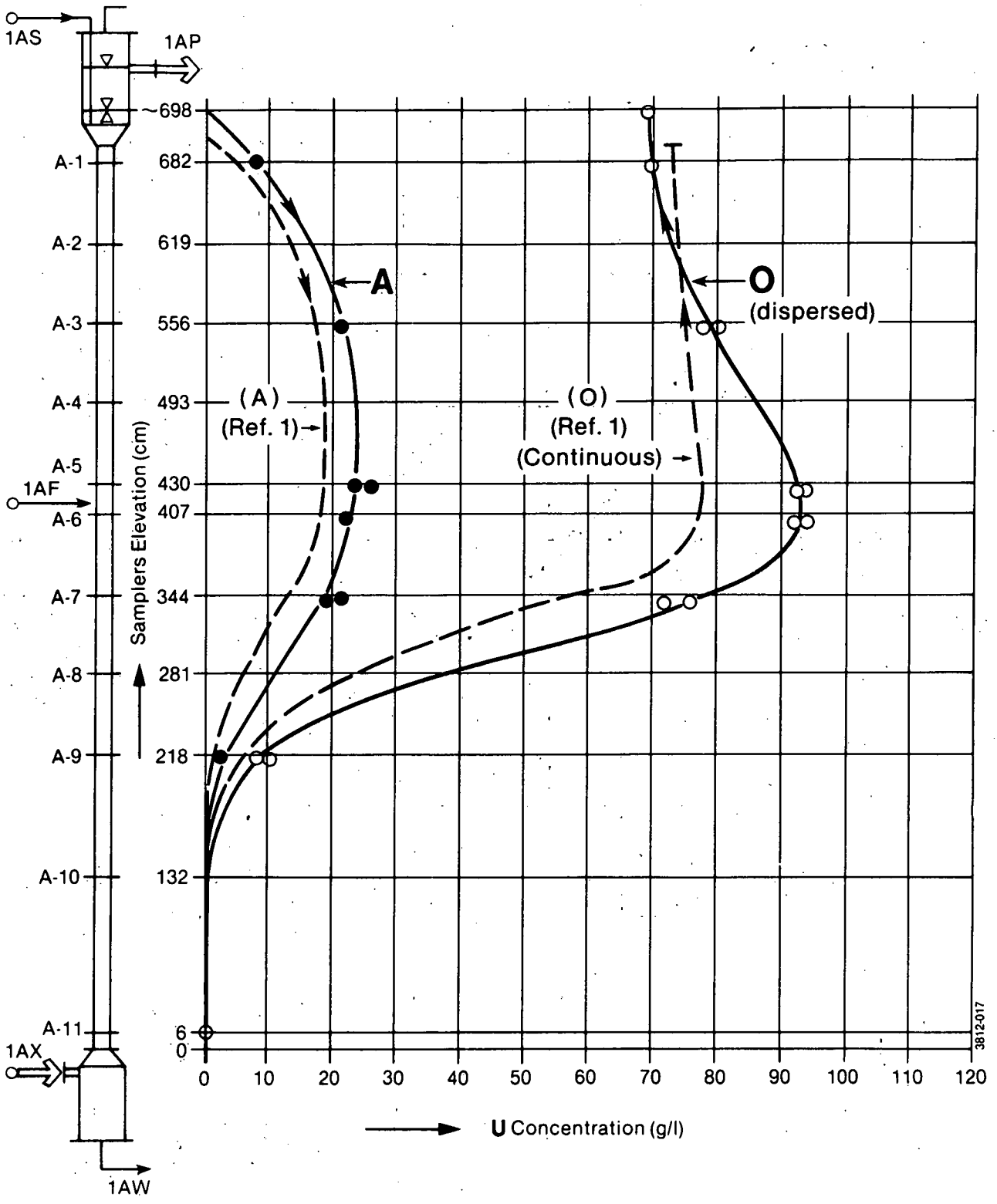
DISPERSED PHASE HOLDUP X_d AS FUNCTION OF EXPRESSION $(2fA_o + \Delta U_{d-c})^{2.8}$

FIGURE 4-4



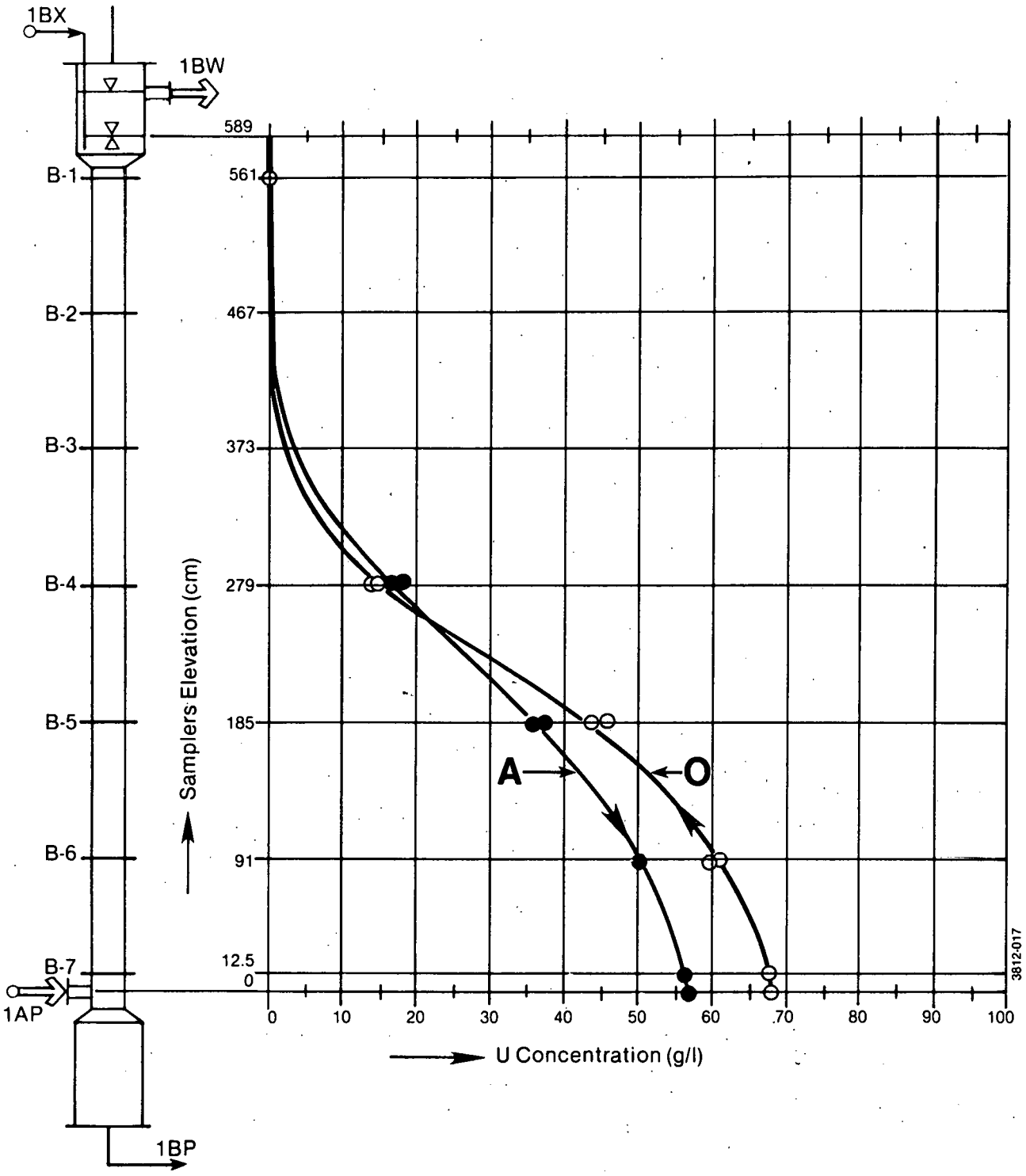
DISPERSED PHASE HOLDUP X_d AS FUNCTION OF EXPRESSION $U_d^{0.11} \cdot (2fA_o + \Delta U_{d-c})^{2.8}$

FIGURE 4-5



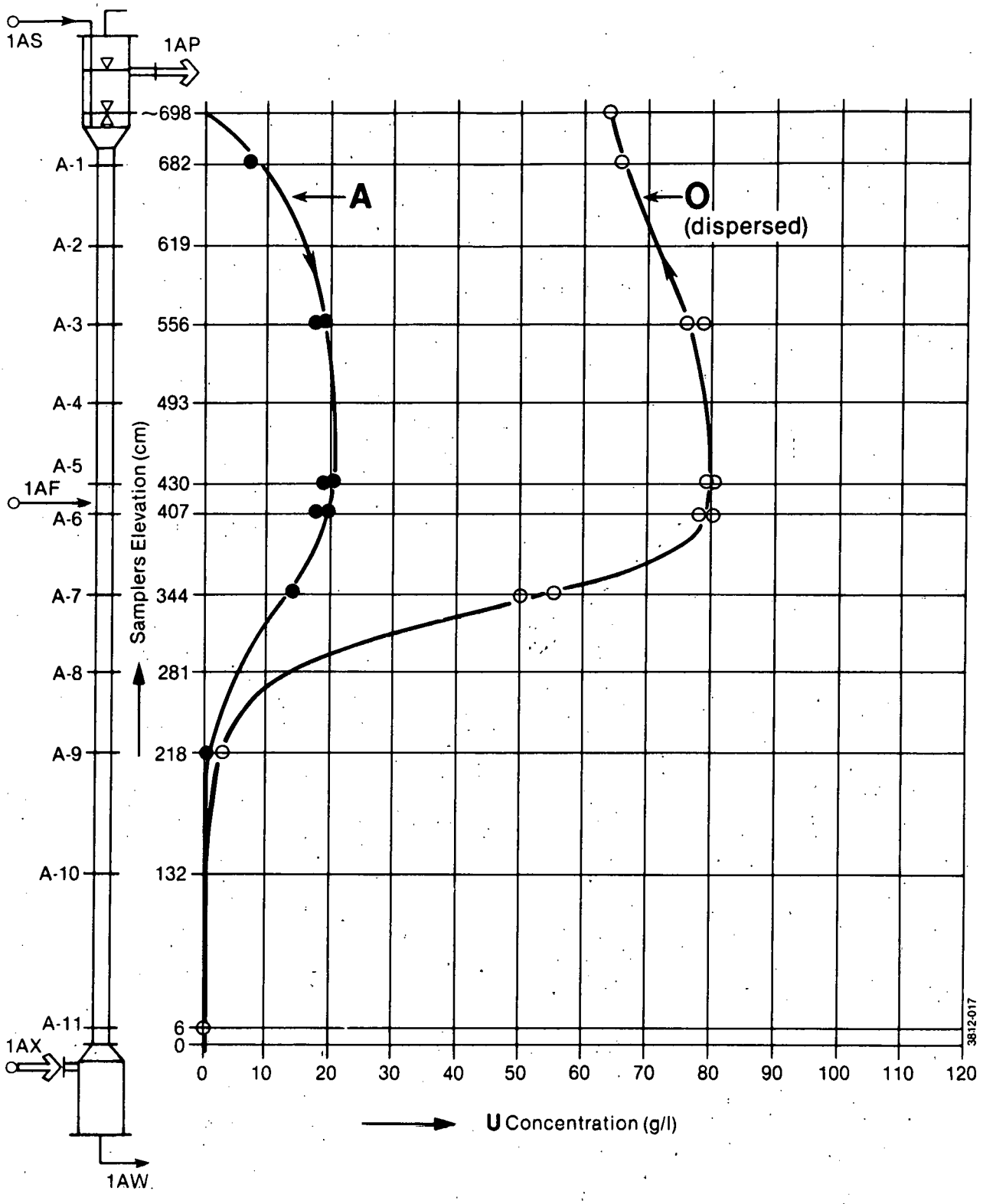
THE 1A (≡HS) COLUMN EXPERIMENTAL CONCENTRATION PROFILES FOR RUN HSA-1

FIGURE 4-6



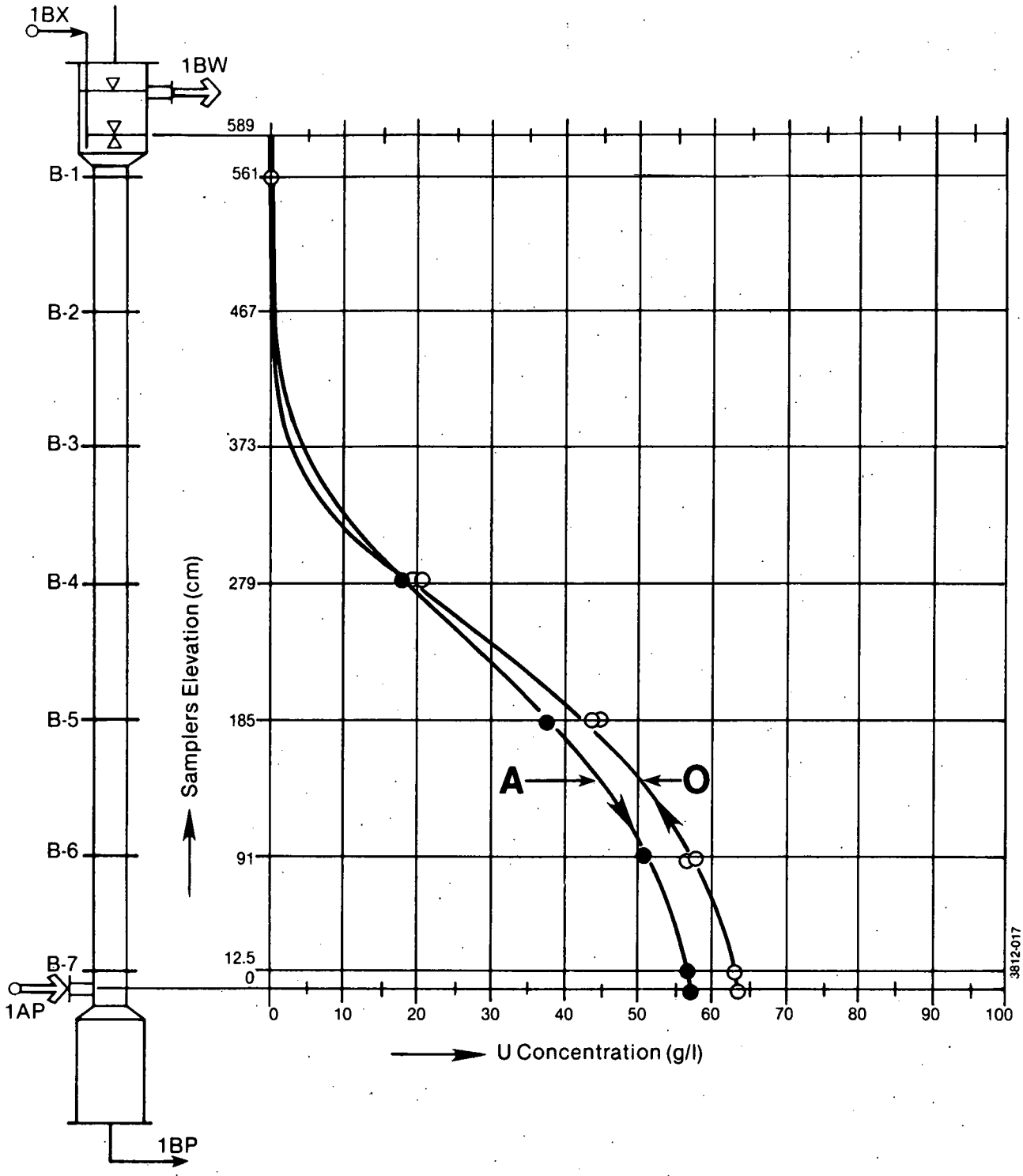
THE 1BX (≡1C) COLUMN EXPERIMENTAL CONCENTRATION PROFILES FOR RUN HSA-1

FIGURE 4-7



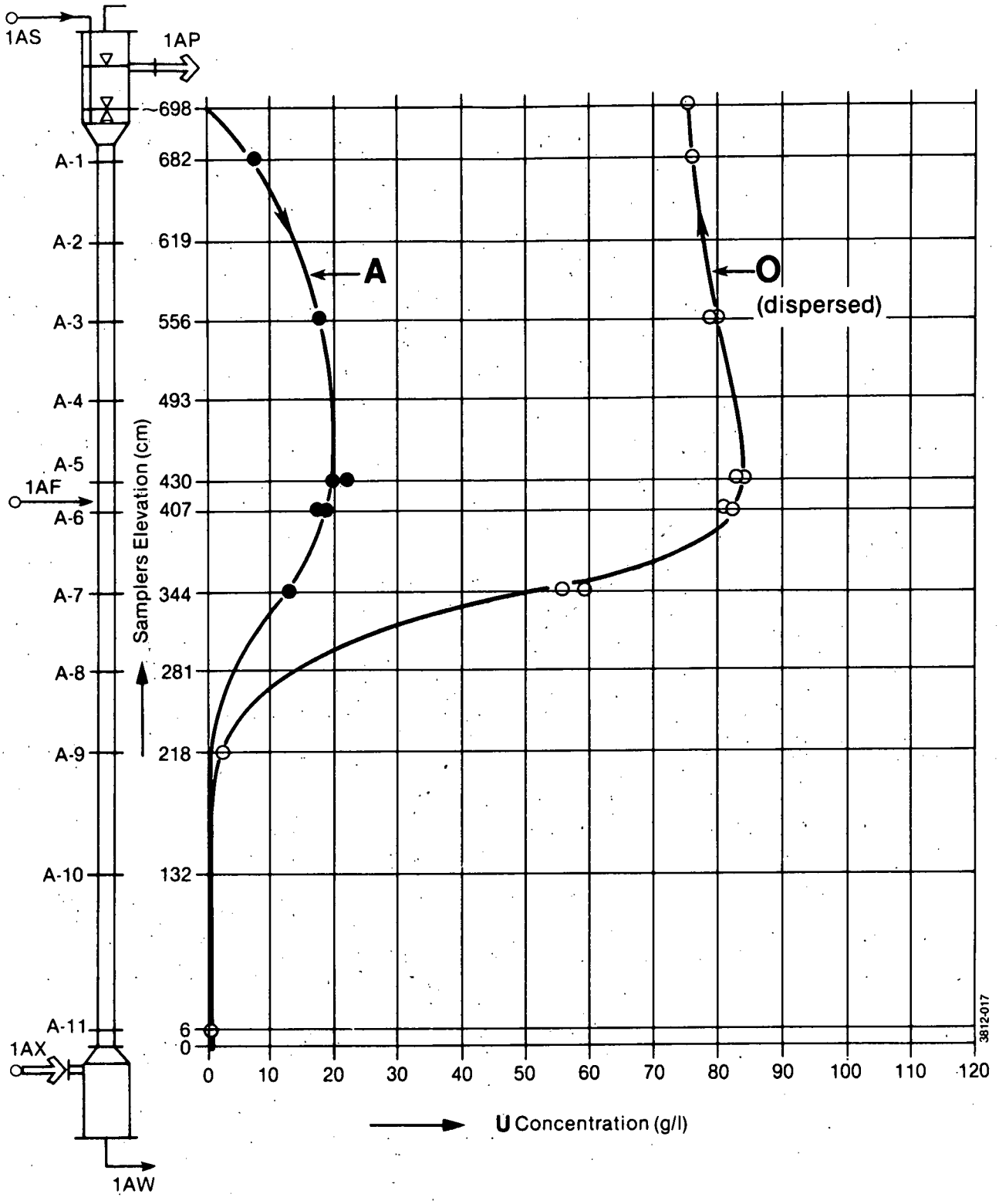
THE 1A (≡HS) COLUMN EXPERIMENTAL CONCENTRATION PROFILES FOR RUN HSA-2

FIGURE 4-8



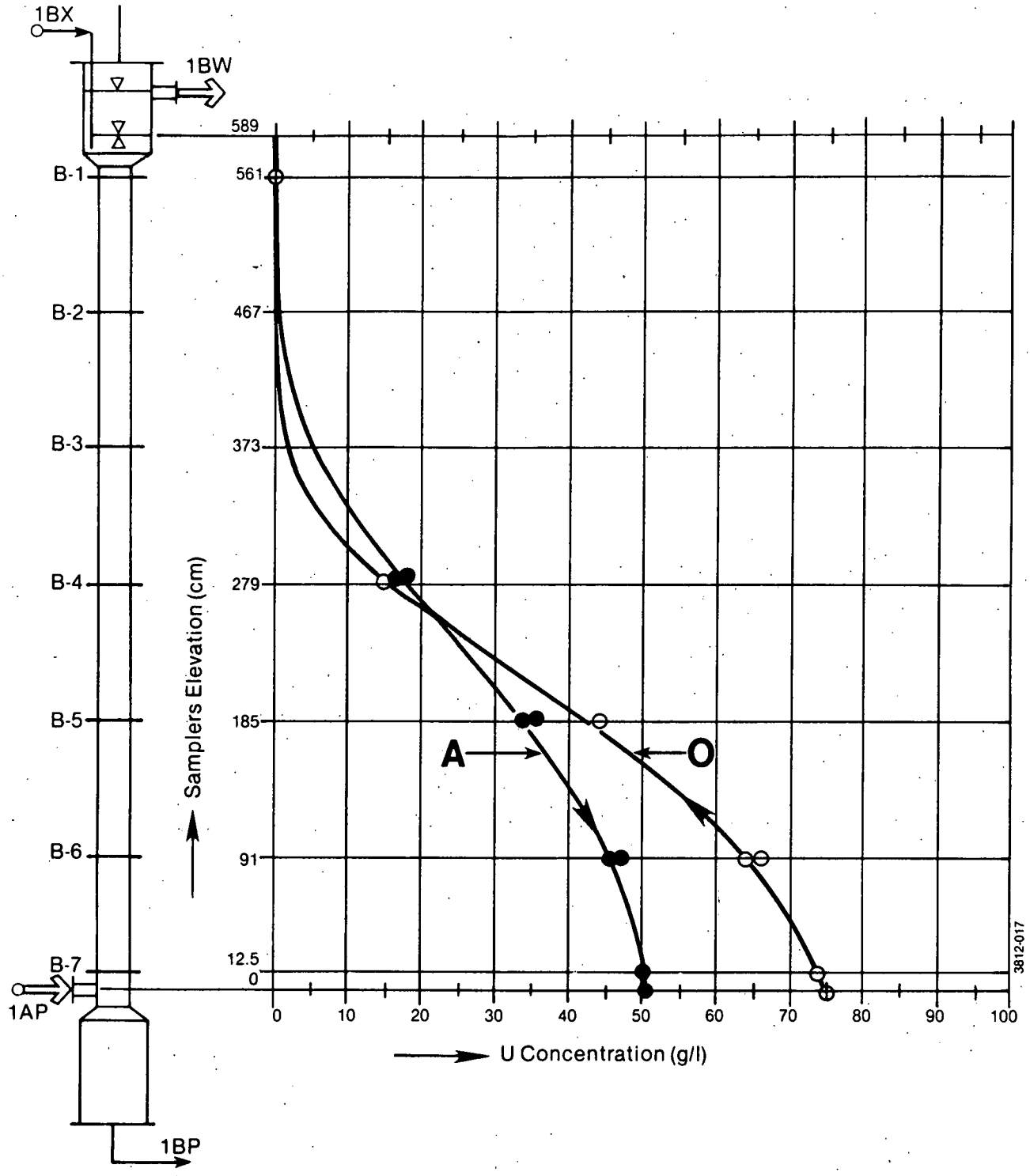
THE 1BX (≡1C) COLUMN EXPERIMENTAL CONCENTRATION PROFILES FOR RUN HSA-2

FIGURE 4-9



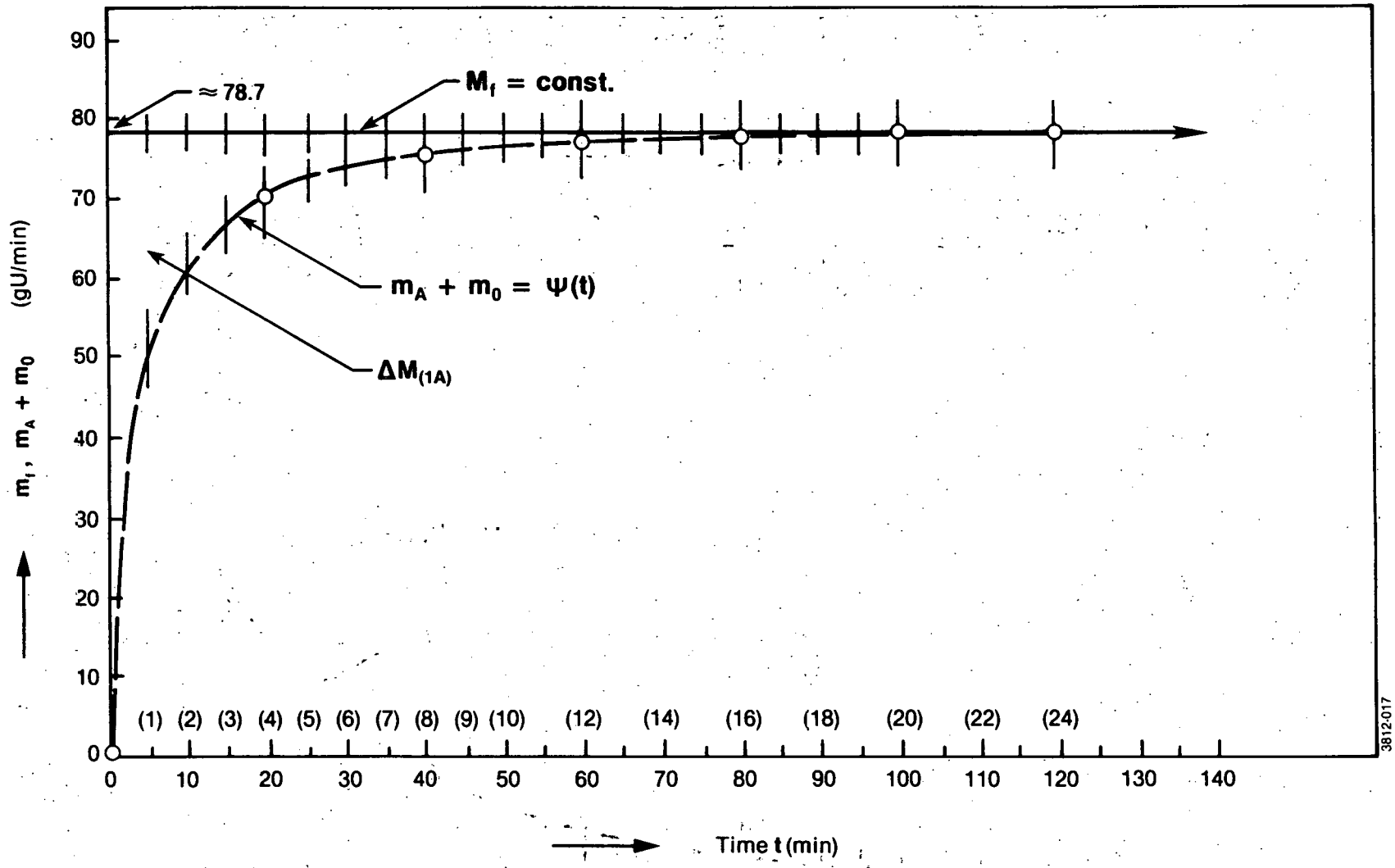
THE 1A (≡HS) COLUMN EXPERIMENTAL CONCENTRATION PROFILES FOR RUN HSA-3

FIGURE 4-10



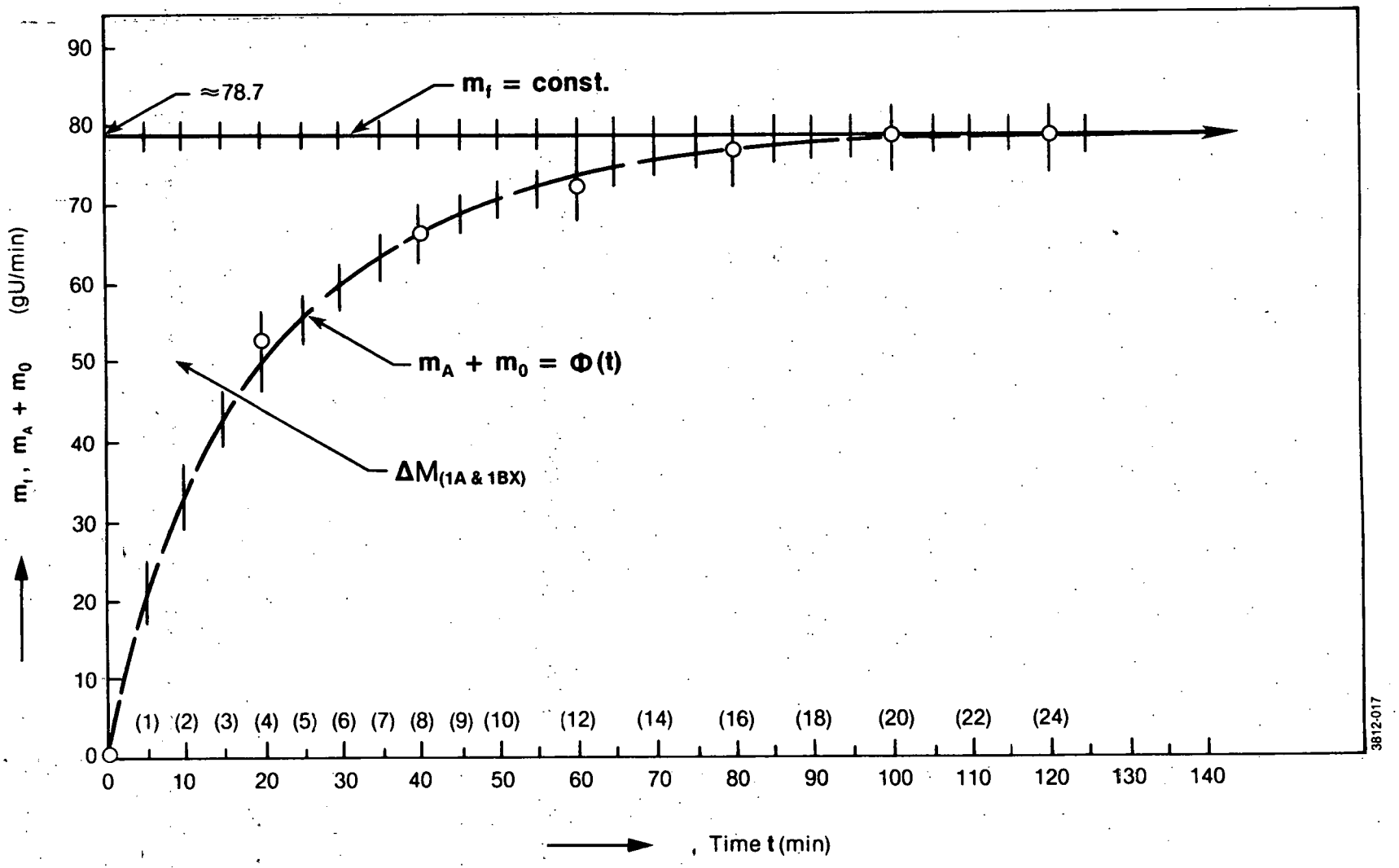
THE 1BX (≡1C) COLUMN EXPERIMENTAL CONCENTRATION PROFILES FOR RUN HSA-3

FIGURE 4-11



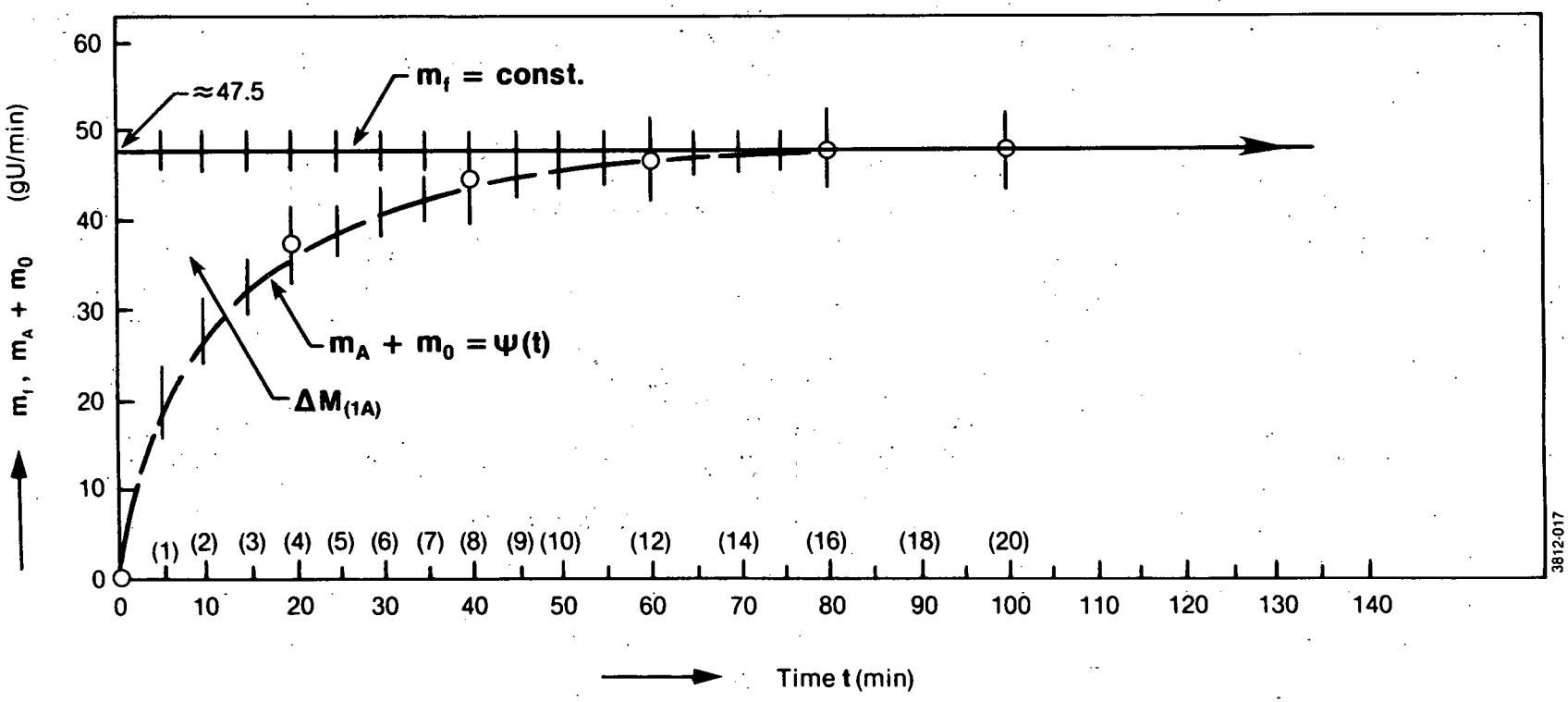
URANIUM MASS RATE VERSUS TIME IN THE 1A COLUMN (RUN HSA-1)

FIGURE 4-12



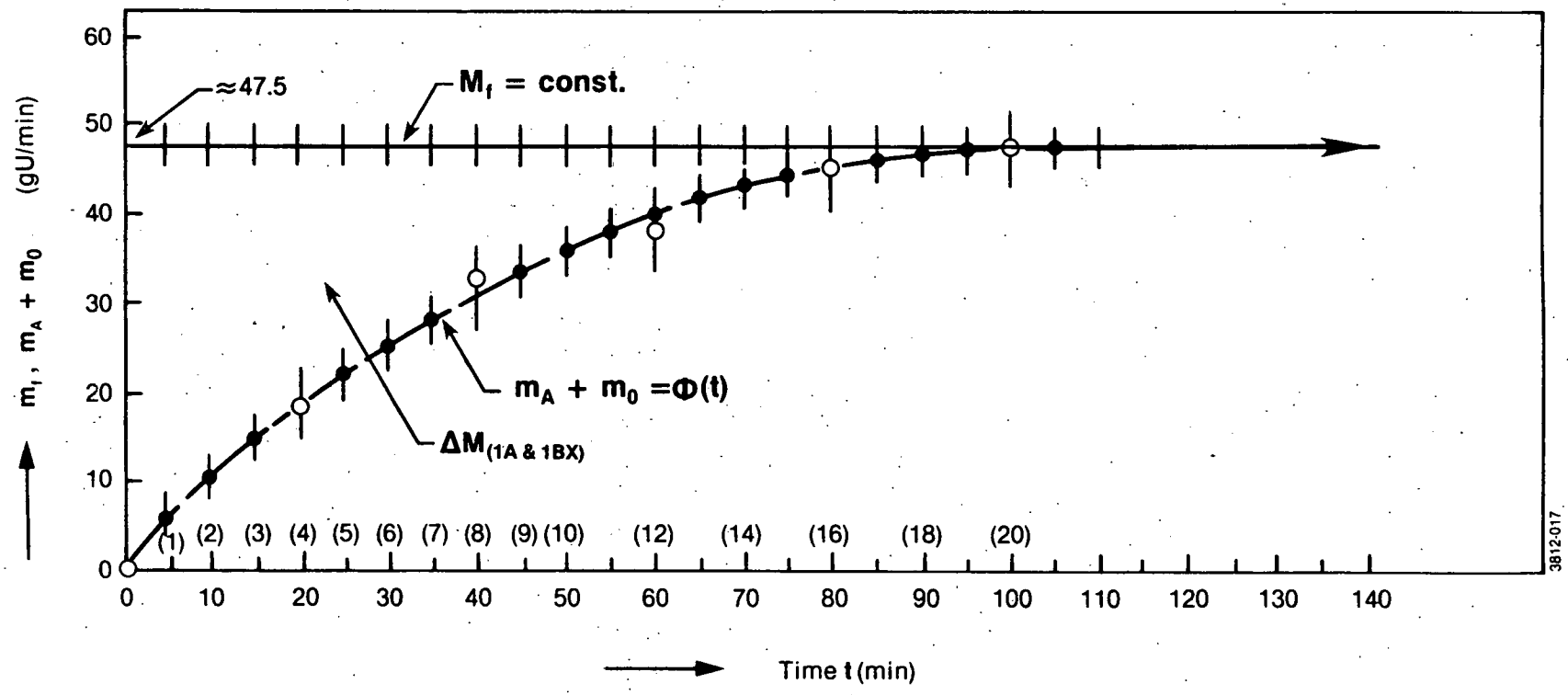
URANIUM MASS RATE VERSUS TIME IN BOTH THE 1A AND 1BX COLUMNS (RUN HSA-1)

FIGURE 4-13



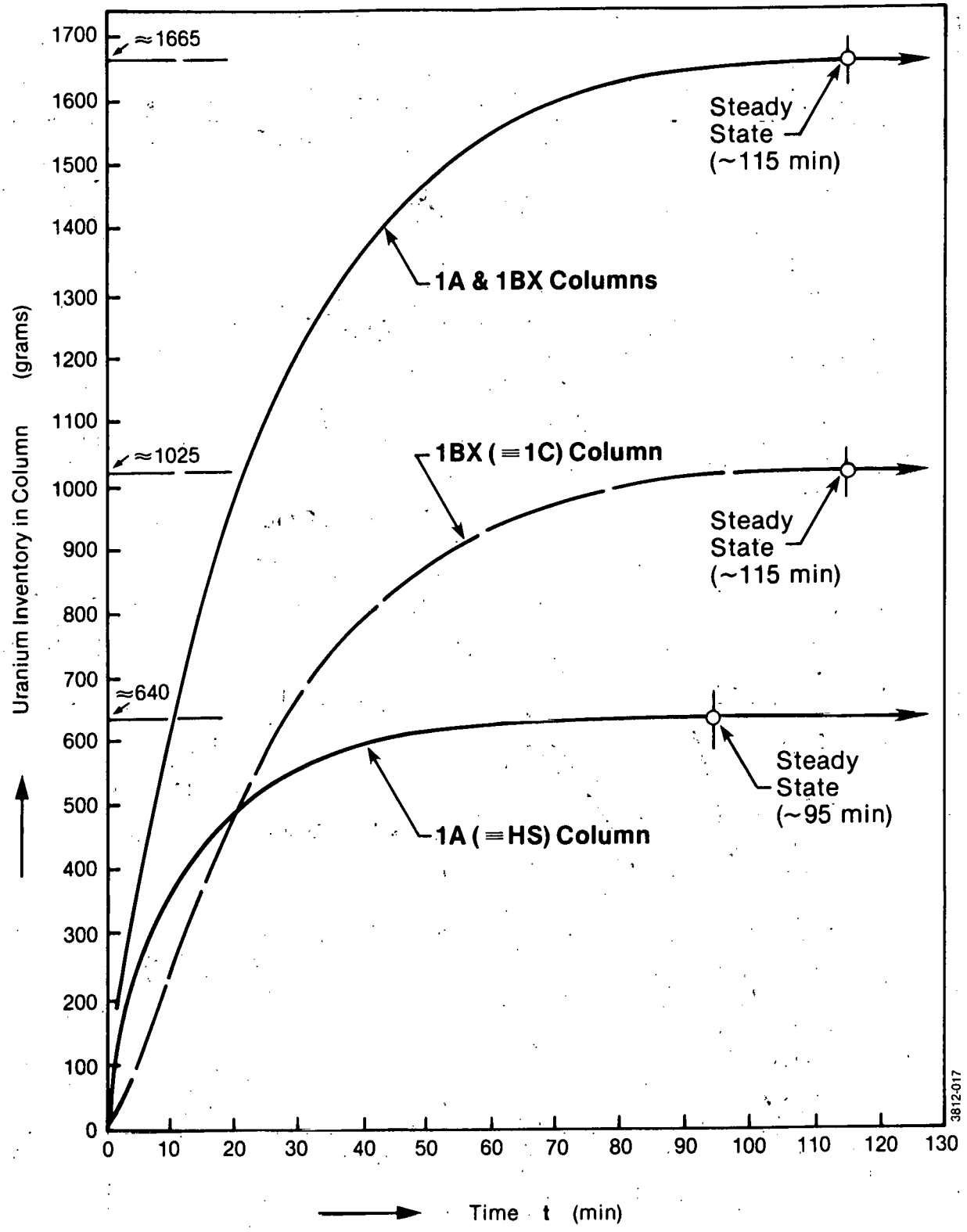
URANIUM MASS RATE VERSUS TIME IN THE 1A COLUMN (RUN HSA-3)

FIGURE 4-14



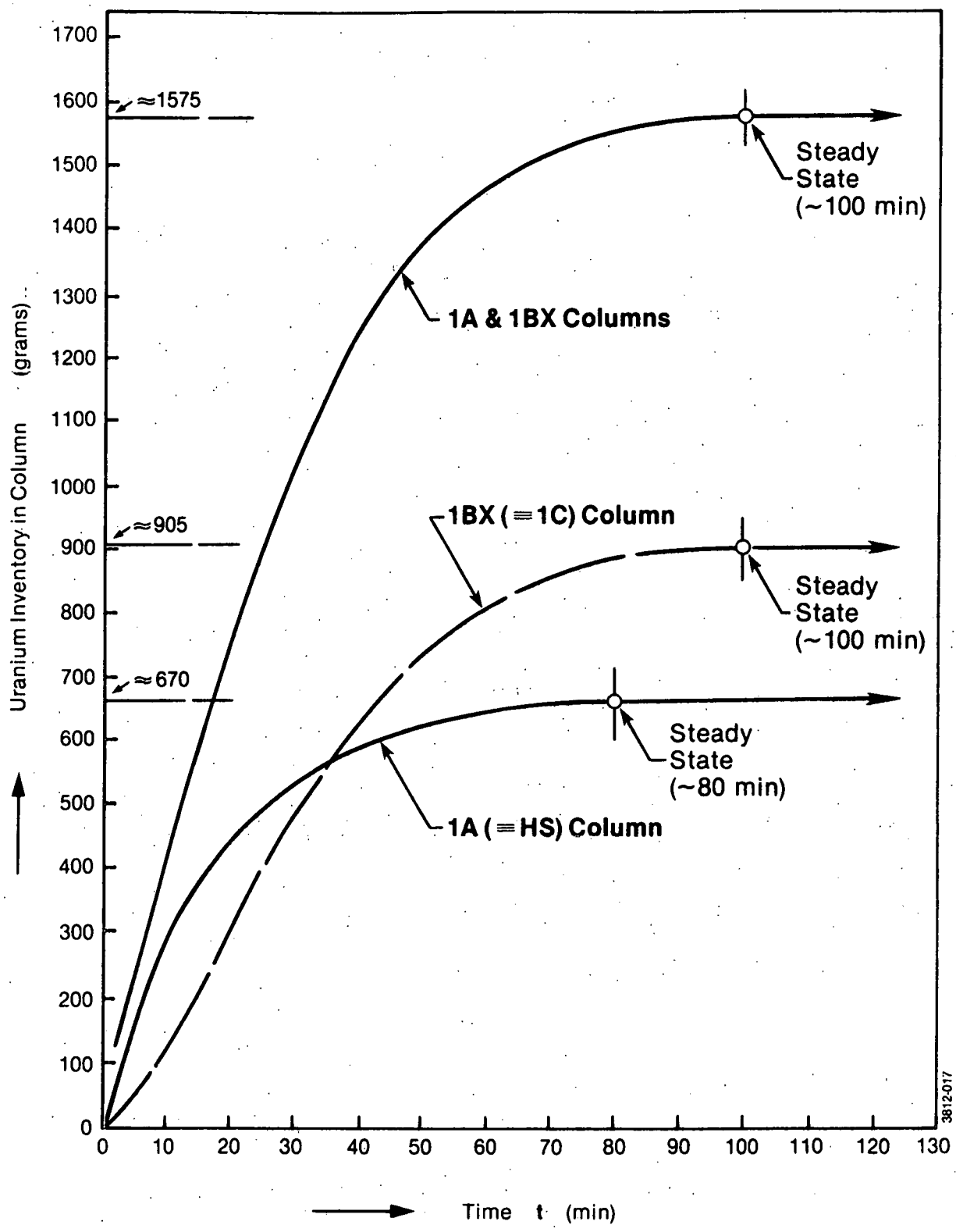
URANIUM MASS RATE VERSUS TIME IN BOTH THE 1A AND 1BX COLUMNS (RUN HSA-3)

FIGURE 4-15



URANIUM INVENTORY IN COLUMN VERSUS TIME (RUN HSA-1)

FIGURE 4-16



URANIUM INVENTORY IN COLUMN VERSUS TIME (RUN HSA-3)

FIGURE 4-17

5.0 CONCLUSIONS AND RECOMMENDATIONS

The dispersed phase holdup in a pulsed column is affected by the pulse frequency, pulse amplitude, and phase flow conditions. Its average value can be calculated by use of equations 9 or 10. Equation 10, however, is more suitable, as it describes the effect of the dispersed phase flow rate on the holdup also at a flow ratio of one.

Tests performed on the pilot-scale dual process pulsed column, simulating the BNFP HS column process conditions when operated as an extraction-scrub column, have shown several advantages when the column is operated with the aqueous phase continuous, which are as follows:

(1) The total uranium inventory within the column is about 35 to 40% lower than when operating the column with the organic phase continuous.

(2) Extraction is very efficient. Uranium in the 1AW (HSW) aqueous effluent stream was undetectable.

(3) The solvent (30 v/o TBP) residence time in the column (of about 6 to 7 minutes) is considerably shorter than in columns operated with the organic phase continuous.

The solvent quality will be degraded if it remains in contact with the highly radioactive solution of the first process cycle for an excessive period of time, when reprocessing short cooled and highly irradiated nuclear fuel by use of the Purex process.[3, 4] The solvent exposure to high radiation on the interface in the bottom disengaging section of the column, operating with the organic phase continuous, may last for hours due to the stagnant organic layer between the bottom interface and organic inlet. Due to diffusion and/or low flow at the interface, the solvent degradation products (DBP and MBP) will be transferred continuously to the main organic stream in the column, resulting in higher losses of uranium, plutonium, and solvent, and in a decrease of the fission products decontamination factor. Operating the 1A (≡HS) column with the aqueous phase continuous, the phases contact time within the column working section will be about 2.5 to 3 times shorter than in the column operated with the organic phase continuous; and there will be no organic stagnant layer causing uncontrollable solvent degradation.

With regard to the above advantages of the aqueous continuous dual process column, it is recommended that further tests be performed. These tests would include the following:

(1) Pilot-scale test with natural uranium simulating both the 2A and 3A column flowsheet conditions.

(2) Mass-transfer tests with the aqueous feed solution containing solids (simulating FP fines), in order to determine solids settling and their removal from the unit, as well as the possible redesign of the column disengaging sections.

(3) Generally, scrubbing is considered more efficient when the organic phase is dispersed into the aqueous phase.[3] It is recommended to prove this through tests with Zr (simulating fission products).

Efficient extraction in the 1A (≡HS) column is assumed to be the result of high back mixing in the continuous aqueous phase, which pushes the solute concentration in the direction of the dispersed phase flow (away from the aqueous waste outlet). Analogously, the same phenomenon is expected in the stripping column, which may improve solute's stripping in the column, when operated with the organic phase continuous. To verify this, it is recommended to test uranium stripping with the organic phase continuous, simulating the 1C, 2E, 2B, and 3B column process conditions.

Data in Table 4-9 show that all three experimental methods, i.e., the "Volume-Concentration Measurement," "Concentration Profile-Holdup Data," and "Mass Rate Measurement" methods, are reliable and can be used for determination of the total solute's inventory in a pulsed column.

The "Volume-Concentration" measurement method can be used only after completion of the run. It is performed by draining the column, separating both liquid phases from each other, and determining the volume and solute concentration in separated phases. The experimental "Concentration Profile-Holdup" measurement method can be applied to the total solute's inventory determination only when the column operates under steady-state conditions. However, the new "Mass Rate Measurement" method allows for determination of the total solute's inventory, as well as its changes during operation by determining both the feed and effluent streams solute's mass rates in short (progressive) time intervals measured from time zero, when the 1AF (≡HSF) feed is turned on, up to the run completion. See Section 3.2.3 for details.

With regard to resultant data shown in Table 4-9 and discussed in Section 4.4, it is recommended to test and use the "Mass Rate Measurement" method for the uranium inventory determination in full-size columns during cold uranium mini-runs in the plant. The new technique requires correct measuring of flow rates and uranium concentration in feed and effluent streams, in short (5 to 15 minutes) time intervals, and computer programming of equations 4 and 6 shown in Section 3.2.3. This can be accomplished in the present arrangement of the BNFP Separation Facilities. The method was preliminary tested and its usability verified at AGNS, using computerized equation 4 and 2A column data from last year's mini-runs. Results are shown in Appendix A.

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(2) Cermak, A. F.; Neace, J. C.; and Spaunburgh, R. G., Coprocessing Tests with Uranium on the Electropulse Column, and Pilot-Scale Pulsed Columns Simulating the Second and Third Plutonium Process Cycles, Allied-General Nuclear Services, Report AGNS-35900-4.1-20 (1979).

(3) Cermak, A. F., Liquid-Liquid System Contactors for Nuclear Fuel Reprocessing Plant, Allied-General Nuclear Service, Report AGNS-35900-3.2-64 (1980).

(4) Reactor Handbook, Second Edition, Volume II, "Fuel Reprocessing" (1960).

(5) Argonne National Laboratory, Nitric Acid-Organic Material Reactions, USAEC Report, ANL-5128 (1953).

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(7) Hakkila, E. A., et al, Material Management in an Internationally Safeguarded Fuels Reprocessing Plant, Los Alamos Scientific Laboratory, Report LA-8042 (1980).

(8) Ibid, Estimation of In-Process Inventory in Solvent Extraction Contactors, Volume III, Appendix J.

(9) Cobb, D. D., and Ostenak, C. A., Dynamic Material Accounting for Solvent Extraction Systems, Los Alamos Scientific Laboratory, Report LA-UR-79-3221 (1979).

REPORT NO. AGNS-35900-2.5-124

PILOT-SCALE PULSED COLUMNS, AQUEOUS CONTINUOUS DUAL PROCESS AND HOLDUP STUDIES

APPENDIX A

THE 2A COLUMN URANIUM INVENTORY FROM MINI-RUN DATA

M. H. Ehinger

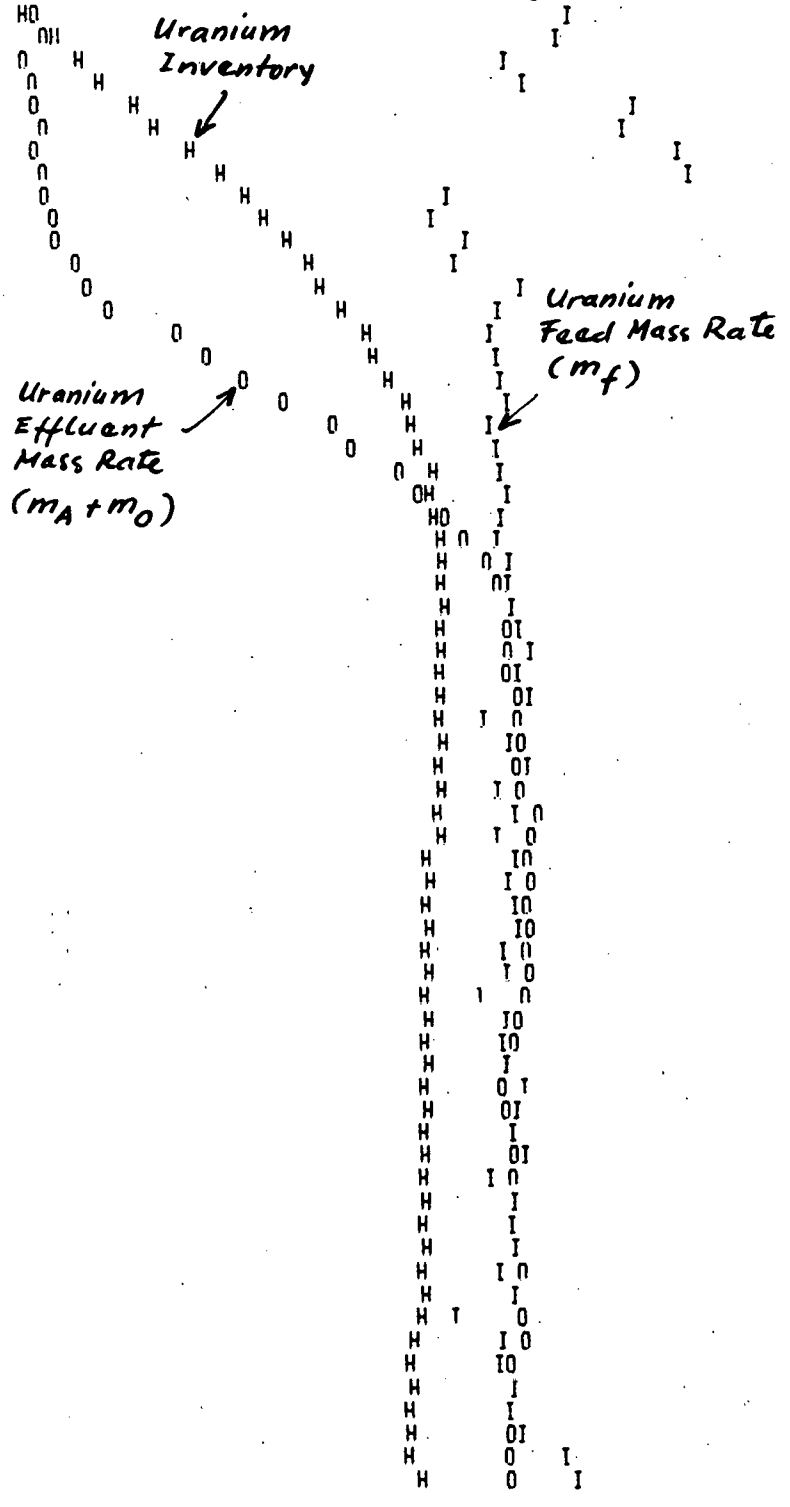
April 1981

Allied-General Nuclear Services
Post Office Box 847
Barnwell, South Carolina 29812

THIS IS A PLOT OF THE 2A COLUMN HOLDUP CALCULATED FROM MASS IN/MASS OUT MEASUREMENTS AS DESCRIBED BY A.F.CERMAK
THE DATA ARE FROM MINI RUN NO.4
FROM NO. 38 AT 07:15 AM ON 03-Sep-80
TO NO. 220 AT 05:00 AM ON 05-Sep-80

THE 2A MASS IN IS REPRESENTED BY AN (I) IN KGS/HR
THE MASS OUT BY AN (O) IN KGS/HR
AND THE CUMULATIVE HOLDUP BY AN (H) IN KGS

Time	CUM HLDP KGS	MASS IN KG/HR	MASS OUT KG/HR	INTEG I-O KGS	HDP SCALE 10 20 30 / I/O SCALE 1 3 5 7
03-Sep-80					
38 07:15AM	.6	4.9	.4	.6	
39 07:30AM	1.7	5.2	.4	1.2	
40 07:45AM	2.9	5.1	.5	1.2	
41 08:00AM	4	4.6	.3	1.1	
42 08:15AM	5.1	4.8	.4	1.1	
43 08:30AM	6.3	5.8	.4	1.2	
44 08:45AM	7.7	5.7	.5	1.3	
45 09:00AM	9	6.2	.4	1.4	
46 09:15AM	10.5	6.3	.5	1.5	
47 09:30AM	11.7	4.1	.5	1.2	
48 09:45AM	12.5	4	.6	.9	
49 10:00AM	13.4	4.3	.6	.9	
50 10:15AM	14.3	4.2	.8	.9	
51 10:30AM	15.2	4.8	.9	.9	
52 10:45AM	16.1	4.6	1.1	.9	
53 11:00AM	16.9	4.5	1.7	.8	
54 11:15AM	17.6	4.6	2	.7	
55 11:30AM	18.2	4.6	2.3	.6	
56 11:45AM	18.8	4.7	2.7	.5	
57 12:00PM	19.2	4.5	3.1	.4	
58 12:15PM	19.5	4.6	3.3	.3	
59 12:30PM	19.8	4.6	3.7	.3	
60 12:45PM	20	4.7	3.9	.2	
61 01:00PM	20.2	4.6	4.1	.2	
62 01:15PM	20.3	4.6	4.3	.1	
63 01:30PM	20.3	4.7	4.5	.1	
64 01:45PM	20.4	4.7	4.6	0	
65 02:00PM	20.4	4.7	4.7	0	
66 02:15PM	20.4	4.8	4.7	0	
67 02:30PM	20.4	4.9	4.7	0	
68 02:45PM	20.5	4.8	4.7	0	
69 03:00PM	20.5	4.9	4.8	0	
70 03:15PM	20.5	4.5	4.8	0	
71 03:30PM	20.4	4.7	4.8	-.1	
72 03:45PM	20.4	4.9	4.8	0	
73 04:00PM	20.4	4.6	4.8	0	
74 04:15PM	20.4	4.8	5	0	
75 04:30PM	20.3	4.6	4.9	-.1	
76 04:45PM	20.2	4.8	4.9	0	
77 05:00PM	20.2	4.7	4.9	0	
78 05:15PM	20.2	4.8	4.9	0	
79 05:30PM	20.1	4.8	4.9	0	
80 05:45PM	20.1	4.7	4.9	0	
81 06:00PM	20.1	4.7	4.9	-.1	
82 06:15PM	20	4.5	4.9	-.1	
83 06:30PM	19.9	4.7	4.8	-.1	
84 06:45PM	19.9	4.7	4.8	0	
85 07:00PM	19.9	4.7	4.7	0	
86 07:15PM	19.9	4.9	4.7	0	
87 07:30PM	19.9	4.8	4.7	0	
88 07:45PM	20	4.8	4.8	0	
89 08:00PM	20	4.9	4.8	0	
90 08:15PM	20	4.6	4.8	0	
91 08:30PM	19.9	4.8	4.8	0	
92 08:45PM	19.9	4.8	4.8	0	
93 09:00PM	19.9	4.8	4.8	0	
94 09:15PM	19.9	4.7	4.9	0	
95 09:30PM	19.9	4.8	4.8	0	
96 09:45PM	19.8	4.3	4.9	-.1	
97 10:00PM	19.7	4.7	4.9	-.1	
98 10:15PM	19.7	4.7	4.8	0	
99 10:30PM	19.7	4.8	4.8	0	
100 10:45PM	19.7	4.8	4.8	0	
101 11:00PM	19.7	4.9	4.8	0	
102 11:15PM	19.7	5.3	4.8	.1	
103 11:30PM	19.9	5.4	4.8	.1	



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