APPENDIX A

DISSOLVER COLD-FLOW MODELLING

INDEX OF APPENDIX

1. Report, Juan Lopez, "Technical Progress Memorandum #6, Coal Liquefaction/Coke-Flex: SRC Cold-Flow Model Studies," October 1978, Project 87-1-X705.

2. Interoffice Memorandum, R. F. Weimer to G. W. Roberts, "Mathematical Modelling of Solids Buildup in Tubular Dissolvers," 12 January 1979, Project 87-1-X921.

3. Interoffice Memorandum, D. H. S. Ying to R. F. Weimer, "Entrance Effects on Gas Holdup," 25 January 1980, Project 87-0-8884.

4. Interoffice Memorandum, E. N. Givens to Distribution, "Coal Liquefaction Group February Progress Report - (Foam Formation in Wilsonville Recycle Solvent by D. H. S. Ying)," 25 March 1980, Project 87-1-X921.

* Report, D. H. S. Ying, "Stirred Reactor Hydrodynamics," April-June 1980, Project 87-1-X921.

* Report, E. N. Givens, R. F. Weimer, and D. H. S. Ying, "Gas Holdup in Bubble Column," Project 87-1-X921.

RESEARCH REPORT COVER PAGE

(COMPANY CONFIDENTIAL)

RRRS STANDARD TECHNICAL REPORT NO. APCI-CRD-78-020

DATE ISSUED October 1978

SECURITY (CHECK ONE) ____ RC X C

ORIGINATING R & D DEPARTMENT Corporate Research & Development Department

LOCATION (FACILITY, CITY, STATE) Allentown Laboratories, Allentown, PA

GROUP OR DIVISION Corporate Research Group

AIR PRODUCTS AND CHEMICALS, INC.

Technical Progress Memorandum #6, Coal Liquefaction/Coke-Flex: SRC Cold-Flow Model Studies
(TITLE)

Progress
(INDICATE PROGRESS OR FINAL REPORT)

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REPORT WRITTEN BY: Juan Lopez

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PREVIOUS RELATED REPORTS: None

DEPARTMENT OVERHEAD NUMBER: 107-2000

PROJECT NUMBER: 87-1-X705

PERIOD COVERED: June to September 1978

NOTEBOOK NUMBER(S): None

PROPRIETARY INFORMATION FOR AUTHORIZED COMPANY USE ONLY

FORM 5686 (REV. 7/78)

ABSTRACT

A simulation of the APCI Solvent Refined Coal pilot plant tubular reactor-dissolver is described. A water-nitrogen-silica system was used to simulate the fluid behavior of the reactor at room temperature and pressure. An attempt was made to apply theoretical models which: a) describe the effect of different flow rates on the liquid mixing and b) quantify the solid particles distribution in the vessel. Most of these effects are illustrated, compared, and explained from transport phenomena principles. A summary of a literature survey of three-phase systems is reported.

INDEXING PHRASES

Coal, coal conversion, solvent refined coal, dissolvers, reactors, tubular reactors, mixing, turbulence, fluid flow, reactor dynamics, cold-flow models, Plexiglas models, reactor simulator, three-phase flow.

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INTRODUCTION

The Solvent Refined Coal (SRC) process, currently under development, involves the use of a liquefaction reactor where coal is dissolved in a hydrogen donor solvent in the presence of hydrogen gas. This reactor, which operates at high temperatures and pressures, consists of a vertical tube containing an upward flowing three-phase mixture of coal, solvent, and hydrogen gas. The complexities associated with a three phase flow system present a series of problems to the efficient design of the SRC dissolver column. The occurrence of unequal residence times of the phases involved, the existence of different mixing patterns of those phases, and the tendency of solids to accumulate at the bottom of the column greatly complicate the reactor design equations. These phenomena, in addition to the still unpredictable kinetic properties of coal and the absence of reliable physical properties data at the operating conditions, make the optimum SRC dissolver design a very difficult task at the present stage of knowledge.

This study was aimed at enhancing the understanding of this complex system by presenting the results of a literature survey on the flow of three phase tubular reactors and by performing a series of studies on a simple system designed to simulate the flow through the tubular reactor of the SRC pilot plant at the Trexlertown facilities of APCI. It was intended to provide a basic knowledge of the hydrodynamics of such systems to serve as a foundation for further studies on larger scale reactors.

SUMMARY AND CONCLUSIONS

A literature evaluation led to the conclusion that bubble column slurry operations have been characterized at zero liquid flow rates. Almost no data exist for three-phase flow through tubular columns. Preliminary observations on the 2 inch ID column indicated the bubble diameter easily bridged the column in liquid and slurry systems. Particles of 140 mesh could be easily handled in the slurry system. However, 80 mesh particles rapidly accumulated in the column causing severe burping and would presumably lead to ultimate plugging.

Extensive attention was given to simulating a coal dissolver operation. The most obvious weaknesses in the water-silica-nitrogen system were surface tension of the liquid and density of the nitrogen. Insufficient time was available to examine closely a methanol-silica-Freon system.

In a 500-minute experiment the holdup of solids in the reactor was observed. A gradient existed along the axis with the higher concentration and larger particle sizes found at the bottom of the reactor. During the experiment the increase in solids tended to level out.

Dispersion of salt solution through the reactor was studied at a variety of liquid, slurry and gas velocities. In the absence of gas the liquid tended to have the maximum plugflow characteristic observed. Addition of extremely small amounts of gas tended to promote extensive backmixing. Further addition of gas has an effect, but to a lesser extent.

Dispersion numbers were calculated for the data obtained in the experiments. The shape of the C-curves for the specified dispersion numbers showed some variance from the experimental curves. This variance may be explained by the presence of semi-stagnant sections in the column.

Extensive data and discussion are presented. Unusual observations made during the course of this study are also presented. The main conclusions are presented here:

- The dispersion model can be applied to this system if stagnant or semi-stagnant regions do not exist.
- Liquid mixing to suspend solids is mainly caused by gas bubbles.
- At constant gas flow mixing decreases as liquid flow increases.
- Distribution of solids in the reactor is a function of gas velocity through its dependence on the axial dispersion coefficient.
- Particle distribution profile within the reactor may be essential to the heat transfer design since the solid particles catalyze the hydrogenation reaction.

BACKGROUND

Although considerable effort has been put into SRC plant design studies, not until recently did some people start to realize the problem of solids accumulating at the bottom of the SRC dissolver reactor. This problem which is a challenging one at the pilot plant might be a very significant obstacle to the success of continuous operation in a commercial plant.

It is of common belief that most of the coal dissolves throughout the preheater before entering the reactor. However, approximately 10 percent of this coal can be identified as mineral matter that does not dissolve and presumably has a catalytic effect on the desired solvent regenerating reactions. Under the influence of gravity, these undissolved particles tend to accumulate in the lower section of the dissolver causing tremendous complications that eventually prevent continuous operation.

Since the design of liquefaction reactors must ensure that gas and liquid velocities through the reactor are sufficient to maintain the unconverted coal and ash (or mineral matter) particles in suspension, several studies have been recently carried out to attack this problem. One of the most conclusive ones was conducted by Exxon Corporation (12). They suggest that, since some particles are never going to reach the top of the column by using the flow rates required for operation, a withdrawal system must be used to extract the particles larger than 8 mesh (Figures 1 - 3).

Other publications put more emphasis on the dimensional analysis of gas-slurry systems. Although they describe most of the factors affecting the flow patterns, their reports are based on information obtained from systems using significantly higher flow rates than the simulation requirements. However, it is the author's opinion that their conclusions are relevant to this study. The main conclusions of these reports are:

1) Bubble column slurry operations are usually characterized by zero net liquid flow, and the particles are held suspended by momentum transferred from the gas phase to the solid phase via the liquid medium. (10)

2) The critical gas velocity for complete suspension of particles is mainly affected by liquid flow near the gas distribution, and therefore to obtain a small critical gas velocity the shape of the bottom of the column and the position of the gas distributor may become important. (4)

3) The gas holdup and the concentration distribution of solid particles do not depend on the gas distributor nor on the shape of the bottom of the column when solid particles are suspended completely. (4)

BACKGROUND (cont.)

4) The range of gas velocity over which a bubble column slurry reactor may be operated decreases with increasing solids concentration. The lower limiting gas velocity increases because of an increasing tendency towards sedimentation, and the upper limiting gas velocity decreases because of an increasing tendency towards bubble coalescence and the corresponding increase in bubble size. (7)

5) The larger solid particles show a somewhat smaller gas holdup. This is considered to be caused by the larger rising velocity of coalesced bubbles in the presence of solid particles. In the region of high gas velocity, where large coalesced bubbles rise frequently, the effect of the concentration of solid particles on gas holdup becomes gradually smaller as gas velocity increases. The gas holdup was found to decrease by increasing the amount of solids in the reactor. (6)

6) The maximum quantity of solids that can be held in suspension in a liquid medium agitated by bubbles in a defined system is termed critical solids holdup. There are two regions of critical solids holdup with gas velocity. In the first region of lower gas velocity the holdup is a function of gas distribution and data indicate that an arrangement with about six orifices per square inch provides a reasonably uniform closed gas distribution. In the second region of higher gas velocity, the critical solids holdup is independent of gas distribution. (11)

7) The critical solids holdup decreases with increasing particle size of the solids and the surface tension of the liquid. (11)

8) The critical solids holdup is a function of the solid surface properties and for solids of different degrees of wettability in a liquid, the solids holdup is much greater with a less-wettable solid under identical operating conditions. (11)

9) The dispersion coefficient of suspended solid particles is the same as that of the liquid within the column without solid particles. (4)

10) The longitudinal dispersion coefficients of the liquid and of the slurry increase with an increase in the superficial gas velocity and are proportional to the 1 to 1.5th power of the column diameter. (6)

The last two observations are related with mass and heat transfer in slurry bubble columns:

11) The overall volumetric absorption coefficient kgA_6 was observed to increase with gas velocity toward an upper limiting value of about 1.3×10^{-6} mole/min for the absorption of oxygen from air in aqueous sodium sulfite solutions containing cupric ions as catalyst, and sand particles (particle size 0.015 to 0.030 cm). At low gas velocities, the absorption was observed to decrease with increasing particle size, increasing amount of

solids in the column, and increasing density difference between solid and liquid, whereas at higher velocities it was nearly independent of these variables. The interfacial area A_6 is reported to be nearly constant at a value of 10 sq cm/cu cm for gas velocities corresponding to a constant volumetric absorption coefficient. The gas velocity above which k_gA_6 is nearly constant is related by analytical and graphical correlations to the amount of solids in the column and to the critical gas velocity corresponding to complete suspension of the solids. (5)

12) Experiments with bubble columns containing suspended solid particles of varying diameters have demonstrated an increase of heat transfer coefficient with increasing particle size. (10)

EXPERIMENTAL APPARATUS AND PROCEDURE

A schematic diagram of the experimental apparatus is shown in Figure 4. A Plexiglas column of 152.4 cm in length, having an inner diameter of 5.08 cm was used. The slurry and gas were introduced simultaneously into the column through a 0.48 cm diameter orifice located on the column wall 3.8 cm from the bottom. A gas distributor was not in use. It was observed that when the same line was used to feed both slurry and gas it was possible to diminish the bubble size in the column by increasing the slurry flow rate. This phenomenum was explained by the fact that after a critical fluid flow rate through the inlet tube the gas flow is cut by slugs of liquid.

The measurements and observations were obtained from the continuous operation for slurry flow. The gases used were nitrogen and Freon 12. A slurry of 4 weight percent of silica particles in the tap water was fed into the column. Three size ranges of silica particles with a density of 2.41 g/cu.cm were used - about 240 mesh, about 140 mesh, and from 80-120 mesh.

The slurry was allowed to flow upwards concurrently with the gas and to exit the column through a 0.48 cm diameter orifice located on the column wall 3.8 cm from the top. A conductivity prove was positioned at the slurry exit. Another orifice was provided at the top of the column so that the gas would not interfere with the conductivity readings. The liquid mixing was measured by the method of delta response using a NaCl solution as the tracer. Seven milliliters of 2.4 molar NaCl solution were introduced into the feed at an injection rate of 2.3 cu.cm/sec. The effect of the injection rate was shown to be negligible in the range of 0.6 to 4.0 cu.cm/sec.

After a steady concentration distribution of solid particles was established, samples of slurry were withdrawn through sampling taps into measuring cylinders. The weight of each sample was measured and then solid particles were separated from the liquid, dried and weighed. It was ascertained by Imafuku (4) that the solid content of the sampling liquid withdrawn is the same as that of the liquid within the column. The samplings were collected at intervals of 10 minutes from top to bottom since it is probably a good way to rapidly restore equilibrium.

The gas holdup was calculated from the height of the column and the liquid level containing solid particles by keeping the slurry stationary and stopping the gas flow at increasing flow rates. The deviation of the results obtained by this simple method from the actual gas holdup should be negligible at the low liquid flow rates used.

The experimental system was then modified to feed both slurry and gas from the bottom of the column as well as to separate the entrance of the phases. Comparative results are reported in following sections.

SIMULATION

Since the experimental system was originally designed to simulate the operating conditions of the SRC pilot plant at Trexlertown Laboratories, the same operating parameters were chosen.

Dimensional analysis was used to obtain similar flow patterns similar to the actual SRC reactor. It was decided to maintain both the Reynolds and Froude numbers constant. This criterium was based on previous experience with flow systems in addition to the difficulty related to the calculation of other dimensionless groups such as the ones containing liquid surface tension or solid-liquid wettability parameters.

The parameter values for the SRC pilot plant were estimated as shown in Appendix 1 and summarized in Table 1. There is extensive data available on the components used in the cold flow model. These properties are also summarized in Table 1. The dimensionless groups were then calculated in the following manner:

From the pilot plant:

$$Re_M = \frac{p_M\, v_M\, d}{\mu_L} = 34.60$$

$$Fr_M = \frac{p_L\, v_M^2}{p_e\, g\, d_p} = 3.31 \times 10^{-4}$$

$$Re_G = \frac{p_M\, v_G\, d}{\mu_L} = 316.95$$

$$Fr_G = \frac{p_L\, v_G^2}{p_e\, g\, d_p} = 2.78 \times 10^{-2}$$

These groups were used to calculate the experimental system flow rates in the following way:

$$\left.\begin{aligned} v_M &= \frac{Re_M\, \mu_L}{p_M\, d} = 4.01 \text{ cm/min} \\ v_M &= \frac{Fr_M\, p_e\, g\, d_p}{p_L} = 4.06 \text{ cm/min} \end{aligned}\right\} \quad (v_M)_{AVG} = 4.03 \text{ cm/min}$$

$$v_G = \frac{Re_G \mu_L}{p_M \; d} = 36.70 \text{ cm/min} \qquad (v_G)_{AVG} = 36.94 \text{ cm/min}$$

$$v_G = \sqrt{\frac{Fr_G \; p_e \; g \; d_p}{p_L}} = 37.19 \text{ cm/min}$$

These flow velocities represent the following volumetric flow rates in the system under study:

v_M = 4.03 cm/min $\qquad$ Q_M = 82 cu.cm/min

v_G = 36.94 cm/min $\qquad$ Q_G = 749 cu.cm/min

Although these rates proved to satisfy both Froude's and Reynold's numbers within 1.4 percent, such low slurry flow rates were unattainable in the experimental system for reasonable periods of time. Therefore, the bulk of the experimental data were taken at slurry flow rates higher than 100 cu.cm/min.

THEORY

REACTOR MODELLING

To predict the performance of equipment we must know: (a) the rate at which fluid is modified as a function of the pertinent variables and (b) the way fluid passes through the equipment.

There is extensive literature (9) dealing with studies on the possible different flow patterns and the procedure to model them. Of particular interest are the dispersion models which are useful mainly to represent flow in empty tubes. This type of flow is much closer to the ideal case of plug flow than to the opposite extreme of backmix flow. In empty tubes, the mixing is caused by molecular diffusion, superposed on the velocity profile effect.

Reactor flow studies are typically carried out by introducing a pulse of tracer with the feed and monitoring its concentration as a function of time at the exit of the vessel. When flow is turbulent the resulting concentration fluctuations are rapid, numerous, and also small with respect to vessel size. They might be considered to be random, which would lead to a diffusion-type equation to model the mixing. This is best known as the dispersion model.

It was attempted to study the SRC cold flow simulator by using the mentioned dispersion model. In order to be able to compare different sets of experiments and to perform further calculations, the C-curve, as described in Figure 5 (8) was used. The C-curve is a graphical representation of the residence time distribution of any given set of fluid molecules in the reactor.

Frequently it is possible to characterize the shape of the residence time distribution curve by obtaining its mean (μ_1) and its variance (σ^2). The calculation of these parameters is presented in Figure 6 (8). The variance of a curve can be associated to a dispersion number (D/uL) which is characteristic of a curve such as in Figure 7 (8). This association was possible by numerically solving the equations that describe a closed system such as the one used in this study. These boundary equations and their solution for the mean and variance are shown in Figure 8 (9). In this figure P stands for the inverse of the dispersion number.

Due to the difficulty of numerically solving the equations of Figure 8 to represent a residence time distribution of a given dispersion number, it was decided to use the curves of Figure 7 as the criteria to prove the validity of the dispersion model for the system under study.

When one-parameter models are unable to account satisfactorily for deviations of the dispersion model then more complicated models should be attempted. These usually consider the real reactor to consist of different regions (plug, dispersed plug, mixed, deadwater) interconnected in various ways (bypass, recycle, or cross-flow).

REACTOR MODELLING (cont.)

Of particular interest is a very flexible model which considers that there is a slow interchange or cross flow between the fluid in "deadwater regions" and the active fluid passing through the vessel. This type of combined model which was presented by Adler and Hovorka (2) is shown in Figure 9 and is very advantageous in the study of flow patterns found in short tubular vessels and tubular reactors at low flow rates.

THEORY

SOLIDS CONCENTRATION PROFILE

A model to predict the solids profile in a reactor as a function of physical properties and operating conditions was developed by Cova (3). This model has been applied with success by Cova (3) and Kato (6) to both laboratory and plant scale reactors operating with superficial velocities in the range of 18.3 to 137.2 cm/min and superficial gas velocities in the range of 109.7 to 868.7 cm/min (STP).

The motion of particles suspended in a liquid medium may be analyzed through consideration of the various components of flow. Thus, particles in a still liquid will exhibit a downward flow under the influence of gravity. If the liquid medium is flowing in a vertical direction, its velocity, v_L, will be superimposed on the particle fall velocity, v_F. In the case of upward liquid flow, the resultant particle velocity then is $v_L - v_F$. Thus, in the concurrent tubular reactor (gas and liquid both flowing upward) the concentration of the particles in the reactor will be greater than, or at least equal to, the feed concentration.

Introducing gas into the system there is a third motion to be considered, that of mixing. It has been observed by the injection of a dye into the feed that the flow of gas induces considerable mixing of the liquid. It is suspected that this agitated liquid will bring about a similar flow of the solid particles. This flow has been successfully represented by a diffusion mechanism.

The following derivation applies when it is assumed that particle mixing can be described by diffusion superimposed on a bulk flow.

For a differential element of reactor volume the continuity equation for a given species of particles may be written as:

$$\frac{\partial c}{\partial t} + \nabla \cdot (\vec{n}_p) = 0 \qquad (1)$$

With the assumption that there are no radial gradients:

$$\frac{\partial c}{\partial t} + \frac{\partial}{\partial x}(n_p) = 0 \qquad (2)$$

The mass flux, n_p, may be represented by Fick's law modified to take into account the effect of gravitational forces on the particle:

SOLIDS CONCENTRATION PROFILE (cont.)

$$n_p = \frac{C\,(n_p + n_l)}{p_M} - Cv_F - D\,\frac{\partial C}{\partial x} \qquad (3)$$

At the low solid concentrations studied it can be assumed that n_p is small relative to n_l, so that

$$\frac{n_p + n_l}{p_M} = v_M \qquad (4)$$

Substituting into Equation 2:

$$\frac{\partial C}{\partial t} + (v_M - v_F)\,\frac{\partial C}{\partial x} - D\,\frac{\partial^2 C}{\partial x^2} = 0 \qquad (5)$$

At steady state:

$$\frac{\partial C}{\partial t} = -(v_M - v_F)\,\frac{\partial C}{\partial x} + D\,\frac{\partial^2 C}{\partial x^2} = 0 \qquad (6)$$

The general solution of Equation 6 is:

$$C = A + B \exp\,(v_M - v_F)\,\frac{x}{D} \qquad (7)$$

The boundary conditions that express the fact that the rate of feed into the reactor must equal the flow from the inlet plane by bulk flow and diffusion are sufficient:

At x = 0 (bottom and feed end)

$$v_L\,C_F = (v_M - v_F)\,C\Big|_0 - D\,\frac{\partial C}{\partial x}\Big|_0 \qquad (8)$$

At x = L (top and exit end)

$$v_L\,C_F = (v_M - v_F)\,C\Big|_L - D\,\frac{\partial C}{\partial x}\Big|_L \qquad (9)$$

<u>SOLIDS CONCENTRATION PROFILE</u> (cont.)

The steady state solution then is:

$$C = \left\{ \frac{v_M C_F}{v_M - v_F} + \left(C_T - \frac{v_M C_F}{v_M - v_F} \right) \exp\left[-(v_M - v_F)\left(\frac{L-X}{D}\right) \right] \right\} \qquad (10)$$

Cova in his report continued the derivation toward a transient state solution where the solids concentration in the reactor may increase indefinitely. This solution, although worth mentioning, is considerably more complex than Equation 10 and was not of practical interest in this study.

DISCUSSION OF RESULTS

TRACER STUDIES

Tracer studies were conducted using a slurry containing 4 percent by weight of silica in water. Pure nitrogen was bubbled through the slurry at various flow rates. A computer program was developed to calculate Peclet numbers using the dispersion model. The program was also used to convert the data into normalized C-curves and to plot such residence time distribution curves.

As an indication of the validity of the observed residence time distribution, an attempt was made to obtain reproducible results. A typical sample of the degree of reproducibility is shown in Figure 10.

The dispersion numbers were calculated under a variety of conditions and are reported with their residence time distribution curves. Before discussing any trends, it should be examined how the experimental data compares with the dispersion model. For this purpose the curves presented in Figure 7 were plotted versus the experimental C-curves at constant dispersion numbers. These comparisons are shown in Figures 11 and 12.

The experimental C-curves satisfy all the characteristics defined by Adler (1) to indicate the existence of a stagnant or semi-stagnant section. This section is present when using either liquid or slurry and for both side and bottom feeds. It was observed by injecting a dye that this area is not completely stagnant and that it probably occurs because of the low flow rate operating regime. This semi-stagnant section seemed to be slowly interchanging fluid with the active area suggesting the applicability of the Adler-Hovorka model mentioned in a previous chapter. Unfortunately, the solution of this four parameter model is tedious and was considered irrelevant at the present stage of study.

Nevertheless, the author wants to emphasize the significance of the residence time distribution data which proved to be reproducible. These curves can easily be compared by using Figures 17 to 21 where the gas flow rates were kept constant and Figures 22 to 24 where the fluid flow rates were kept constant.

Before attempting to analyze the effect of the gas and liquid flow, it is important to study the effect of the solids in the mixing. Figures 13 to 16 show the influence of the solid particles in the residence time distribution curves. According to these curves the studied concentration of solids in the reactor does not indicate a definite trend of change in the mixing. These curves only suggest that at higher gas rates the mixing decreases its functionality on the solids concentration.

TRACER STUDIES (cont.)

The following set of C-curves indicates the effect of the liquid rates on the mixing. Figures 17 to 20 show something unexpected although not conclusive. At gas flow rates under 5 cm/min it was observed that by increasing the liquid volumetric rate the mixing consistently increased, while at higher gas rates the higher the liquid rate, the lesser was the mixing. This seems to be that at the low gas flow domain the liquid turbulent eddies increases the overall mixing while at the high gas flow domain the linear turbulent velocity profile reduces the backmixing caused by the gas.

At constant slurry flow rates, the observations reported by Kato (6) at higher flow rates were confirmed at the studied conditions; the degree of mixing increases at a lesser rate as the gas flow is increased. This is shown in Figures 22 to 24.

DISCUSSION OF RESULTS

SOLIDS CONCENTRATION PROFILES

Experiments designed to study the concentration profiles were conducted to enhance the understanding of the solid particles in the reactor.

Figure 25 shows how steady state was approached but never reached by operating for 520 minutes. The particle size distribution was obtained at each of the sampling heights. These distributions are presented in Figure 26 and show significant segregation of the different particle sizes along the column. This indicates the accumulation of the heavy particles that were unable to reach the top. On the other hand, normalized concentration ratios proved to remain almost constant as a function of time. This fact suggests the possibility of misleading high concentrations obtained at the feed under the present method of sampling.

In Figure 27 it was observed again the insignificant effect of the gas on the mixing when operating above the range where the gas is essential for the mixing. This can also be explained from Figure 23 and Equation 10. Figure 23 shows an almost constant D after a gas flow rate above 405 cu. cm/min. Therefore, Equation 10 loses its gas flow rate functionality above the mentioned flow rate.

To test the validity of Equation 10, an experimental solid concentration profile was compared with the theoretical results. (See Figure 28). It was assumed that: a) the silica concentration at the exit of the vessel equals the concentration at the feed, and that b) the fall velocity (v_F) could be calculated from the mean particle size obtained from Figure 26 by using Stokes law,

$$v_F = \frac{d_p^2 \; g \; \rho_e}{18 \mu_L} \qquad (11)$$

The solids concentration profile in a vessel operating without gas was not investigated. The gas presence lead to model this profile by a diffusion mechanism. Therefore, in a liquid-solid system the solids concentration should remain constant throughout the length of the reactor.

DISCUSSION OF RESULTS

MISCELLANEOUS OBSERVATIONS

An attempt was made to examine the effect of larger particle sizes in the reactor's behavior. The introduction of a slurry containing particle sizes from 80-120 mesh was very problematic. After forcing these solids into the reactor, ebullated bed type of behavior was observed. Occasionally a solid slug formed and was carried up the column by a gas slug. These solid slugs eventually collapsed before reaching the top of the column. An overall tendency for bubbles to coalesce more readily was observed under this condition.

Freon 12 was used as an alternative gas which more closely simulates the gas-liquid density rates in the reactor. Since not much difference was observed in the bubble behavior nor in the residence time distribution curves, it was decided to keep using nitrogen, which is more conveniently handled.

Alternative manners of getting the feed into the reactor were studied. At low gas flow rates the mixing turned out to be better when the feed was introduced through the bottom of the vessel. At high gas flow rates the position of the inlet did not make a difference.

Figure 29 presents the gas-holdup data obtained from displacement measurements on the experimental column. This holdup data was used by the computer to calculate the dispersion numbers.

CONCLUSIONS

The understanding of the phenomena occurring in the SRC reactor is essential to the efficient design of the coal liquefaction process. The following conclusions were drawn from this study:

1) It is reasonably safe to apply the dispersion model to obtain the residence time distribution of a fluid in a gas-slurry reactor provided that such reactor does not have any stagnant or semi-stagnant regions.

2) The liquid mixing essential to suspend the solids is mainly caused by the gas bubbles. This mixing proved to be a diminishing function of the gas rate.

3) At the required operating flow rates, the mixing decreases when the liquid flow is increased.

4) A method of calculating solids concentration profiles was presented and compared with the actual data. The solids distribution through the reactor is a function of the gas only through its dependence on the axial dispersion coefficient. Therefore, according to Conclusion 2 there will be a point when adding enormous amounts of gas would not help to suspend any significant amount of settled particles. So if settling can not be solved by increasing the gas flow up to that "limit" two alternatives are possible: to vary the physical design of the column or to implement a solids withdrawal system.

5) The particle distribution profile within the reactor may be essential to the heat transfer design since the solid particle presumably catalyze the hydrogenation reaction.

6) Other factors such as: a) the bubble size effect on the mixing; b) the influence of the feed entrance arrangement; c) the vessel's length-diameter ratio; and d) the physical properties of the components are suspected to be significant in the hydrodynamic study of the reactor and should be further investigated.

NOMENCLATURE

A, B	constants of integration
A_6	surface area of bubble
A_c	SRC commercial plant projected horizontal cross-sectional area
A_p	SRC pilot plant horizontal cross-sectional area
C	concentration
C-curve	tracer response to an ideal pulse input
C_F	concentration of particles in feed stream
C_o	area under the concentration-time curve
C_T	concentration of particles at the exit of the reactor
d	diameter of the tube
D	dispersion or axial dispersion coefficient
dp	diameter of particle
F	volumetric feed rate
Fr	$= pv^2/p_e\ g\ dp$, Froude number, dimensionless
g	acceleration of gravity
kg	gas-to-liquid mass transfer coefficient
L	length of reactor
n_1	mass flux of liquid in longitudinal direction
$\vec{n}_p$	mass flux of particles, a vectorial quantity
n_p	mass flux of particles in longitudinal direction
P	$= \mu L/D$, Peclet number, dimensionless
Re	$= pvd/u$, Reynold's number, dimensionless
t	time
$\bar{t}$	reactor holding time or mean residence time of fluid in a flow reactor

NOMENCLATURE (cont.)

u	fluid velocity
v	linear velocity
v_F	fall velocity of particle through still liquid
W	weight fraction of solids
x	height of the reactor measured from the bottom
ρ	density
ρ_e	effective density, difference between particle and liquid densities
ρ_M	mean density of slurry
μ	viscosity
μ_1	mean of a tracer curve
θ	$= t/\bar{t}$, reduced time, dimensionless
σ^2	variance of a tracer curve or distribution function

Subscripts

C	SRC commercial plant
G	gas
L	liquid
M	slurry
P	SRC pilot plant

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APPENDIX 1

ESTIMATION OF THE PARAMETER VALUES OF THE TREXLERTOWN SRC PILOT PLANT

Operating conditions:

T = 850°F
P = 137 atm

Physical properties of the gas:

Density: According to Wilsonville's pilot plant reports, the density of the gas present at the outlet of the dissolver is 0.065 g/cu.cm (as calculated by using the ideal gas law assuming a constant compressibility factor from a reported value of 0.080 g/cu.cm. at 164 atm and 825°F). On the other hand, the hydrogen feed density at the above mentioned operating conditions is 0.0045 g/cu.cm. Therefore the real value of the mean gas density in the reactor should lay between those extremes.

Viscosity: The viscosity of hydrogen at such temperatures and pressures is 0.012 g/cm. min. The viscosity of the gas mixture is larger than the viscosity of hydrogen alone. There was not a way of estimating what would be the upper viscosity limit.

Physical properties of the slurry:

Density: According to Wilsonville ( ), the densities of the slurry at 825°F and of the solids from blow down were 0.90 and 2.40 g/cu.cm. respectively. The liquid density can then be calculated assuming a value for the weight fraction of solids in the slurry, W. Although there is uncertainty involved in choosing a value for W, it was estimated that 10 percent would be a reasonable intermediate value between the inlet and outlet weight fraction.

$$\rho_M = \left(\frac{W}{\rho_S} + \frac{(1 - W)}{\rho_L}\right)^{-1}$$

or

$$\rho_L = 0.86 \text{ g/cu.cm.}$$

APPENDIX 1 (cont.)

It is worth noting to avoid further confusion that a significant fraction of the solids in the reactor is not mineral matter. This undissolved coal is not relevant to the accumulation problem since it eventually dissolves.

Viscosity: A value for the viscosity of the slurry was obtained from the "Preparation of Coal Conversion Systems Technical Data Book" ( ) which used a 3:1 Recycle Oil to Kentucky No. 9/14 Coal Slurry. At the pertinent conditions this value ranged between 0.69 and 0.87 g/cm. min.

Particle Diameter: An average value for the diameter of the ash particles would be in the proximity of 0.015 cm.

Solids Concentration: The concentration of the mineral matter in the reactor has been reported as 10 percent of the coal fed. A typical concentration of the coal in the solvent is 38 percent. Thus, there is an estimate of 4 weight percent of ash in the feed.

Flow Rates: The flow rates to be assigned to the pilot plant system were scaled-down from the commercial plant projected rates by keeping a constant residence time.

Commercial Plant

Column dimensions		
Diameter	(d)	11 ft
Height	(L)	110 ft
Fluid feed		
Total	(F_M)	15,790 ton/day
Coal		6,000 ton/day
Gas feed	(F_G)	17-24 Mscf/ton MF coal

For the fluid:

$$\frac{\bar{t}_p}{\bar{t}_c} = \left(\frac{L_p}{L_c}\right)\left(\frac{F_{MC}}{F_{MP}}\right)\left(\frac{A_p}{A_c}\right)\left(\frac{P_p}{P_c}\right) = 1$$

APPENDIX 1 (cont.)

Therefore,

$$F_{MP} = F_c \left(\frac{L_P}{L_c}\right)\left(\frac{A_P}{A_c}\right)\left(\frac{P_{MP}}{P_{MC}}\right)$$

$$= (15790 \text{ ton/day})\ (0.045)\ (0.00023)\ (1)$$

$$= 0.163 \text{ ton/day} = 103 \text{ g/min}$$

$$V_{MP} = \frac{F_{MP}}{P_{MP}\ A_P} = 5.6 \text{ cm/min}$$

For the gas:

$$F_{GC} = 17{,}000 - 25{,}000 \text{ scf/ton MF coal}$$

$$= 690 - 1015 \text{ cf/ton MF coal at the operating conditions}$$

Therefore,

$$F_{GP} = (0.163 \text{ ton/day})\ (.38 \text{ ton coal/ton})\ (690 - 1015 \text{ cf/ton coal})$$

$$= 42.7 \text{ to } 62.9 \text{ cf/day} = 840 - 1240 \text{ cu.cm/min.}$$

$$V_{GP} = \frac{F_{GP}}{A_P} = 41.4 \text{ to } 61.2 \text{ cm/min}$$

APPENDIX 2

COMPUTER PROGRAM TO ANALYZE THE COLD-FLOW MODEL OUTPUT

A computer program was developed to plot the normalized residence time distributions and to calculate the characteristic properties of such curves.

The input of the enclosed program consists in:

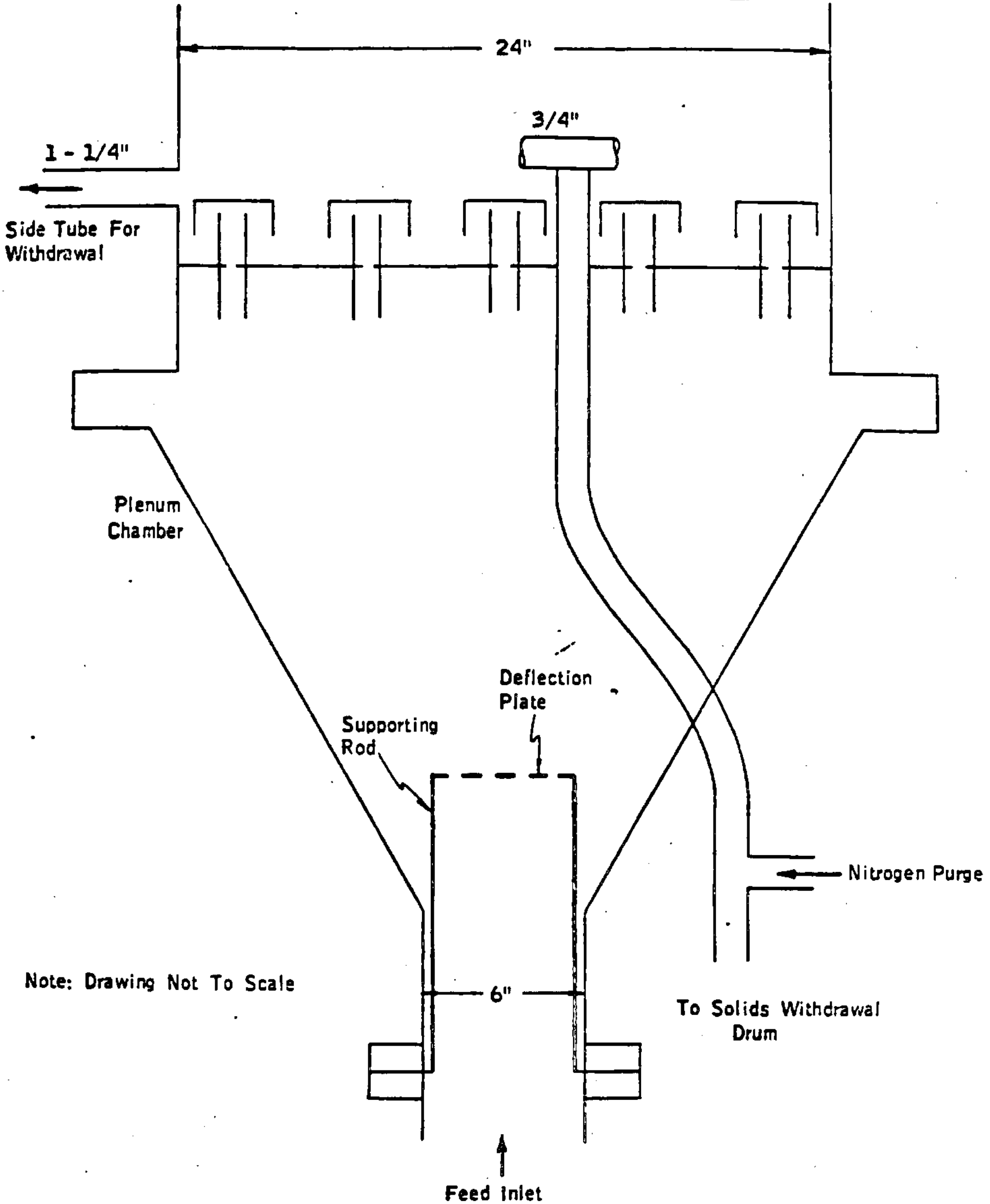
Card 1	- number of runs to be analyzed
Card 2	- slurry volumetric flow rate (liter/min) - gas volumetric flow rate (liter/min) - gas holdup (volume fraction of gas in the reactor) - vessel volume (liter) - number of selected time-concentration points
Card 3 to Card n	- time (min) - concentration (cm) points NOTE: the program includes a factor to convert the cm of chart paper into tracer concentrations (g moles/liter).
Card n+1 to Card n+11	- graphing specifications
Card n+12 to end	- other sets of runs as described from Card 2 to Card n.

TABLE 1

PARAMETERS OF THE SYSTEMS UNDER STUDY

	Ash-Solvent-Hydrogen System	Silica-Water-Nitrogen System
Liquid Properties:		
Viscosity (μL)	0.74 $\frac{g}{cm\ min}$	0.60 $\frac{g}{cm\ min}$
Density (ρL)	0.86 $\frac{g}{cu\ cm}$	1.00 $\frac{g}{cu\ cm}$
Gas Properties:		
Viscosity (μG)	0.012 $\frac{g}{cm\ min}$	0.011 $\frac{g}{cm\ min}$
Density (ρG)	0.048 $\frac{g}{cu\ cm}$	0.0011 $\frac{g}{cu\ cm}$
Solid Properties:		
Particle Diameter (dp)	0.015 cm	0.010 cm (140 mesh)
Density (ρs)	2.40 $\frac{g}{cu\ cm}$	2.41 $\frac{g}{cu\ cm}$
Concentration (W)	0.04	0.04
Slurry Properties:		
Mean Density (ρm)	0.90 $\frac{g}{cu\ cm}$	1.02 $\frac{g}{cu\ cm}$
Effective Density (ρe)	1.54 $\frac{g}{cu\ cm}$	1.41 $\frac{g}{cu\ cm}$
Flow Velocities:		
Slurry ($v M$)	5.6 $\frac{cm}{min}$	4.0 $\frac{cm}{min}$
Gas ($v G$)	51.3 $\frac{cm}{min}$	37.0 $\frac{cm}{min}$
Residence Time:		
Slurry ($\bar{t}$)	27.2 min	38.1 min

SOLIDS WITHDRAWAL DESIGN CONFIGURATION COLUMN INTERNALS



Note: Drawing Not To Scale

FIGURE 1

COLD MODEL DISTRIBUTOR ELEMENT

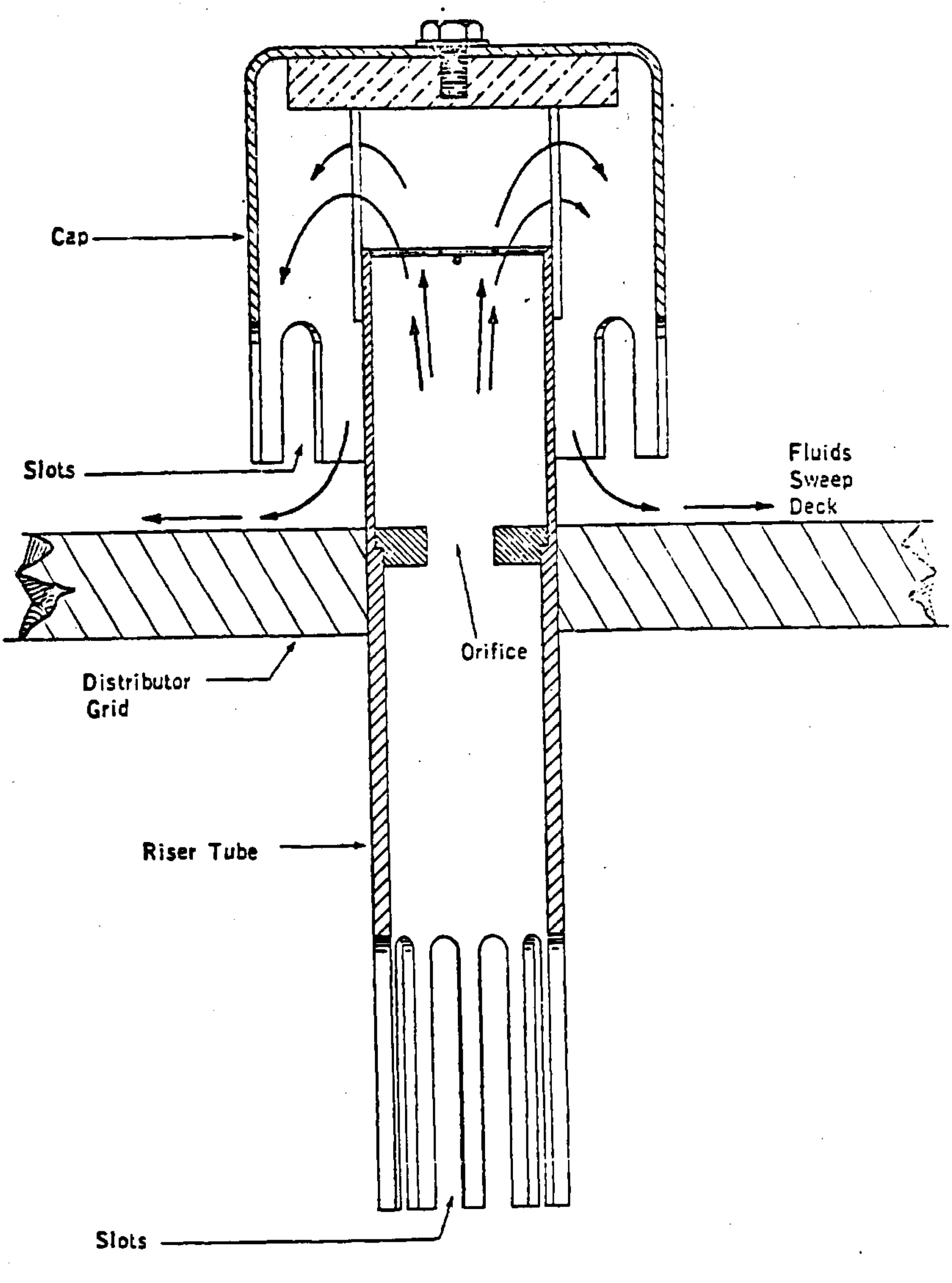
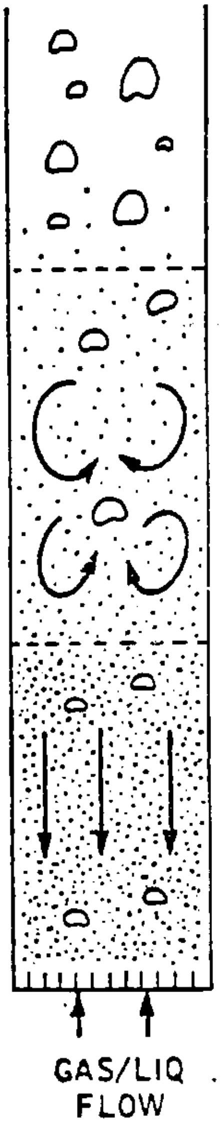


FIGURE 2

FIGURE 3

LARGE COAL PARTICLES SEGREGATE
SOLIDS WITHDRAWAL SYSTEM NEEDED



6"Ø Column	24" Ø Column		
Batch Fluidization Pattern	Column Height (Ft)	ΔP/L Psi/ft	Coal Larger Than 8 Mesh (wt.%)
Dilute Coal Region	18	0.30	-
Intense Backmixing Region	14	0.37	<0.5
	10	0.39	<0.5
Dense Bed Region (Large Particles)	3.5	0.42	2
	1	-	55

FIGURE 4

SRC Dissolver Model

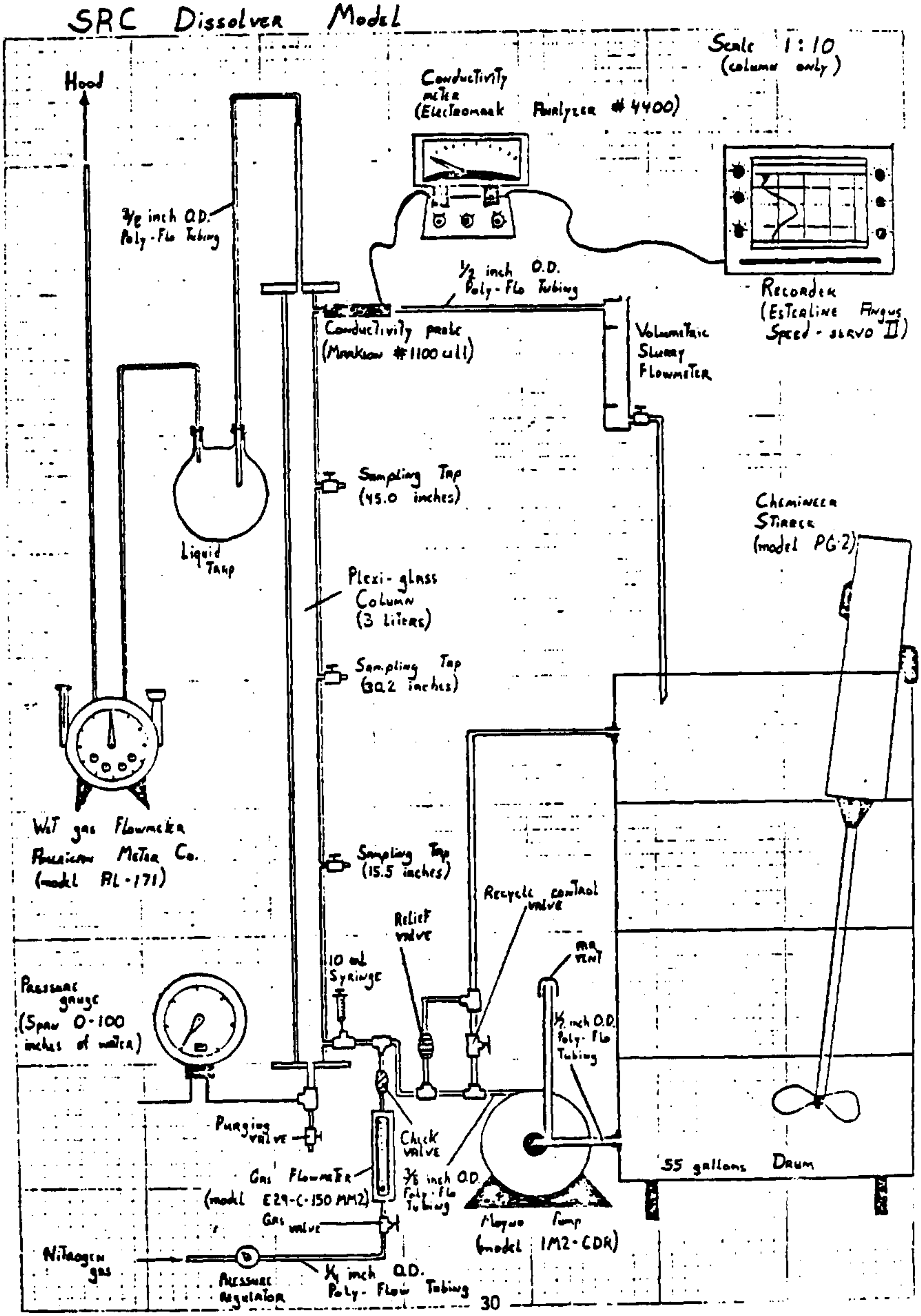


FIGURE 5

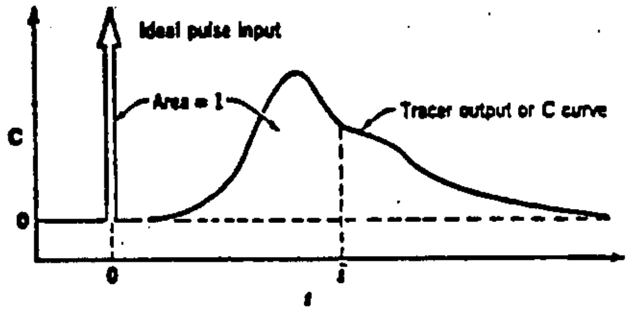
The C Curve

With no tracer initially present anywhere impose an idealized instantaneous pulse of tracer on the stream entering the vessel. Such an input is often called a delta function or impulse. The normalized response is then called the C curve.

To perform this normalization we divide the measured concentration by Q, the area under the concentration-time curve. Thus we have on normalization

$$\int_0^\infty C\,dt = \int_0^\infty \frac{C}{Q}\,dt = 1 \qquad \text{where} \qquad Q = \int_0^\infty C\,dt$$

Figure 5 shows the C curve and its properties.



Typical downstream signal, called the C curve, in response to an upstream δ-function input signal.

FIGURE 6

The Mean and Variance. It is frequently desirable to characterize a distribution by a few numerical values. For this purpose the most important measure is the location of the distribution. This is called the mean value or the centroid of the distribution. Thus for a C versus t curve the mean is given by

$$\mu_1 = \bar{t} = \frac{\int_0^\infty tC\,dt}{\int_0^\infty C\,dt}$$

If the distribution curve is only known at a number of discrete time values t_i then

$$\mu_1 = \bar{t} \cong \frac{\sum t_i C_i \,\Delta t_i}{\sum C_i \,\Delta t_i}$$

The next most important descriptive quantity is the spread of the distribution. This is commonly measured by the variance σ^2, defined as

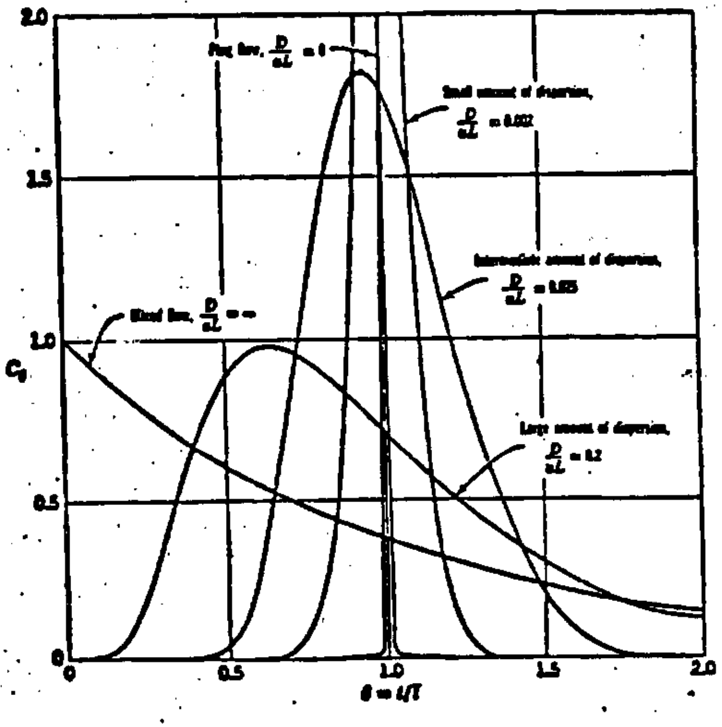
$$\sigma^2 = \frac{\int_0^\infty (t - \bar{t})^2 C\,dt}{\int_0^\infty C\,dt} = \frac{\int_0^\infty t^2 C\,dt}{\int_0^\infty C\,dt} - \bar{t}^2$$

Again, in discrete form

$$\sigma^2 \cong \frac{\sum (t_i - \bar{t})^2 C_i \,\Delta t_i}{\sum C_i \,\Delta t_i} = \frac{\sum t_i^2 C_i \,\Delta t_i}{\sum C_i \,\Delta t_i} - \bar{t}^2$$

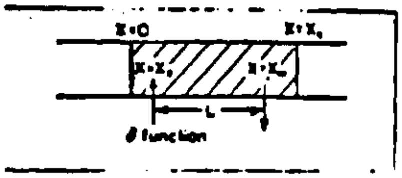
The variance represents the square of the spread of the distribution and has units of (time)^2. It is particularly useful for matching experimental curves to one of a family of theoretical curves.

FIGURE 7



C curves in closed vessels for various extents of backmixing as predicted by the dispersion model.

FIGURE 8



The boundary-value problem then had the form:

$$\frac{\partial c_a'}{\partial \theta} + \frac{\partial c_a'}{\partial x} - \frac{1}{P_a}\frac{\partial^2 c_a'}{\partial x^2} = 0 \qquad x \le 0$$

$$\frac{\partial c'}{\partial \theta} + \frac{\partial c'}{\partial x} - \frac{1}{P}\frac{\partial^2 c'}{\partial x^2} = \delta(x - x_0)\delta(\theta) \qquad 0 \le x \le x_1$$

$$\frac{\partial c_b'}{\partial \theta} + \frac{\partial c_b'}{\partial x} - \frac{1}{P_b}\frac{\partial^2 c'}{\partial x^2} = 0 \qquad x_1 \le x$$

with boundary conditions,

$$c_a'(x, 0) = c'(x, 0) = c_b(x, 0) = 0$$

$$c_a'(-\infty, \theta) = \text{finite}$$

$$c_a'(0^-, \theta) = c'(0^+, \theta)$$

$$c_a'(0^-, \theta) - \frac{1}{P_a}\frac{\partial c_a'}{\partial x}(0^-, \theta) = c'(0^+, \theta) - \frac{1}{P}\frac{\partial c'}{\partial x}(0^+, \theta)$$

$$c'(x_1^-, \theta) - \frac{1}{P}\frac{\partial c'}{\partial x}(x_1^-, \theta) = c_b'(x_1^+, \theta) - \frac{1}{P_b}\frac{\partial c_b'}{\partial x}(x_1^+, \theta)$$

$$c'(x_1^-, \theta) = c_b'(x_1^+, \theta)$$

$$c_b'(+\infty, \theta) = \text{finite}$$

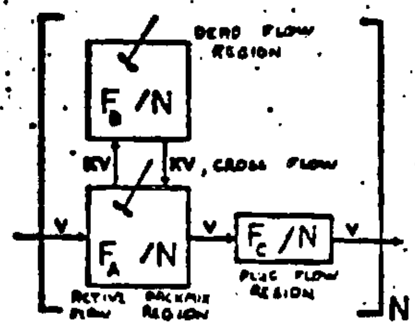
solution for the special case where $D_a = D_b = 0$.

$$\mu_1 = 1 + \frac{1}{P}\left[2 - (1 - a)e^{-Px_0} - (1 - b)e^{-P(x_1 - x_m)}\right]$$

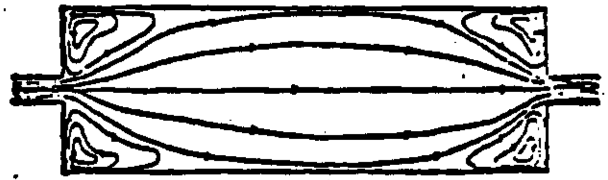
$$\sigma^2 = \frac{2}{P} + \frac{1}{P^2}\Big(8 + 2(1 - a)(1 - b)e^{-Px_1}$$
$$- (1 - a)e^{-Px_0}\left[4x_0P + 4(1 + a) + (1 - a)e^{-Px_0}\right]$$
$$- (1 - b)e^{-P(x_1 - x_m)}\left[4(x_1 - x_m)P + 4(1 + b) + (1 - b)e^{-P(x_1 - x_m)}\right]\Big)$$

where $a = P/P_a$ and $b = P/P_b$.

FIGURE 9



—Finite-stage model approximating actual flow patterns. Agitators in tanks *a* and *b* indicate perfectly stirred units. Tank *c* is a plug-flow unit. The symbols within each block indicate their fractional volume.



—Flow patterns in a short tube. Dead-flow regions are found in the corners.

FIGURE 10

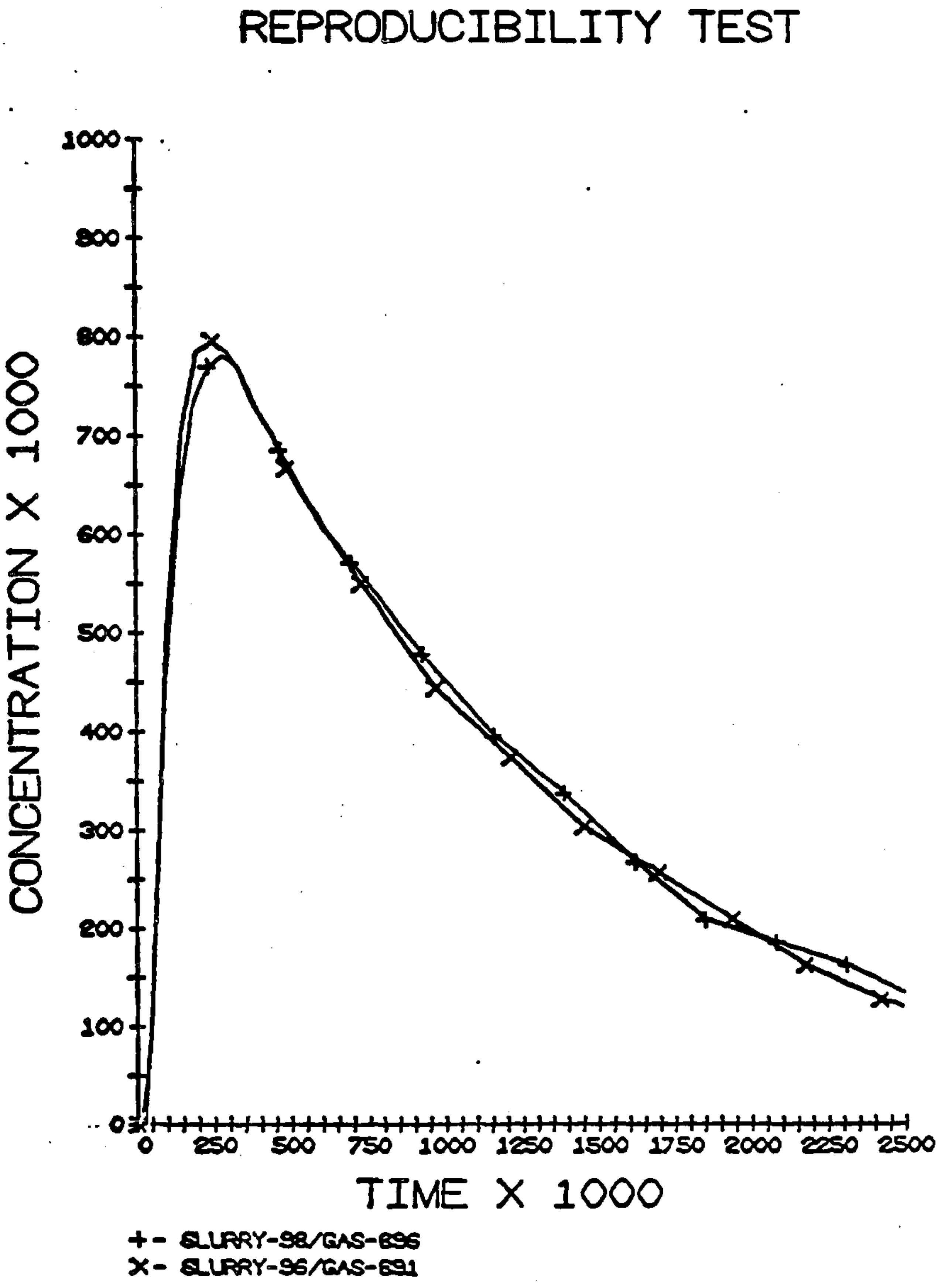
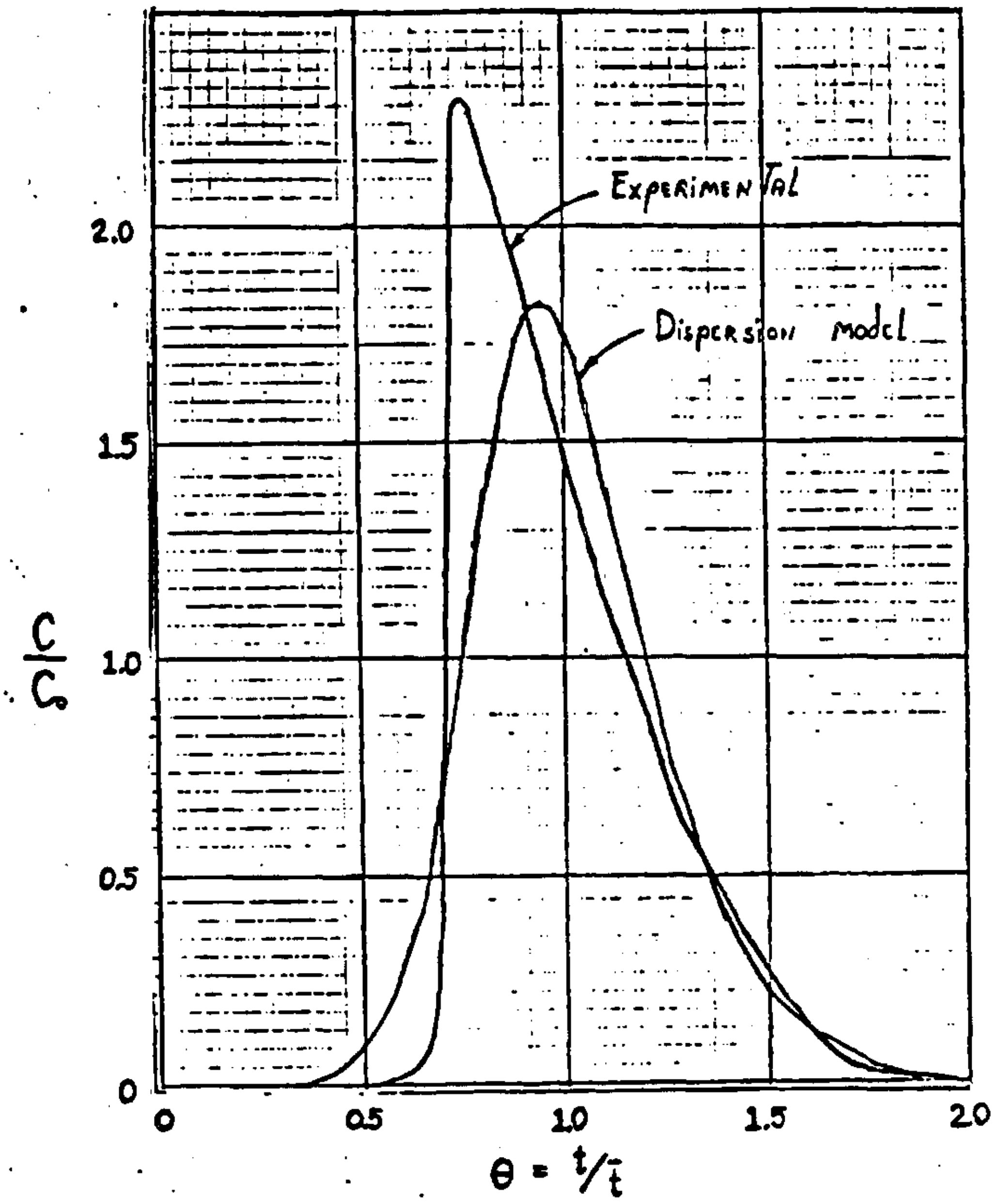
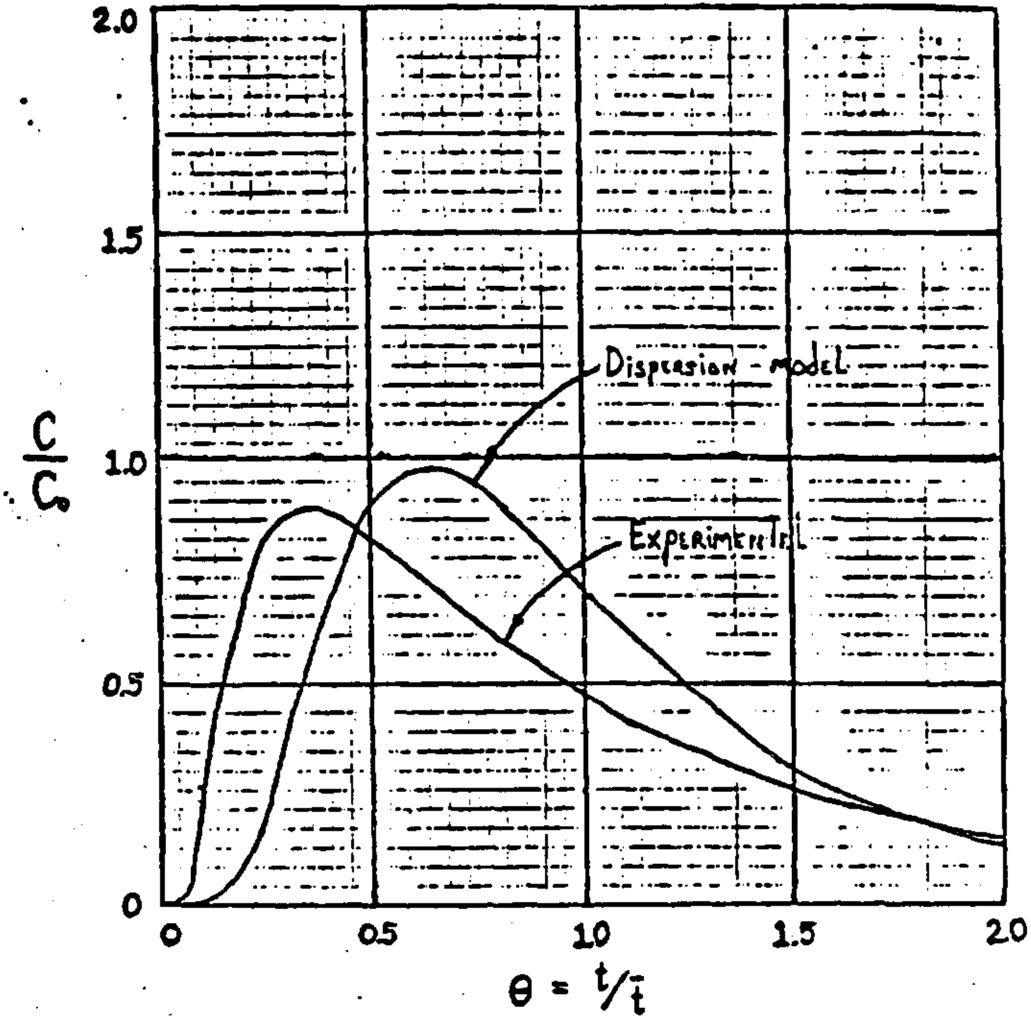

REPRODUCIBILITY TEST
CONCENTRATION X 1000
1000
900
800
700
600
500
400
300
200
100
0
TIME X 1000
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
+- SLURRY-98/GAS-696
X- SLURRY-96/GAS-691

FIGURE 11



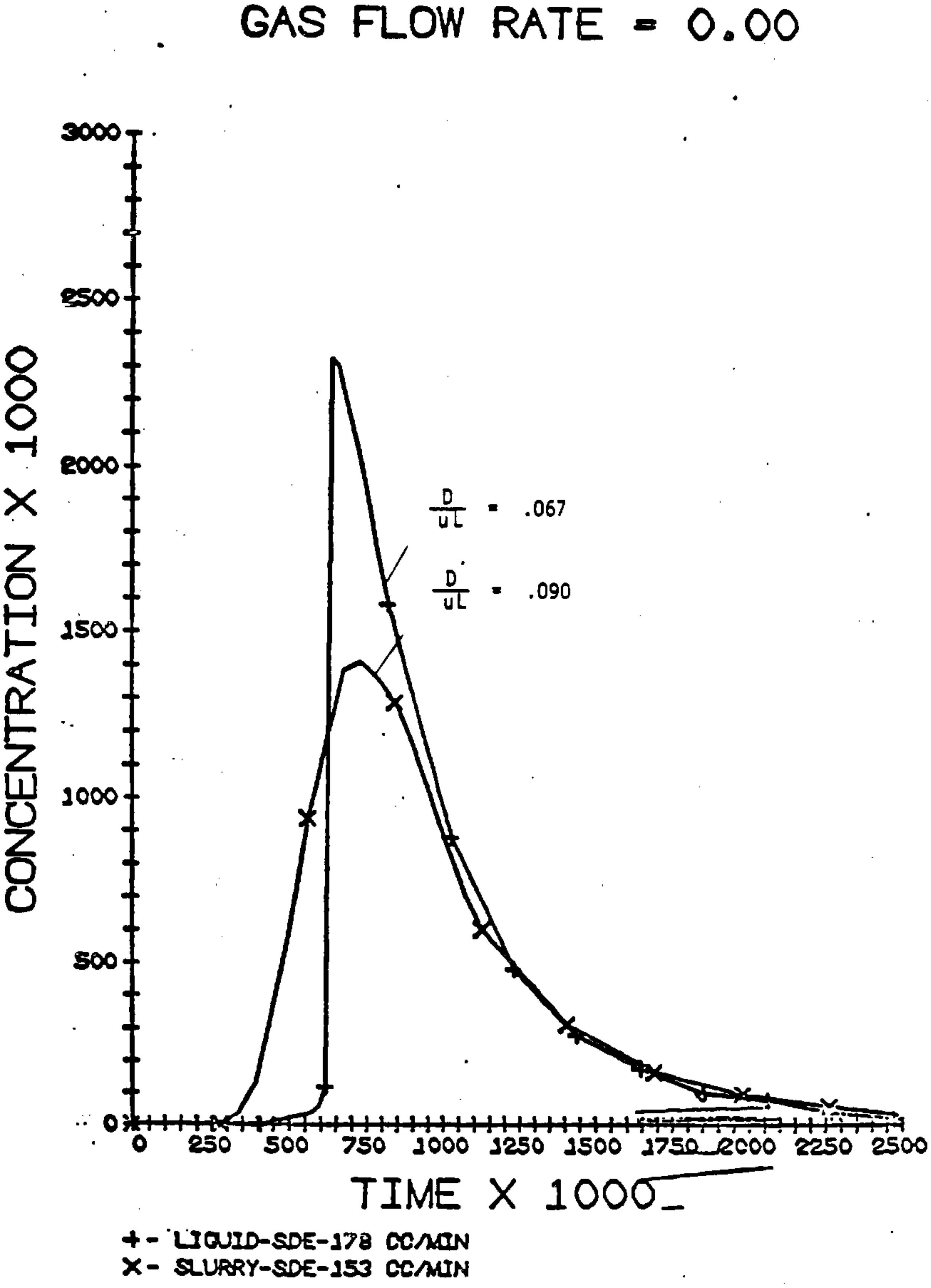
"DISPERSION NUMBER = .025"

FIGURE 12



"DISPERSION NUMBER = .20"

FIGURE 13



GAS FLOW RATE = 0.00

FIGURE 14

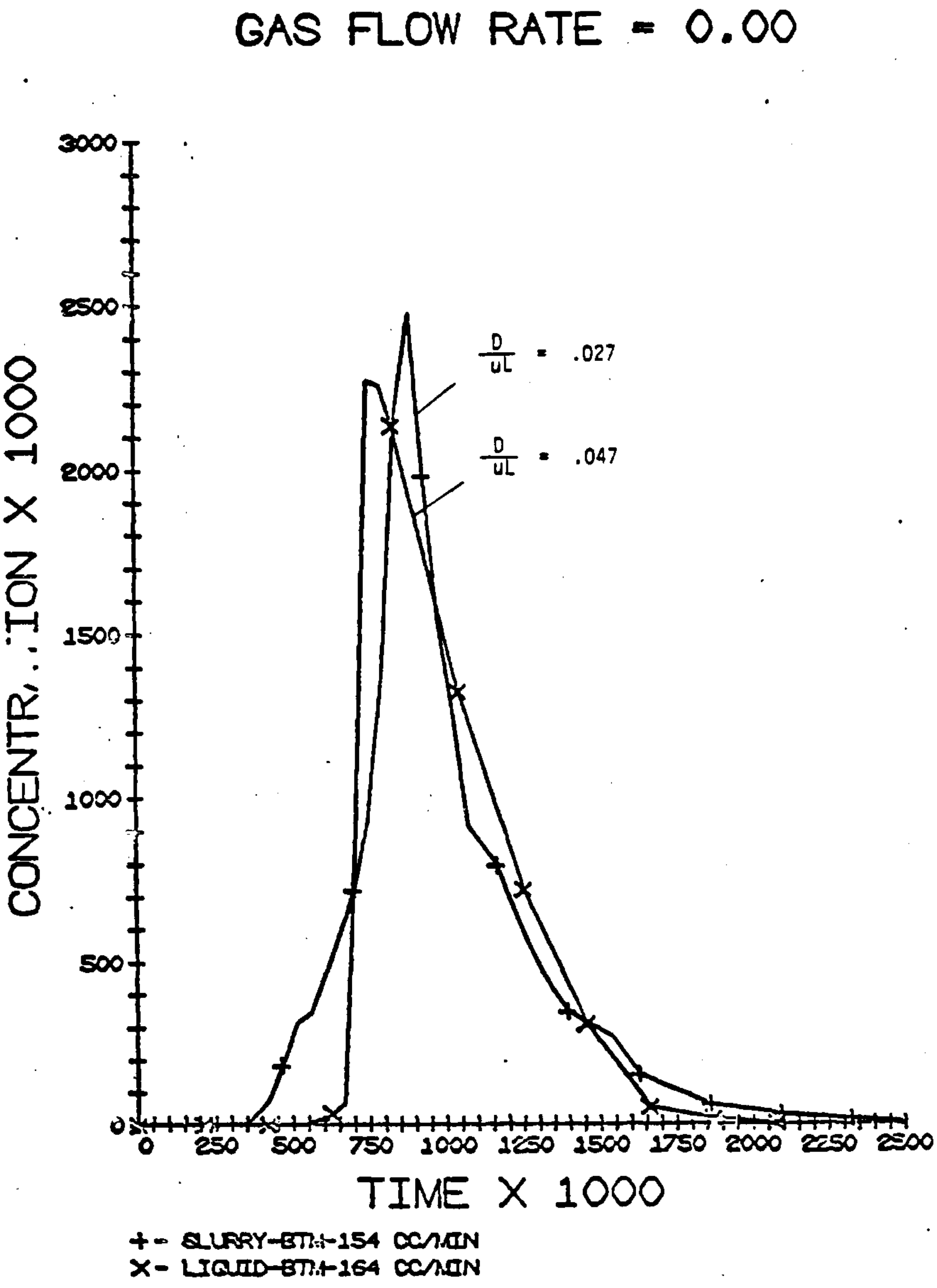

GAS FLOW RATE = 0.00
CONCENTR..ION X 1000
3000
2500
2000
1500
1000
500
0
TIME X 1000
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
$\frac{D}{uL}$ = .027
$\frac{D}{uL}$ = .047
+ - SLURRY-BTM-154 CC/MIN
X - LIQUID-BTM-164 CC/MIN

FIGURE 15

GAS FLOW RATE = 82.

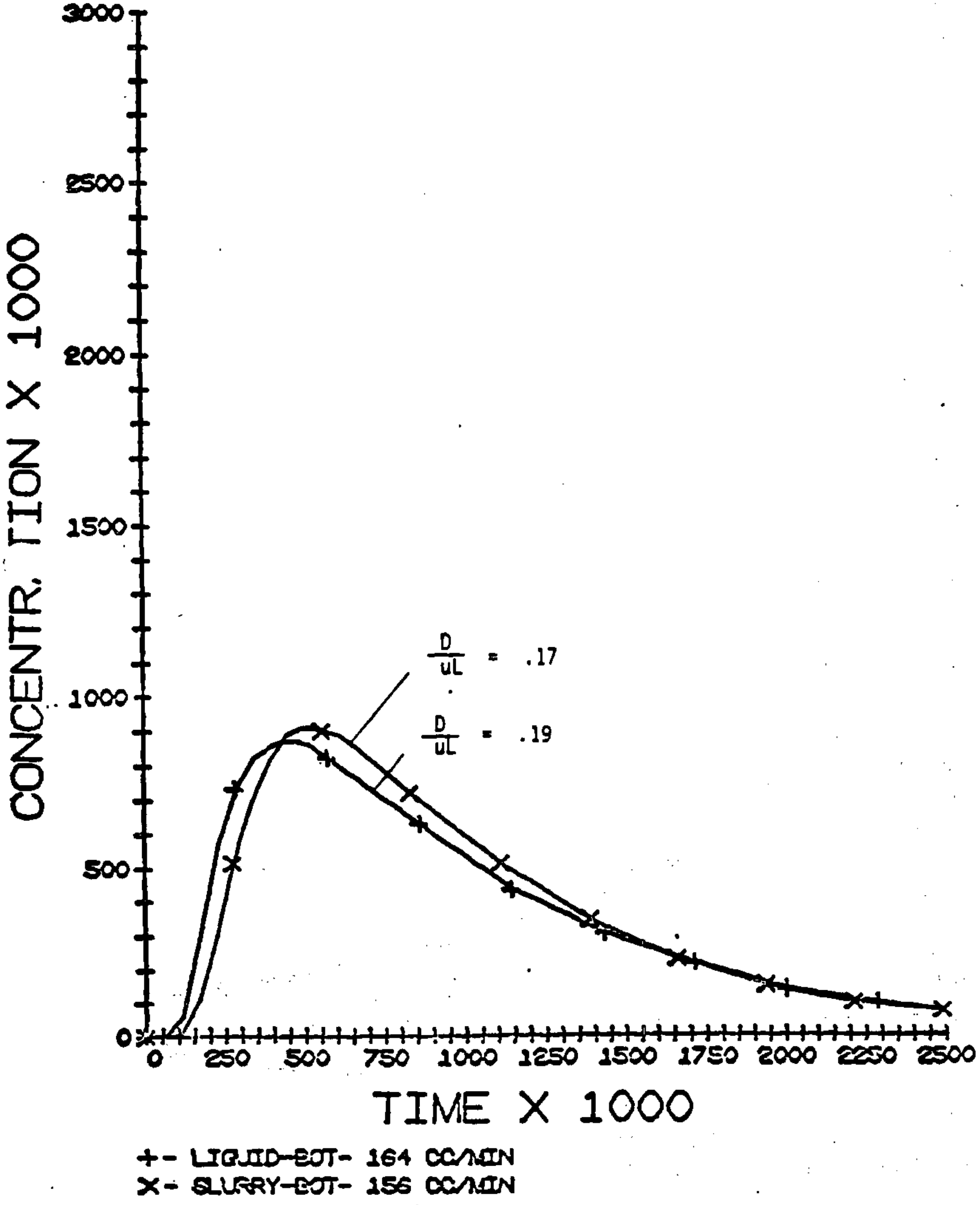


FIGURE 16

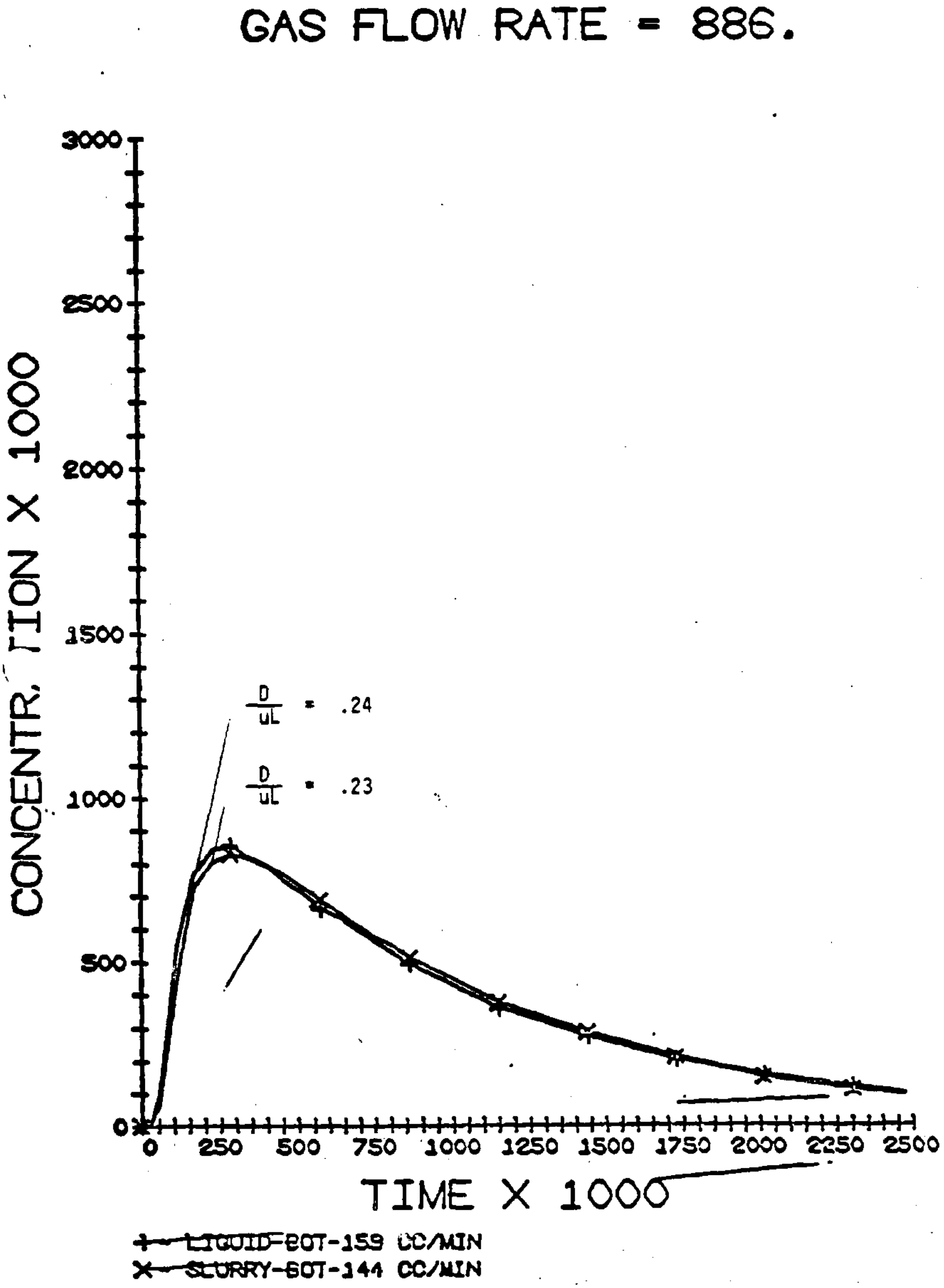

GAS FLOW RATE = 886.

FIGURE 17

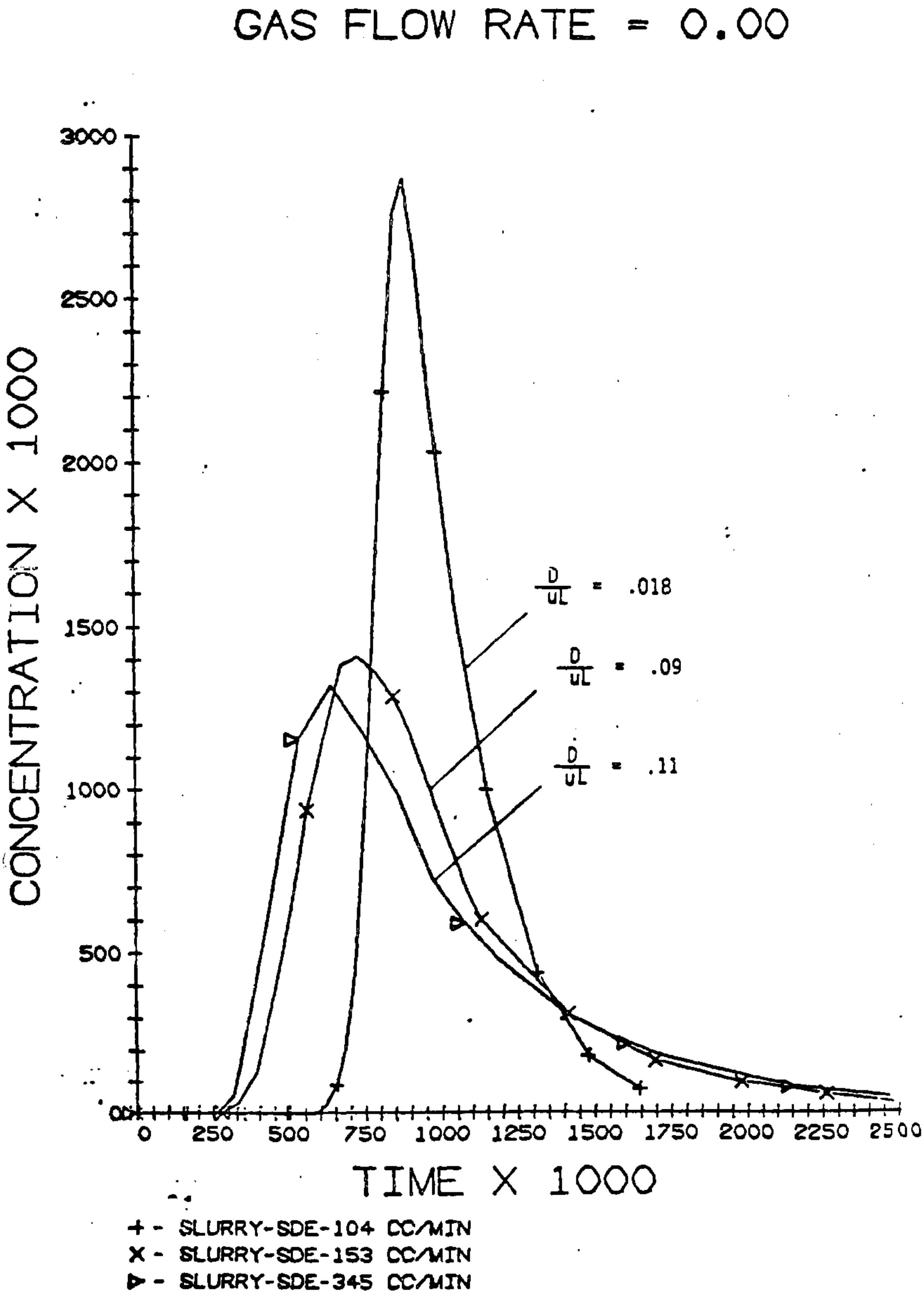

GAS FLOW RATE = 0.00
CONCENTRATION X 1000
3000
2500
2000
1500
1000
500
0
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
TIME X 1000
D/uL = .018
D/uL = .09
D/uL = .11
+ - SLURRY-SDE-104 CC/MIN
X - SLURRY-SDE-153 CC/MIN
▷ - SLURRY-SDE-345 CC/MIN

FIGURE 18

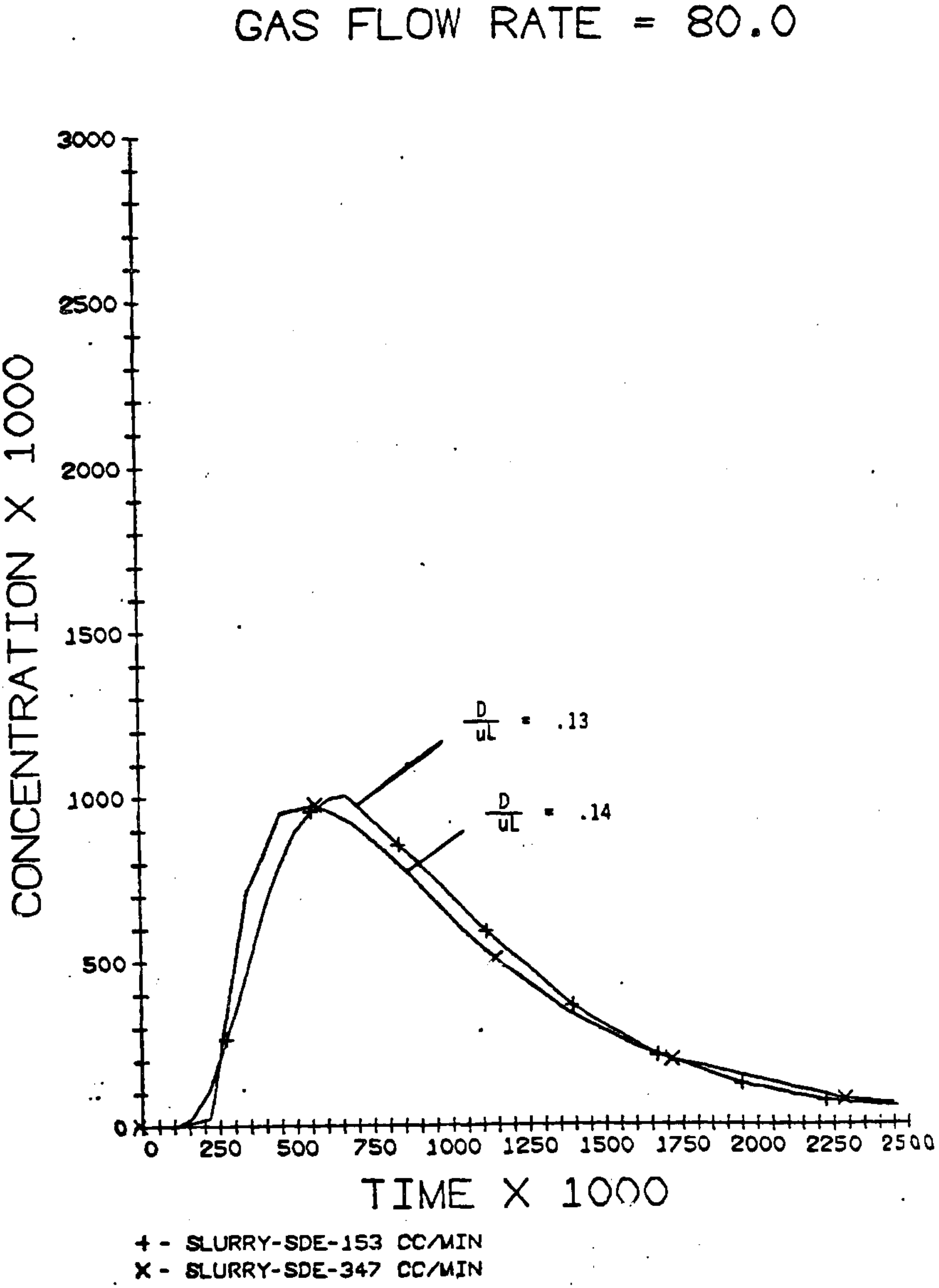

GAS FLOW RATE = 80.0
CONCENTRATION X 1000
3000
2500
2000
1500
1000
500
0
TIME X 1000
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
$\frac{D}{uL}$ = .13
$\frac{D}{uL}$ = .14
+ - SLURRY-SDE-153 CC/MIN
X - SLURRY-SDE-347 CC/MIN

FIGURE 19

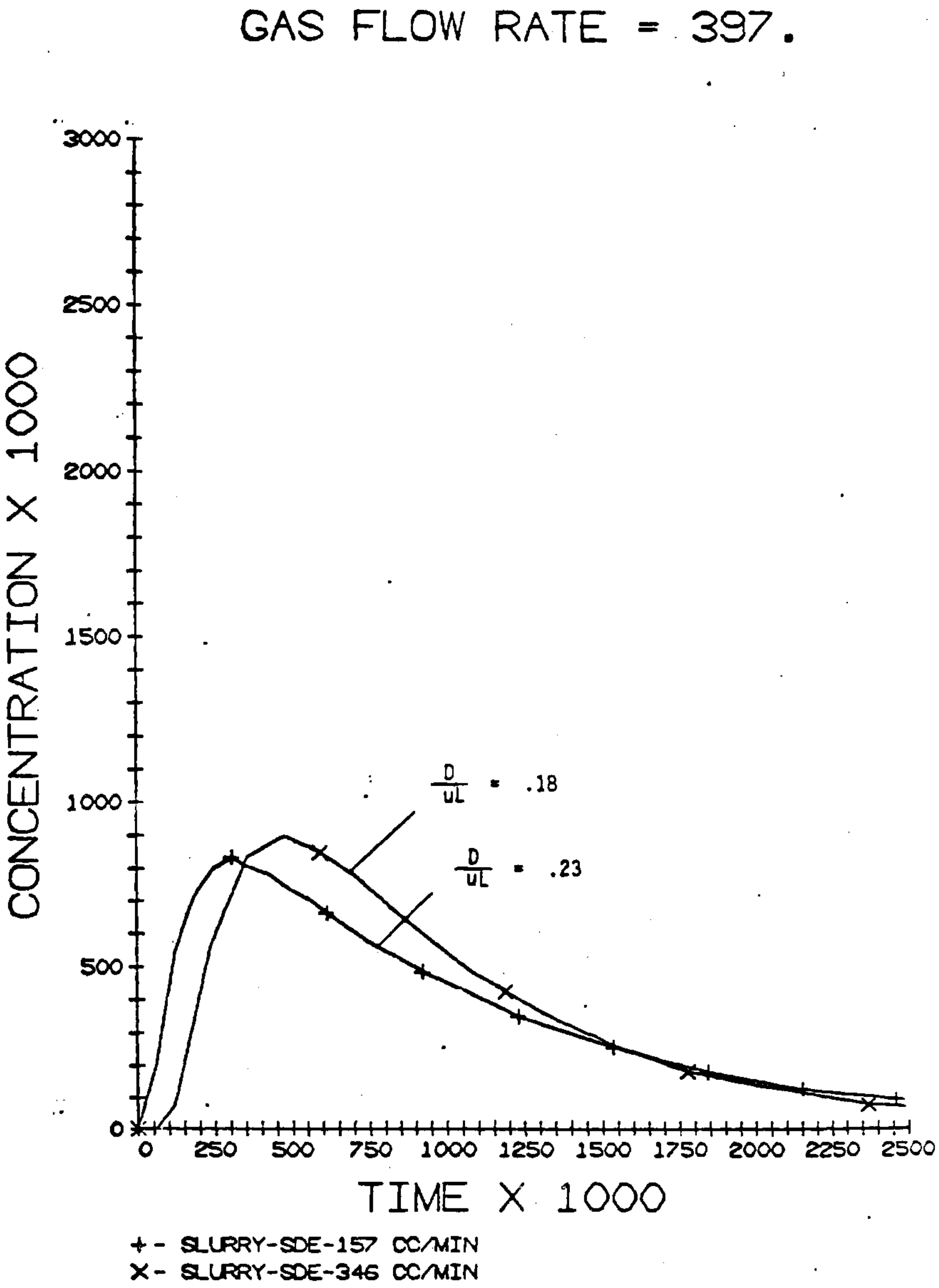

GAS FLOW RATE = 397.
CONCENTRATION X 1000
0
500
1000
1500
2000
2500
3000
TIME X 1000
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
$\frac{D}{uL}$ = .18
$\frac{D}{uL}$ = .23
+ - SLURRY-SDE-157 CC/MIN
X - SLURRY-SDE-346 CC/MIN

FIGURE 20

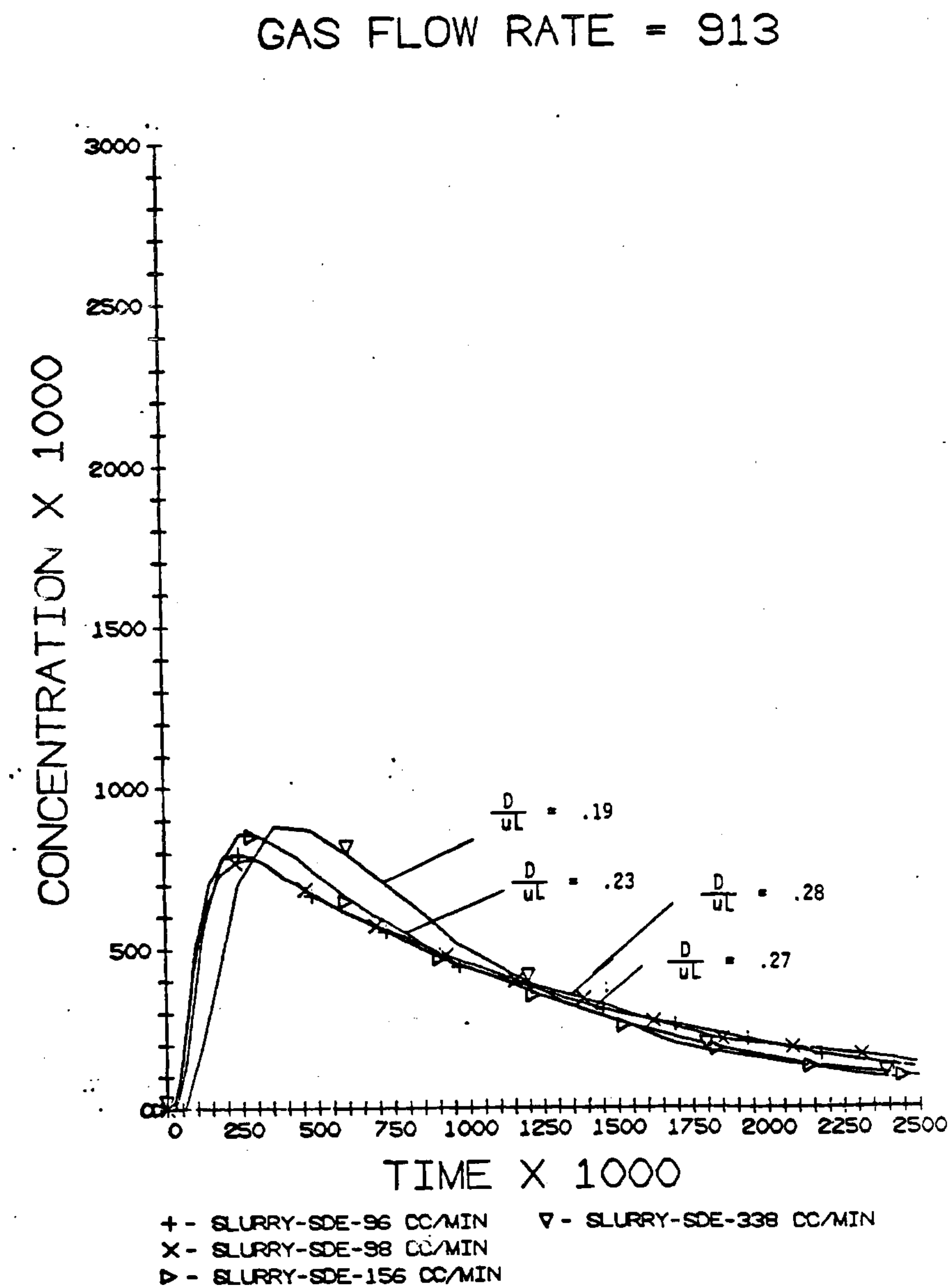
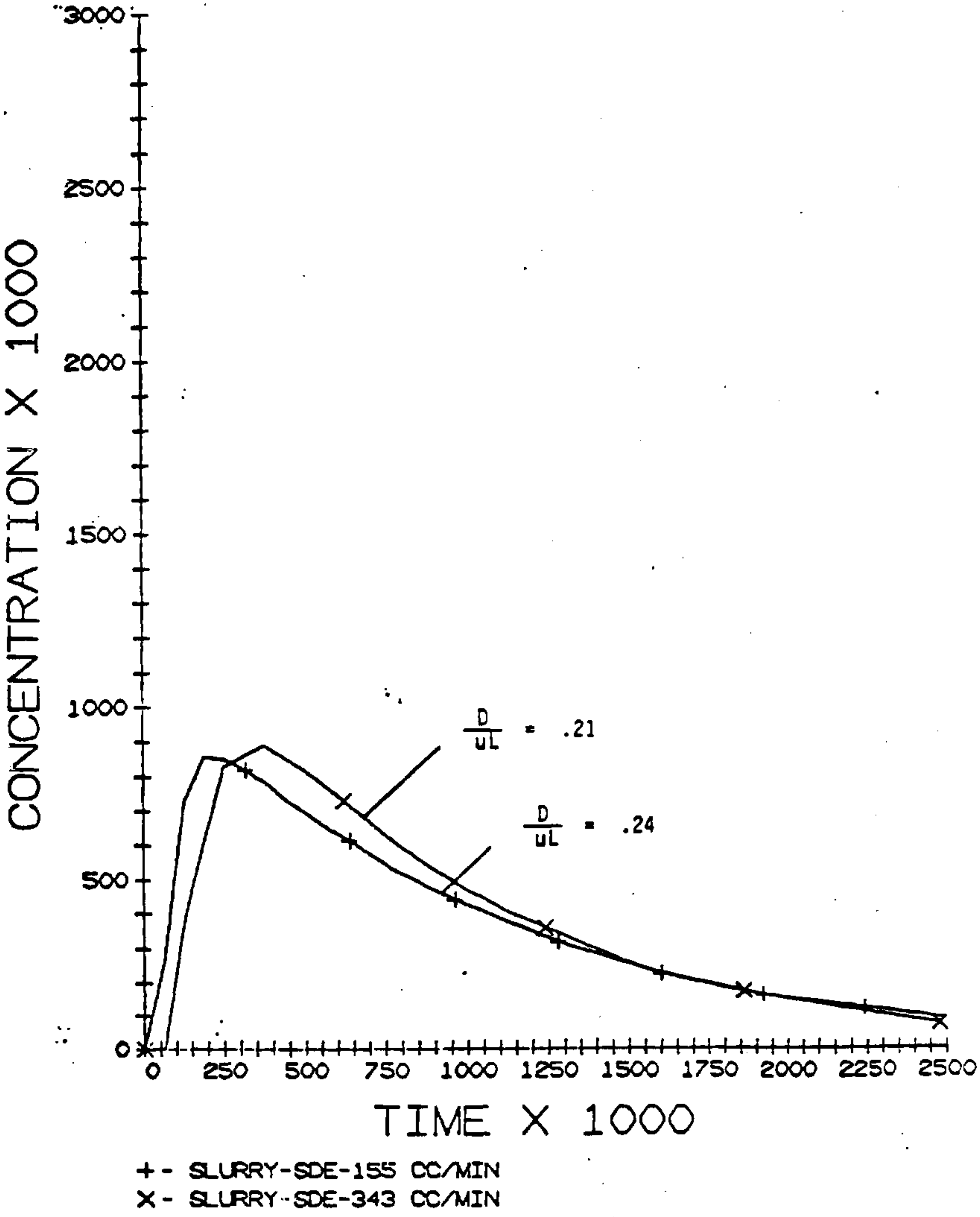

GAS FLOW RATE = 913
3000
2500
2000
1500
1000
500
0
CONCENTRATION X 1000
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
TIME X 1000
$\frac{D}{uL}$ = .19
$\frac{D}{uL}$ = .23
$\frac{D}{uL}$ = .28
$\frac{D}{uL}$ = .27
+ - SLURRY-SDE-96 CC/MIN
X - SLURRY-SDE-98 CC/MIN
▷ - SLURRY-SDE-156 CC/MIN
▽ - SLURRY-SDE-338 CC/MIN

FIGURE 21

GAS FLOW RATE = 2255



+ - SLURRY-SDE-155 CC/MIN
X - SLURRY-SDE-343 CC/MIN

FIGURE 22

SLURRY-SDE- 99 CC/MIN

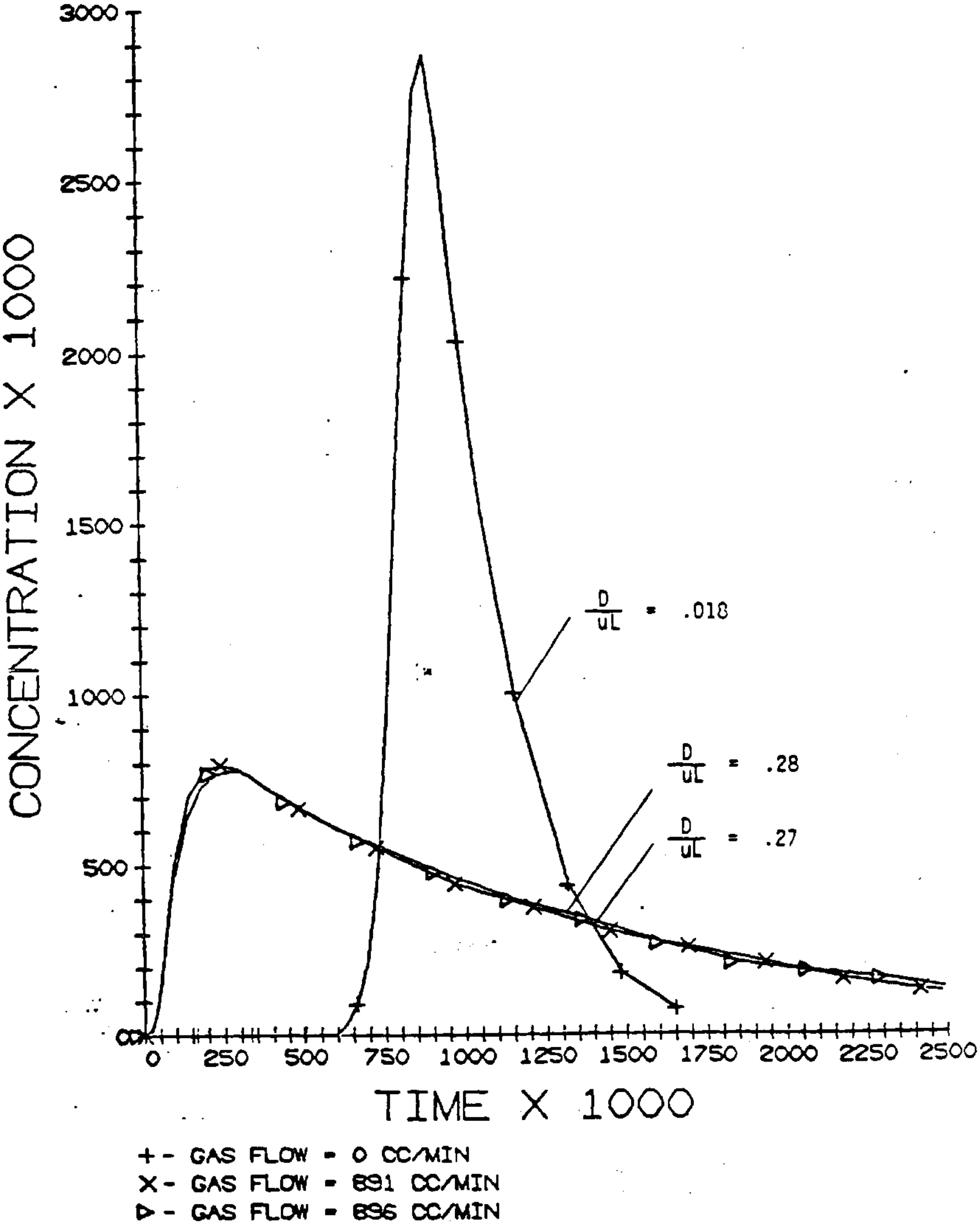


FIGURE 23

SLURRY-SDE-155 CC/MIN

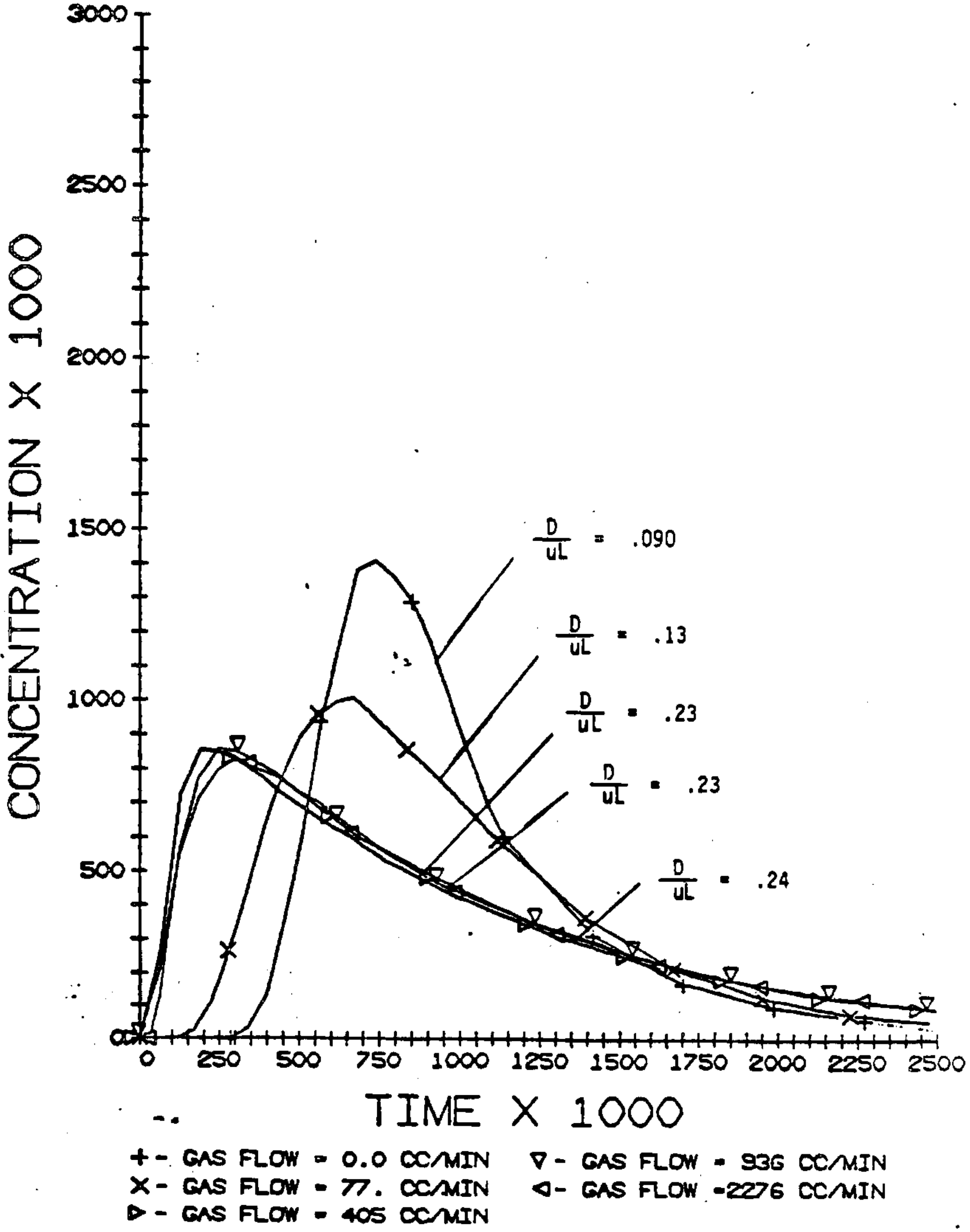


FIGURE 24

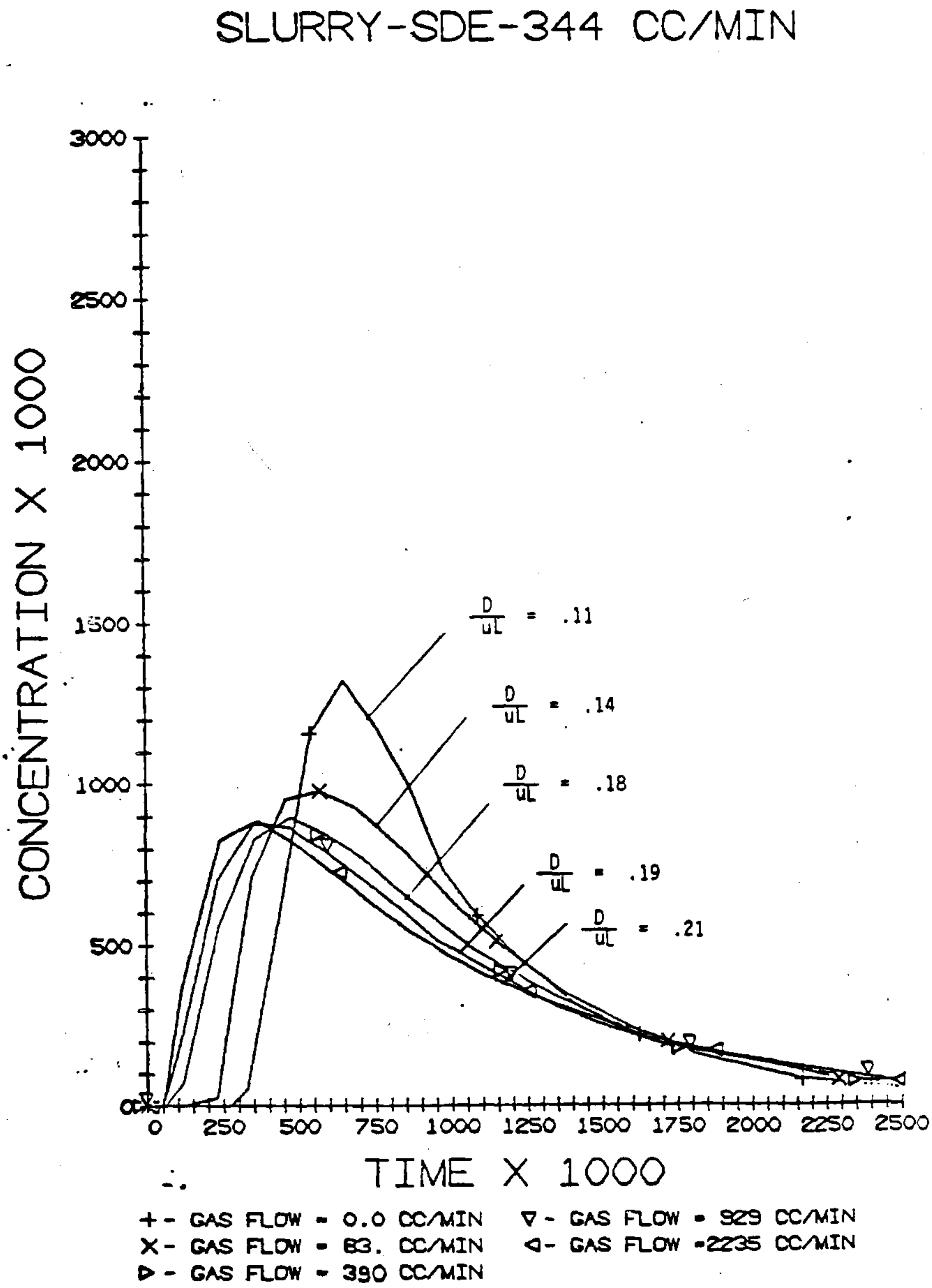
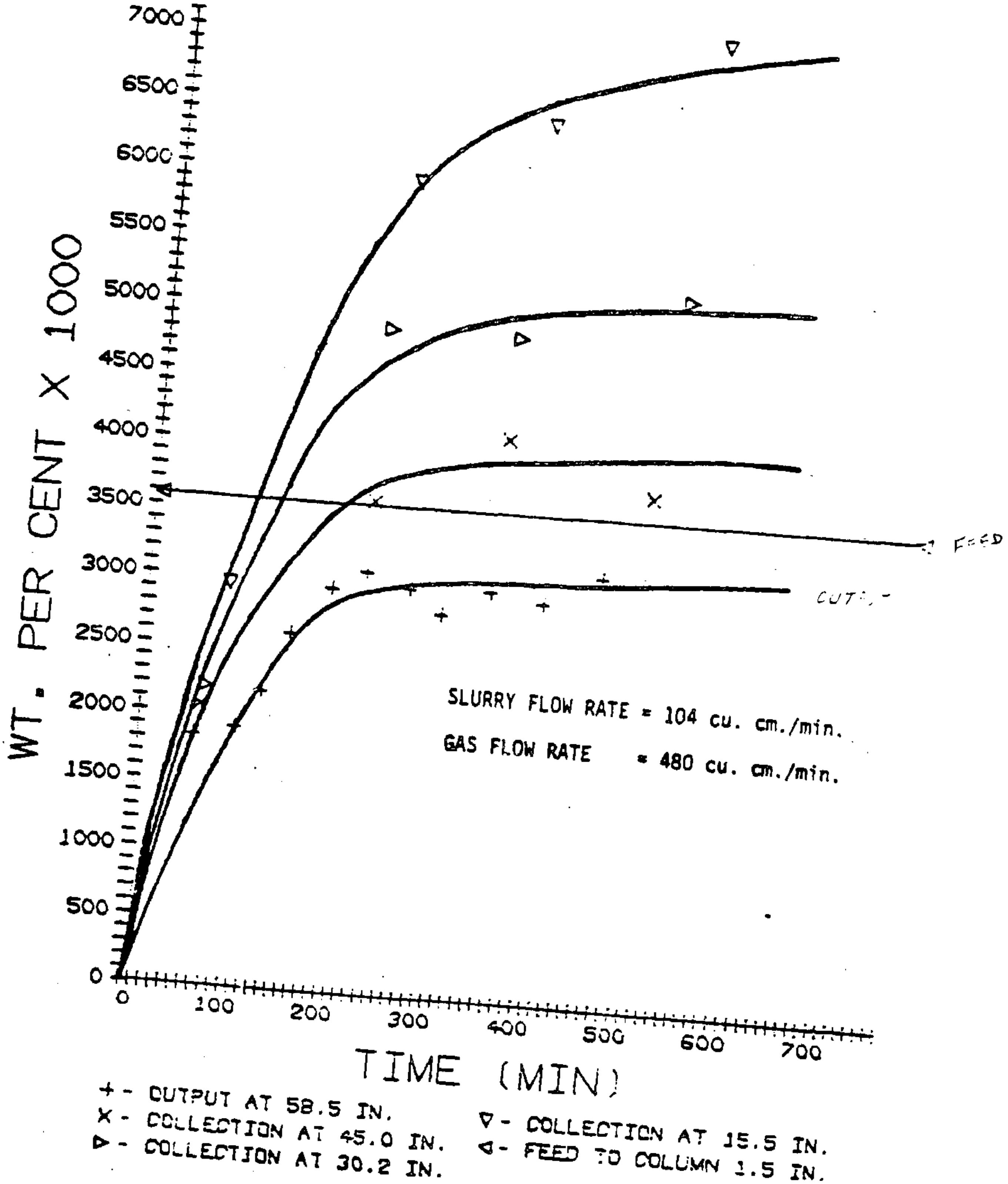

SLURRY-SDE-344 CC/MIN
CONCENTRATION X 1000
0
500
1000
1500
2000
2500
3000
TIME X 1000
0
250
500
750
1000
1250
1500
1750
2000
2250
2500
D/uL = .11
D/uL = .14
D/uL = .18
D/uL = .19
D/uL = .21
+ - GAS FLOW = 0.0 CC/MIN
X - GAS FLOW = 83. CC/MIN
▷ - GAS FLOW = 390 CC/MIN
▽ - GAS FLOW = 929 CC/MIN
◁ - GAS FLOW = 2235 CC/MIN

FIGURE 25

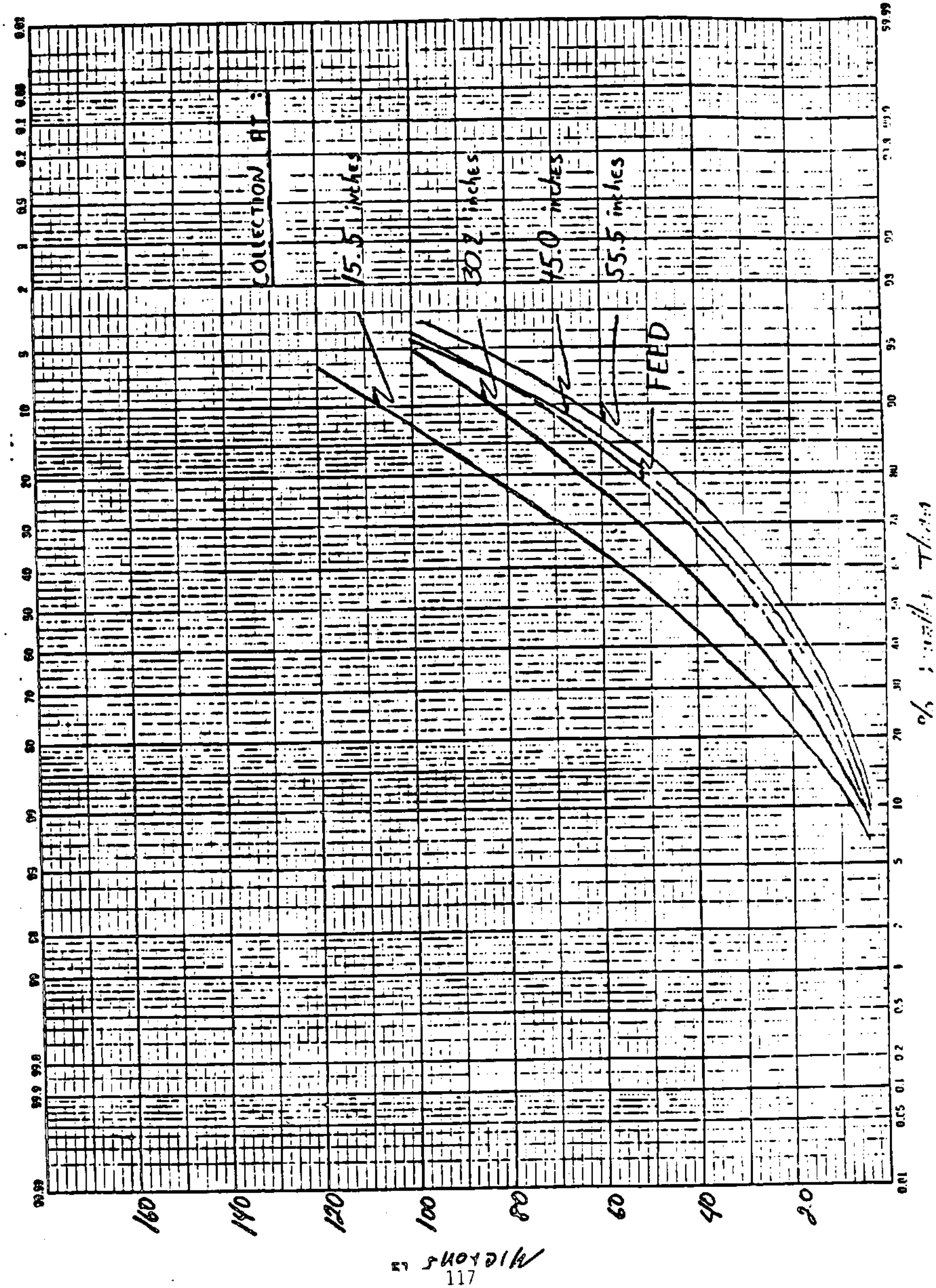
CONCENTRATION PROFILES



SLURRY FLOW RATE = 104 cu. cm./min.

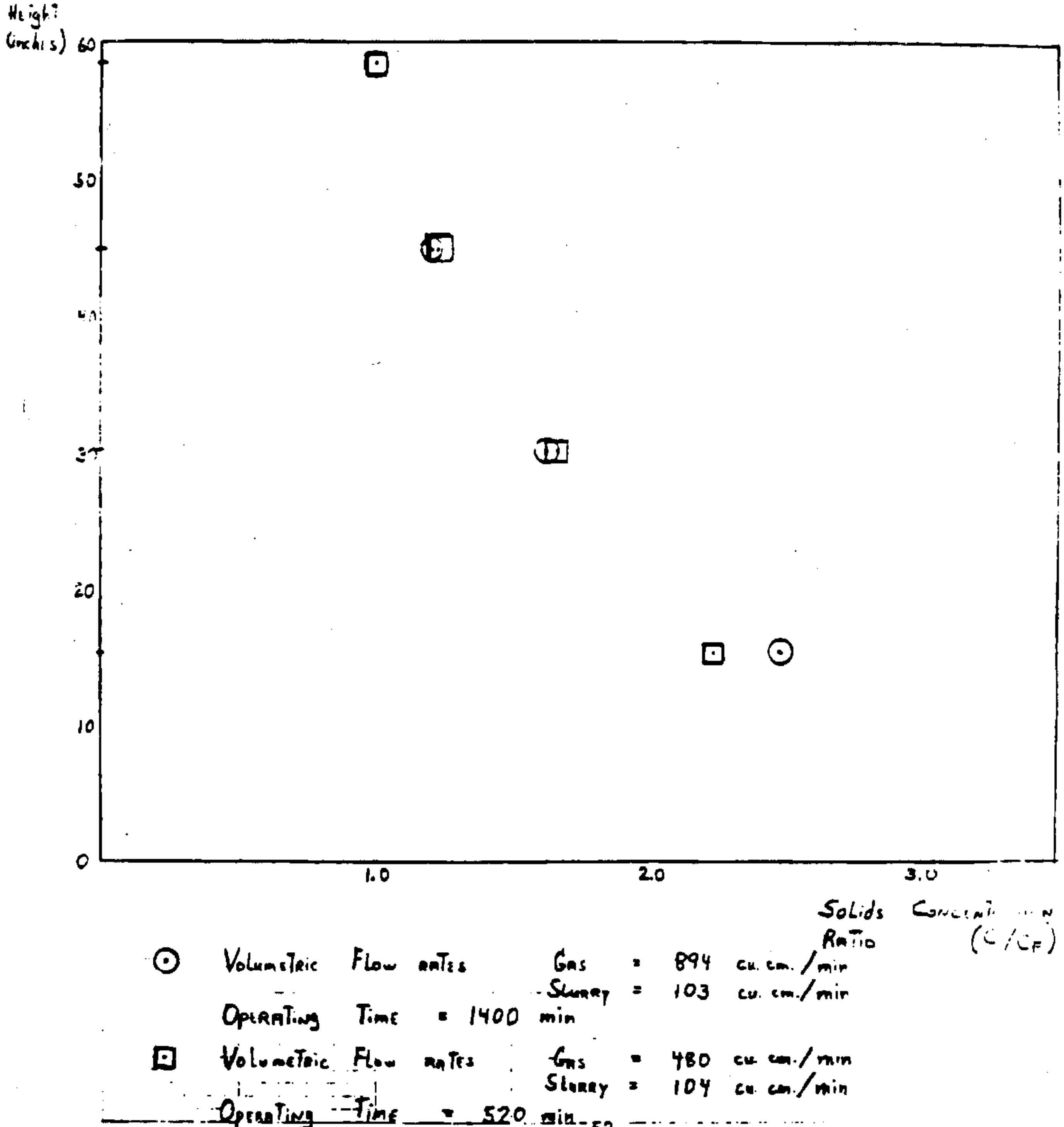
GAS FLOW RATE = 480 cu. cm./min.

FIGURE 26



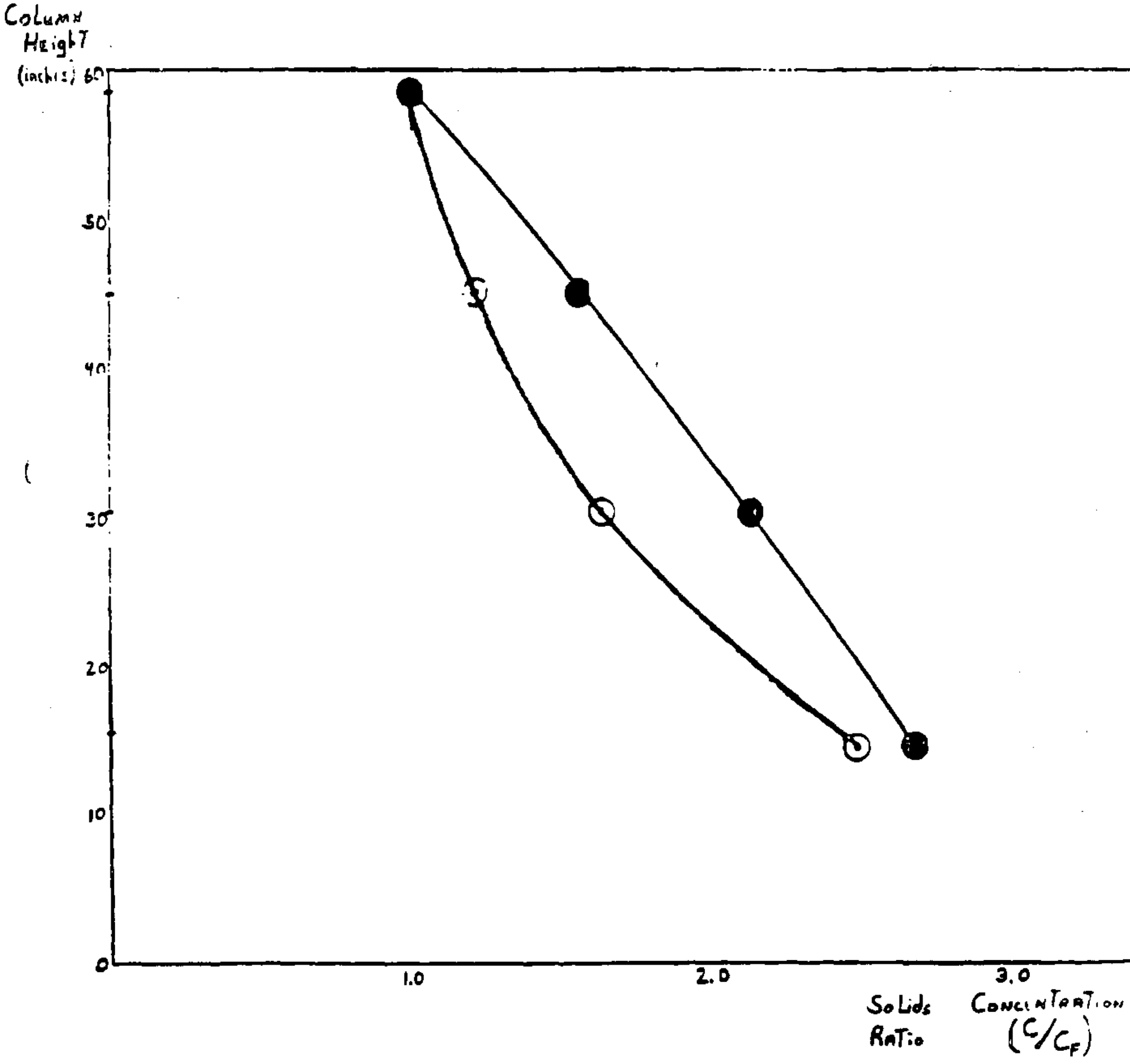
COLLECTION AT:
15.5 inches
30.2 inches
45.0 inches
55.5 inches
FEED
160
140
120
100
80
60
40
20
Microns
% ... T...

FIGURE 27



Height (inches)
60
50
40
30
20
10
0
1.0
2.0
3.0
Solids Concentration Ratio (C/CF)
⊙ Volumetric Flow rates Gas = 894 cu. cm./min
Slurry = 103 cu. cm./min
Operating Time = 1400 min
▣ Volumetric Flow rates Gas = 480 cu. cm./min
Slurry = 104 cu. cm./min
Operating Time = 520 min

FIGURE 28



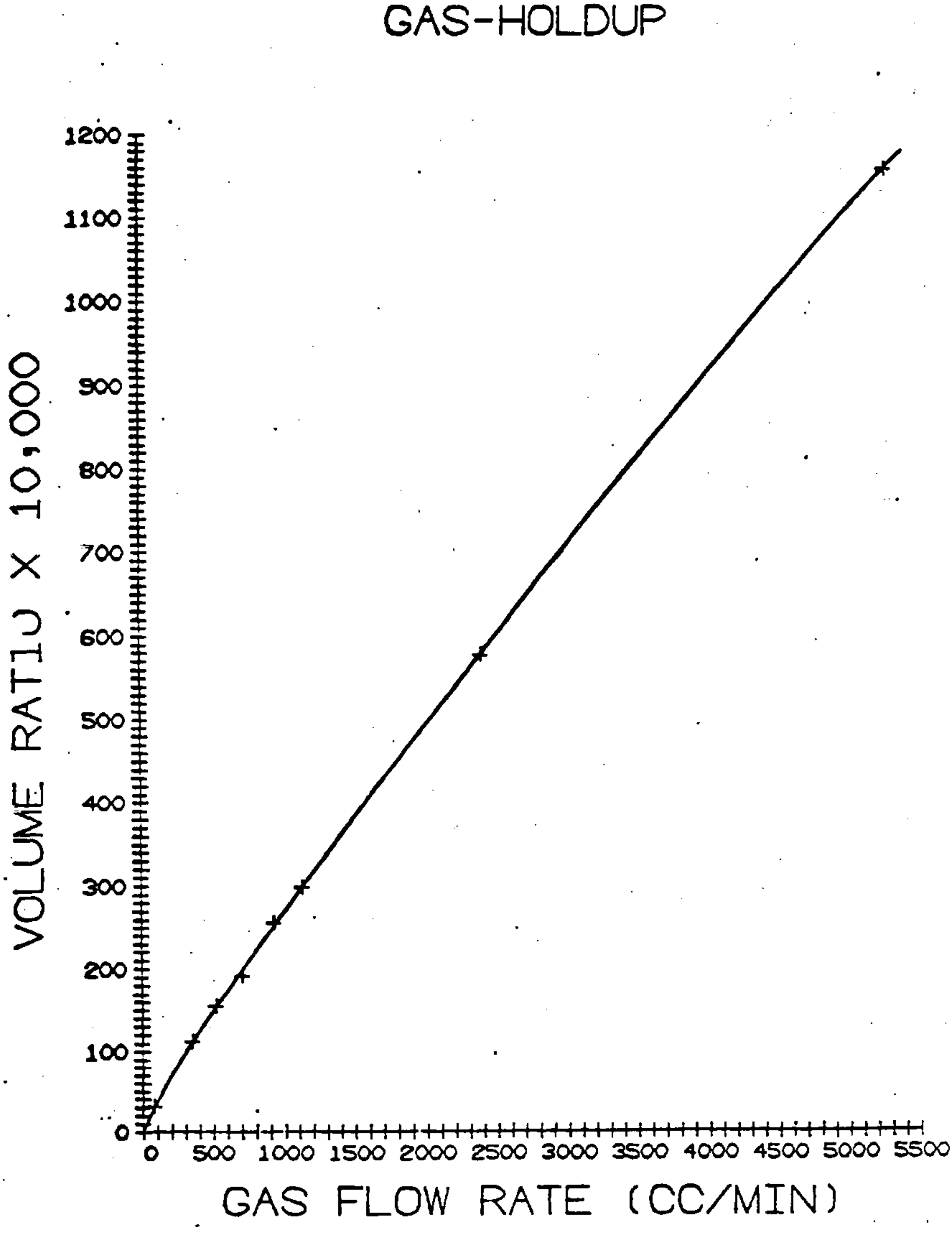
⊙ Experimental : Volumetric Flow rates Gas = 894 cu. cm./min

Slurry = 103 cu. cm/min

Dispersion number D/uL = 0.28

● Theoretical

FIGURE 29


GAS-HOLDUP
VOLUME RATIO X 10,000
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
0
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
5500
GAS FLOW RATE (CC/MIN)

```
FORTRAN IV REV M.20                  09-03-78  17:27 PAGE    1

          DIMENSION A(2,100),P(2,100),C(1000),CR(2,1000)
          READ(5,600) IRUN
    600   FORMAT (I2)
          DO 700 NRUN=1,IRUN
          READ(5,100) FLUID,GAS,HOLDUP,VOLUME,N
    100   FORMAT (4F10.5,I2)
          DO 150 I=1,N
          READ (5,200) A(1,I),A(2,I)
    200   FORMAT (2F10.5)
          A(2,I)=A(2,I)/17667.56996
    150   CONTINUE
          M=N-1
          DO 300 I=1,M
          J=I+1
          P(1,I)=(A(2,I)-A(2,J))/(A(1,I)-A(1,J))
          P(2,I)=A(2,I)-P(1,I)*A(1,I)
    300   CONTINUE
          L=0
          DO 400 I=1,M
          J=I+1
          DIF=A(1,J)-A(1,I)
          L=L+1
          IREP=IFIX(DIF*2.-1.)
          C(L)=A(2,I)
          T=A(1,I)
          IF(IREP.LT.1) GO TO 400
          DO 500 K=1,IREP
          T=T+.5
          L=L+1
          C(L)=T*P(1,I)+P(2,I)
    500   CONTINUE
    400   CONTINUE
          L=L+1
          C(L)=A(2,J)
          CALL AREAS(C,L,AREA)
          CALL TIMES(C,L,TIME)
          CONC=AREA/TIME
          EFFVOL=VOLUME*(1.-HOLDUP)
          RETIME=EFFVOL/FLUID
          THETA=TIME/RETIME
          CALL VARI (C,TIME,CONC,L,VARIAN,NRUN)
          CALL PEC(VARIAN,PECLET)
          CALL WRITE (TIME,CONC,PECLET,FLUID,GAS,HOLDUP,RETIME,THETA)
    700   CONTINUE
    60    CALL PLOT(1,0.,0.)
          END FILE 7
           END FILE 4
          STOP
          END
```

```
FORTRAN IV REV M.00                    09-03-78  17:27 PAGE   3

          SUBROUTINE AREAS(C,L,AREA)
          DIMENSION C(1000)
          ODD=0.
          EVEN=0.
          K=L-3
          DO 10 I=2,K,2
          EVEN=EVEN+C(I)
          ODD=ODD+C(I+1)
   10     CONTINUE
          H=.5
          AREA=(H/3.0)*(C(1)+4.0*(EVEN+C(L-1))+2.0*ODD+C(L))
          RETURN
          END
```

```
FORTRAN IV REV M.00                    09-03-78  17:27 PAGE   5

          SUBROUTINE TIMES(C,L,TIME)
          DIMENSION C(1000)
          TIM=0.
          T=0.
          CO=0.
          DO 20 I=1,L
          CO=CO+C(I)
          TIM=TIM+(T*C(I))
          T=T+.5
   20     CONTINUE
          TIME=TIM/CO
          RETURN
          END
```

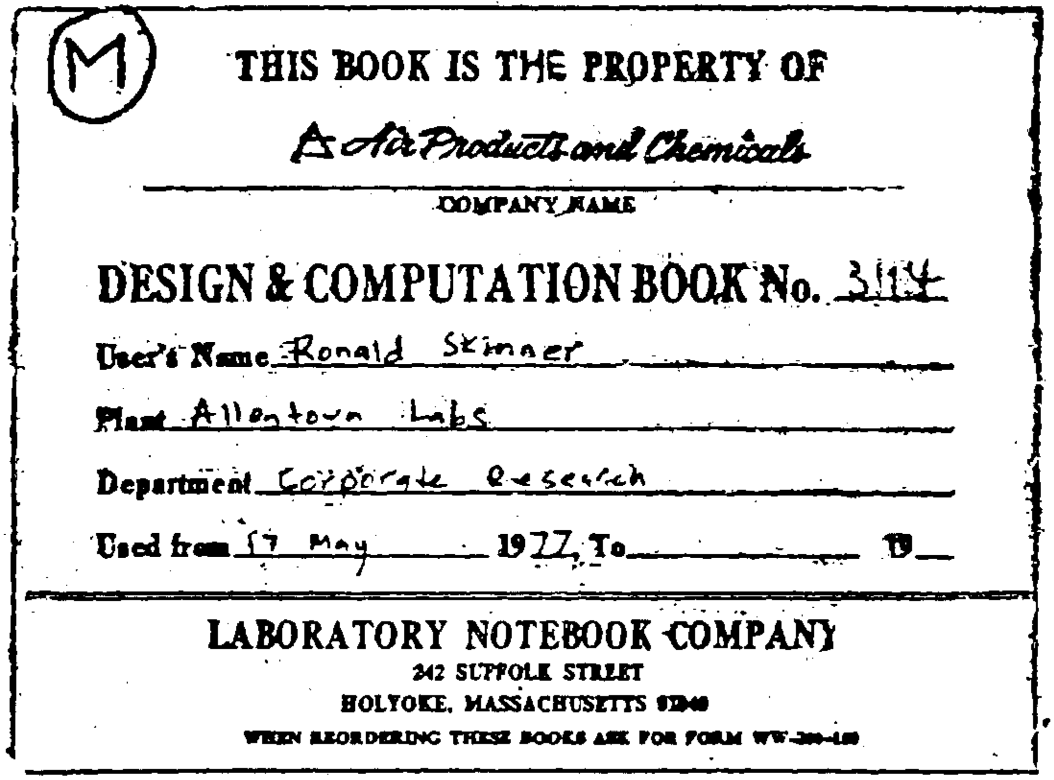
```
FORTRAN IV REV M.00                    09-03-78  17:28 PAGE   11

          SUBROUTINE WRITE (TIME,CONC,PECLET,FLUID,GAS,HOLDUP,RETIME,THET
          WRITE(4,113) FLUID,GAS,HOLDUP,RETIME,THETA
  113     FORMAT('0','SLURRY FLOW RATE (LITER/MIN) = ',F10.5,''/' GAS FL
         1ATE (LITER/MIN)      = ',F10.5,''/' GAS HOLDUP
         2,F10.5,''/' ACTUAL RESIDENCE TIME (MIN)  = ',F10.5,''/' THETA (
         3ENSIONLESS)          = ',F10.5,'')
          WRITE(4,101) TIME,CONC,PECLET
  101     FORMAT('0','CALCULATED MEAN RESIDENCE TIME (MIN)= ',F10.5,''/'
         1CER CONCENTRATION (GMOLES/LITER) = ',F10.5,''/' PECLET NUMBER (
         2ENSIONLESS)          = ',F10.5,'')
          RETURN
          END
```

```
FORTRAN IV REV M.00                     09-03-78  17:27 PAGE    7

              SUBROUTINE VARI (C,TIME,CONC,L,VARIAN,NRUN)
              DIMENSION C(1000),CR(2,1000)
              X=0.
              LA=0
              DO 20 I=1,L
              CR(1,I)=X/TIME
              IF(CR(1,I).GE.2.5) GO TO 50
              X=X+.5
              LA=LA+1
        20    CONTINUE
        50    X=0.
              DO 30 J=1,LA
              CR(1,J)=X/TIME
              CR(2,J)=C(J)/CONC
              X=X+.5
        30    CONTINUE
              CALL DRAW (CR,LA,NRUN)
              SUMA=0.
              SUMB=0.
              SUMC=0.
              DO 40 I=1,LA
              SUMA=SUMA+CR(2,I)
              SUMB=SUMB+CR(1,I)*CR(2,I)
              SUMC=SUMC+((CR(1,I)**2.)*CR(2,I))
        40    CONTINUE
              XMEAN=SUMB/SUMA
              VARIAN=(SUMC/SUMA)-(XMEAN**2.0)
              WRITE(4,11) XMEAN,VARIAN
        11    FORMAT('1','MEAN       = ',F10.5,'''/' VARIANCE = ',F10.5,''
              RETURN
              END
```

```
FORTRAN IV REV M.00                     09-03-78  17:28 PAGE    9

              SUBROUTINE PEC(VARIAN,PECLET)
              X=VARIAN/2.
        50    IF(X.LT..006) GO TO 60
              FX=(2.*X)-(2.*X*X)+(2.*X*X*EXP(-1./X))-VARIAN
              FDX=2.-(4.*X)+(4.*X*EXP(-1./X))+(2.*EXP(-1./X))
        80    XNEW=X-(FX/FDX)
              IF(ABS(X-XNEW).LT.1E-6) GO TO 70
              X=XNEW
              GO TO 50
        60    FX=2.*X-VARIAN-(2.*X*X)
              FDX=2.-(4.*X)
              GO TO 80
        70    PECLET=1./XNEW
              RETURN
              END
```

```
            SUBROUTINE DRAW (CR,LA,NRUN)
            INTEGER TIL,TILY,CHA,FMT
            DIMENSION  CR(2,1000)
            DIMENSION XP(4),YP(4),TIL(13),TILX(13),TILY(13),LEG(6,11)
            DIMENSION CHA(3),FMT(4),XF(6),YF(6),D(2)
            DATA XF/0.,0.,.3,0.,-.3,0./
            DATA YF/.5,.5,.5,0.,.5,1./
            DATA FMT/'( ','2 ','A2',' )'/
      1000  FORMAT (I2,13A2)
      1001  FORMAT (11A2)
      1002  FORMAT (I4)
      1003  FORMAT ("- ",11A2)
            IF(NRUN.GT.1) GO TO 70
      1     READ(5,1000)CHA(1),(TIL(I),I=1,13)
            IF(CHA(1).LT.0)STOP
            READ(5,1000)CHA(2),(TILX(I),I=1,13)
            READ(5,1000)CHA(3),(TILY(I),I=1,13)
            DO 10 I=1,6
      10    READ(5,1001)(LEG(I,J),J=1,11)
            WORD=.1
            PI=3.141593
            CALL READF (5,1,4,XP)
            CALL READF (5,1,4,YP)
            XS=5.5/(XP(1)-XP(2))
            YS=7./(YP(1)-YP(2))
            XINCH=1./XS
            YINCH=1./YS
            X0=-1.5*XINCH
            Y0=-2.*YINCH
            CALL SCALE (XS,YS,X0,Y0)
            N=(XP(1)-XP(2))/XP(3)
            CALL GRID (0,0.,0.,XP(3),N)
            J=XP(2)
            K=XP(1)
            L=XP(4)
            DO 20 I=J,K,L
            Y=-2.*WORD*YINCH
            F=3.*WORD*XINCH
            IF(I.GE.100)F=2.5*WORD*XINCH
            IF(I.GE.1000)F=2.*WORD*XINCH
            X=I-J-F
            CALL CHAR (X,Y,WORD,WORD,0.)
      20    WRITE(7,1002)I
            Y=-6.*WORD*YINCH
            X=CHA(2)
            X=2.75*XINCH-X*XINCH*WORD
            CALL CHAR (X,Y,2.*WORD,2.*WORD,0.)
            I=CHA(2)/2
            WRITE(7,FMT)(TILX(K),K=1,I)
            X1=0.
            Y=-9.*WORD*YINCH
            DO 40 I=1,6
            IF (LEG(I,1).EQ.'  ')GO TO 50
            IF (I.NE.4)GO TO 35
            X1=2.75*XINCH
            Y=-9.*WORD*YINCH
      35    X=X1
            CALL PLOT (-2,X+XF(I)*WORD*XINCH,Y+YF(I)*WORD*YINCH)
            CALL POINT(I-1)
            CALL PLOT ( 1,X+XF(I)*WORD*XINCH,Y+YF(I)*WORD*YINCH)
```

```
      X=X+WORD*XINCH
      CALL CHAR (X,Y,WORD,WORD,0.)
      WRITE(7,1003)(LEG(I,J),J=1,11)
40    Y=Y-2.*WORD*YINCH
50    CONTINUE
      N=(YP(1)-YP(2))/YP(3)
      CALL GRID (1,0.,0.,YP(3),N)
      J=YP(2)
      K=YP(1)
      L=YP(4)
      DO 30 I=J,K,L
      X=-5.*WORD*XINCH
      Y=I-J-(WORD/2.)*YINCH
      CALL CHAR (X,Y,WORD,WORD,0.)
30    WRITE(7,1002)I
      X=-7.*WORD*XINCH
      Y=CHA(3)
      Y=3.5*YINCH-Y*YINCH*WORD
      CALL CHAR (X,Y,2.*WORD,2.*WORD,PI/2.)
      I=CHA(3)/2
      WRITE(7,FMT)(TILY(K),K=1,I)
      I=CHA(1)/2
      Y=(YP(1)-YP(2))+2.*WORD*YINCH
      X=CHA(1)
      X=2.75*XINCH-X*WORD*XINCH
      CALL CHAR (X,Y,2.*WORD,2.*WORD,0.)
      WRITE(7,FMT)(TIL(K),K=1,I)
70    IPNT=NRUN
      DO 63 II=1,LA
      D(1)=AINT(CR(1,II)*1000.)
      D(2)=AINT(CR(2,II)*1000.)
      CALL PLOT (-2,D(1)-XP(2),D(2)-YP(2))
      CALL PLOT(2,D(1)-XP(2),D(2)-YP(2))
63    CONTINUE
      CALL PLOT( 1,D(1)-XP(2),D(2)-YP(2))
      DO 64 III=1,LA,10
      D(1)=AINT(CR(1,III)*1000.)
      D(2)=AINT(CR(2,III)*1000.)
      CALL PLOT(-2,D(1)-XP(2),D(2)-YP(2))
      CALL POINT (IPNT-1)
      CALL PLOT( 1,D(1)-XP(2),D(2)-YP(2))
64    CONTINUE
      RETURN
      END
```



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5 OCT, 77 XCL7

RECIPE FOR SLURRY: 243.5 LBS. DISTILLED HYDROTREATED CREOSOTE
40 LBS. SUNTECH HYDROTREATED CREOSOTE (BBL#1)
16.5 LBS SUNTECH UNTREATED CREOSOTE (BBL#1)
165.0 ~~210~~ LBS Indiana ~~KENTUCKY~~ COAL SIEVED THRU 180 MESH SCREEN

2130 HRS 5 OCT 77
Charge Tank #2 - Charging Complete and circulator pump in operation

2200 HRS Charge Tank #3 contains 25 gal Tetralin and the stirrer is on.

2330 ON SHIFT - RD. SCHMALDINST - OPERATOR + DR SCHIENEBERGER

6 OCT

0400 ATTEMPT TO START B-L PUMP - COULD NOT PRIME THROUGH V-27. STARTED B-L PUMP ~~IN EFFORT~~ WITH V-23 & V-27 OPEN, V-25 CLOSED IN EFFORT TO GET FLOW THROUGH CHECK VALVES. PUMP PRESSURE SWITCH DID NOT FUNCTION AND PRESSURE BUILT UP TO 4000 PSI BEFORE IT WAS NOTICED. IN THE FEW SECONDS IT TOOK TO SHUT OFF PUMP THE PRESSURE BUILT UP TO + 7000 PSI AND PEGGED PUMP PRESSURE GUAGE. OPENED PNEUMATIC VALVE AND SLOWLY OPENED V-25. B-L PUMP GUAGE SHOULD READ 2000 PSI BUT NOW READS 3000. READJUSTED SETTING OF PRESSURE SWITCH SO THAT PUMP JUST TURNS OFF AT SLIGHTLY ABOVE SYSTEM PRESSURE. PRESSURE SWITCH IS FUNCTIONING IT WAS JUST OUT OF ADJUSTMENT. DETERMINED THAT V-27 IS OPEN AS THERE IS N_2 FLOW ABLE TO GET THROUGH. V-23 MUST BE BLOCKED AS THERE IS CREOSOTE GOING TO THE SYSTEM.

0500 WITH BRAN-LUEBBE PUMP PRESSURE GUAGE READING INCORRECTLY BUT PRESSURE SWITCH FUNCTIONING AND V-23 BLOCKED START PUMPING CREOSOTE. BEGIN PUMP STROKE AT 30 MM; REDUCE TO 20 MM; REDUCE TO 15 MM - PUMP RATE ACCORDING TO COMPUTER PRINT OUT ≅ 2800 gm/hr
MOYNO RECYCLE PUMP PRESSURE IS >100 PSI CANNOT REDUCE EVEN WITH THROTTLING VALVE WIDE OPEN, BY PASS VALVE WIDE OPEN AND EVEN BY PASSING FLOW THROUGH MILROY PUMP SIDE.

6 OCT 1977 XCL-7

0515 ANNIN VALVE DOES NOT CONTROL - MUST DUMP MANUALLY
0530 TURN ON COOLING WATER, S.P.R STIRRER ON AT 1500 RPM
STIRRED PATH REACTOR TEMP ON - SET TO 850° F, AUX HTR
ON AT 60%
G/L SEP HEATER ON @ 350°
MAIN REC. SET TO 325°
MOBIL-THERM HEAT EX #2A @ 350°, PUMP #2 ON
TRANSFER LINE HEATERS #3, 6, 10 & 13 SET TO 325°
TRANSFER LINE HEATERS #7 & 8 SET TO 350°
0715 MOYNO RECYCLE PUMP PRESSURE NOW DOWN AND ABLE TO
BE CONTROLLED BY THROTTLING VALVE

0730 ON SHIFT: S. CAPIOTIS

0915 ANNIN VALVE STILL NOT WORKING; WILL STOP PUMPING AND
TRY TO CLEAN LINES

0200 REPAIRED BROKEN VALVE PIN IN PUMP PRIME LINE, CLEANED
LINES LEADING TO & GOING FROM ANNIN VALVE

0200 START PUMPING

0215 PUMP PRESSURE >5,000 PSIG

1530 On Shift C Keyhart

17 OCT., 77 XCL 7

0730 ON SHIFT; S. CAPIOTIS
0730 FOUND COKE IN REACTOR + REACTOR INLET + EXIT LINES ON 5 OCT., 77; SHUTDOWN UNTILL TODAY TO CLEAN REACTOR + ALL CLOGGED LINES; ALSO REPLACED FAULTY PRESSURE SHUTOFF SWITCH AND INSTALLED A RUPTURE DISC ON THE BRAN + LUBBE PUMP

1000 ADDED 20 GAL ALLIED 24 CB CREOSOTE OIL TO CHARGE TANK #1

1400 ALLOWED N2 TO PURGE FROM BOTTOM OF TANK #2 TO MIX SETTLED COAL IN TANK. SLURRY IS THE SAME THAT WAS CHARGED 5 OCT., 77 (SEE BBP 3114-51) RECYCLE PUMP ON.

1445 SOLONOID ON B+L LINE LEAKS
1500 SOLONOID WORKING PROPERLY

1520 PUMPING CREOSOTE
1530 Bob Paul & C Mayhart on shift

1700 Slurry Feed Tank would not fill. Solenoid valve was suspect and was removed from slurry system and a straight through line installed. Tank filled OK using manual valve only.

2300 ON SHIFT - R. SCHMALDINST - D. SCHOENEBERGER
2340 TURN ON SYSTEM HEATERS
SP REACTOR - ON, SET POINT 850°F, AUX. HTR @ 60%
G/L SEP - 350°F
MAIN REC. 325°F
MOBIL THERM 2A - 350°F PUMP ON
LINE HEATERS #3, #6, #10, #13 - 325°F
LINE HEATERS #7, #8 - 350°F
COOLING WATER ON - SP REACTOR, HP, LP COND., #2 MOBILTHERM
0001 SUSPECT INLET FLOMETER INDICATION IS READING LOW AS FLOW ON INLET ROTAMETER IS VERY HIGH (MIDDLE BALL AT 60) AND FLOW THROUGH HP DTM SEEMS MUCH HIGHER THAN NORMAL AND WORKING PRESSURE OF DTM IS 6 PSI RESET FLOW THROUGH INLET ROTAMETER SO THAT GLASS BALL IS AT 58
0015 ADJUST ANNIN VALVE (CLOSE DOWN ON SEAT SLIGHTLY)

18 OCT 1977 XCL-7

0200 ANNIN VALVE IS CONTROLLING - SOMEWHAT OVERCOMPENSATING BUT CONTROLLING. REACTOR TEMP AT 700° REDUCE AUX. HEATER TO 21%

0400 FILL SLURRY FEED TANK - THIS MUST BE DONE MANUALLY AS PNEUMATIC VALVE HAS BEEN REMOVED
INCREASE PUMP STROKE TO 12 MM

0730 ON SHIFT: S. CAPIOTIS

0730 DUMP MAIN REC., FILL FEED TANK WITH CREOSOTE MANUALLY

0900 INSTALLED BALL VALVE AT TOP OF SLURRY FEED TANK, REPLACING SOLENOID VALVE

0945 REACTOR TEMPS. DECREASED WHEN STIRRER WAS TURNED OFF.
PUMP STROKE INCREASED TO 30 mm

1000 FILL FEED TANK WITH SLURRY

1015 PUMPING AT 35 MM

1100 DUMP MAIN REC., FILL FEED TANK WITH SLURRY
HPDTM = 164.84 @ 77°F, 4.5 PSIG
GLASS ROTAMETER BALL: 61.0 DIGITAL MASS FLOW METER - 277

1200 XCL7-1 DUMP REC'S; FILL WITH SLURRY
MAIN ~~PROD.~~ REC WT. — 5,093 gms
COND REC WT. — 68 gms
COND H_2O — 8 gms
COND. LT. ORGANIC — 60 gms
HPDTM: 180.00 @ 77°F + 2.5 PSIG
GLASS ROTAMETER BALL — 61.0 MASS FLOW METER — 280

1230 COMPLETED CHECK LIST, EVERYTHING OK

1300 XCL7-1 DUMP REC'S; FILL WITH SLURRY
MAIN REC. WT. — 4,430 gms
COND REC WT. — 158 gms
COND. H_2O — 95 gms
COND LT. ORGANICS — 63 gms
HPDTM: 196.57 @ 77°F + 3.0 PSIG
GLASS ROTAMETER BALL — 61.0 DIGITAL FLOMETER — 275

1330 SYSTEM RUNNING WELL

18 OCT, 77 XCL7

1400 XCL7-3
MAIN PROD WT — 4963 gms
COND REC. WT — 135 gms
H_2O - 95 gms
Lt. INORGANICS - 40 gms
HPDTM - 216.22 @ 77°F + 3.0 PSIG
GLASS BALL = 61 FLOWMETER - 277
1445 INCREASE PUMP STROKE TO 26 MM
1500 XCL7-4
MAIN PROD WT — 4614 gms
COND REC WT — 157 gms
H_2O — 104 gms
Lt. ORG. — 53 gms
H.P.D.T.M = 233.94 @ 77°F + 3.5 PSIG
GLASS BALL - 61.0 FLOWMETER - 274

1530 C Keyhart & R. Paul onshift

1600 XCL7-5
Main Product wT — 5583 Gms
Cond Rec wT — 187.46 Gms
H_2O — 116.6 Gms
Lt org — 70.86 Gms
HPDTM = 253.18 @ 75°F & 15 PSIG
Glass Ball - 61.0 Flowmeter - 277

1645 Pump Rate increased to 28
1650 XCL7-6-RE-NP Sample Taken

1700 XCL7-6
Main Product wt — 4360 Gms
Cond Rec wt — 168.7 Gms
H_2O wt — 101.7
Lt org wt — 67.0
HPDTM = [illegible] @ [illegible] PSIG
Glass Ball - 61 Flowmeter - 274

1720 Pump Rate increased to 30.[illegible]

18 Oct 77 XCL-7

1800 XCL7-7

Main Product wt ———— 5668 Gms

Cond. Rec. wt ———— 198 Gms

H_2O wt ———— 122.9 Gms

Lt. org. wt ———— 75.1 Gms

HPDPM 280.02 @ 2.5 psig and 76°F

Glass Ball 61 Flowmeter : 276

1900 XCL7-8

Main Product wt ———— 5505 Gms.

Cond. Rec. wt ———— 205.7 Gms.

H_2O wt ———— 115.3 Gms

Lt. org. wt ———— 90.4 Gms

HPDPM = 294.40 @ 1.3 psig @ 76°F

Glass Ball = 61 Flowmeter = 270

2000 XCL7-9

Main Product wt ———— 5388 Gms.

Cond. Rec. wt ———— 195.3 Gms

H_2O wt ———— 107.3 Gms

Lt. org. wt ———— 88.0 Gms

HPDPM = 307.82 @ 74°F @ 2.5 psig

Glass Ball = 62 Flowmeter = 287

2017 Loss in System press. Pressure in L.P. Cond. builds up and backs off. H.P. Cond will not build up pressure.
Raised Separator set pt to 50. Separator apparently not completely closing inspite of the fact that it indicates that it is.

2100 XCL7-10

Main Product wt. ———— 4468 Gms

Cond. Rec. wt. ———— 259.2 Gms

H_2O wt ———— 4.7 Gms

Lt. org. wt ———— 254.5 Gms (Looks emulsified)

HPDPM = 311.79 @ 3.5 psig @ 76°F

Glass Ball = 57 Flowmeter = 253

2130 Dumping Separator by hand every few minutes.

10-18-77 XCL7

2200 XCL7-11-PL
Main Product wt. —— 5708 Gms
Cond. Rec. wt. —— 200.3 Gms
H_2O wt —— 535 Gms
Lt. org wt —— 146.8 Gms
HPDTM = 326.78 @ 1.5 psig 76°F
Glass Ball = 61 Flowmeter = 252

2245 XCL7-12-PG-HP @ 45 PSI

2300 XCL7-12-PL
Main Product wT —— 5018 Gms
Cond. Rec. wt. —— 191.4 Gms
H_2O wt —— 55.8 Gms
Lt org. wt. —— 135.6 Gms
HPDTM = 341.12 @ 3.3 psig 76°F
Glass Ball = 61 Flowmeter = 277

2330 ON SHIFT: R. SCHMALDINST ; D. SCHOENEBERGER

2400 XCL7-13

XCL7-13-PL MAIN PRODUCT WT. = 5184 g
TOTAL ~~ORGANIC~~ COND. REC. WT = ~~84g~~ 187.8 g
XCL7-13-PW WATER WT = 75.6 g
LT ORG WT = 112.2 g
H.P. DTM = 358.41 SCF @ 3.0 PSI WP & 76°F
GLASS BALL = 61 FLOWMETER DIGITAL = 277
FEED SAMPLE - XCL7-13-FL

19 OCT 77
0100 XCL7-14

XCL7-14-PL MAIN PRODUCT WT = 4244 g
TOTAL CONDENSOR REC. WT = 201.2 g
XCL7-14-PW H_2O WT = 100.8 g
LT ORG. WT = 100.4 g
HP DTM = 374.15 SCF @ 2.9 PSI W.P. & 76°F
GLASS BALL = 61 FLOWMETER = 278
SYSTEM PRESS = 1554 REAC TEMP 838 °F
SWITCH TO BACK UP COMPUTER TERMINAL
XCL7-14-FL FEED SAMPLE

19 OCT 1977 XCL - 7

0200 XCL 7 - 15
FEED SAMPLE - XCL7-15FL
MAIN PRODUCT WT = 5740g
TOTAL COND. REC WT = 249.6g
H_2O WT = ~~221.6gm~~ 104.3 g
LT. ORGANIC WT = 145.3g 145.3 g
H.P. DTM = 386.23 SCF @ 2.0 PSI (NO FLOW THROUGH) & 76°F
GLASS BALL = 61 FLOWMETER = 281
0240 BUMP H_2 MANIFOLD CYLINDERS - 1400 PSI
0255 ANNIN VALVE IS CONTROLLING QUITE WELL AFTER SEVERAL HOURS OF ADJUSTING
0300 XCL7 - 16
FEED SAMPLE XCL 7-16FL
MAIN REC PRODUCT WT = 4922 g
COND. REC TOTAL WT = ~~104.4gm~~ 167.4g
H_2O WT = 60.5
LT. ORGANIC WT = 106.9 g
H.P. DTM = 400.25 SCF @ 2.0 PSI U.P. & 75°F
GLASS BALL - 61 FLOWMETER 277
0400 XCL7- 17
FEED SAMPLE XCL 7 - 17 - FL
MAIN REC. PRODUCT WT. = 5071g
COND. REC'S TOTAL WT = 214.6g
H_2O WT = 129.1g
LT. ORGANIC WT = 85.5
H.P. DTM = 415.15 SCF @ 1.5 PSI & 75°F
GLASS BALL - 61 FLOWMETER - 274
0500 XCL7 - 18
FEED SAMPLE - XCL7 - 18 FL
MAIN REC PRODUCT WT. 5058 g
CON. REC'S TOTAL WT. 202.1 g
H_2O WT. 130.2 g
LT. ORGANIC WT. 71.9 g
H.P. DTM = 430.60 @ 2.6 PSI & 75°F
GLASS BALL - 61 FLOW METER - 277
PROCESS GAS SAMPLE = XCL7 - 18 - PG - HP

19 OCT 1977 XCL-7

0600 XCL 7 - 19
FEED SAMPLE - XCL 7-19-FL
MAIN REC. PRODUCT WT = 4943 g
COND. REC. TOTAL WT = 193.4 g
H_2O WT = 119.4 g
LT. ORGANIC WT = 74.0 g
HP DTM - 446.05 @ 3.0 PSI & 75 °F
GLASSBALL - 61 FLOWMETER = 271

0610 DRAIN LP DEMISTER - NO LIQUID
DRAIN HP DEMISTER - 32 g LIQUID

0700 XCL 7 - 20
FEED SAMPLE - XCL 7 - 20 FL
MAIN REC. PRODUCT WT = 5492 g
COND REC. TOTAL WT = 210.3 g
H_2O WT = 84.7
LT. ORGANIC WT = 125.6 g
HP DTM = 459.75 @ 2.6 PSI & 75 °F
GLASS BALL - 61 FLOWMETER = 274

0730 ON SHIFT : S. CAPIOTIS

0800 XCL 7-21
MAIN REC WT - 5,378 gms
COND. REC WT. - 197 gms
LT. ORGANICS - 120 gms
H_2O - 77 gms
H.P.D.T.M. - 476.14 @ 75° + 2.5 PSIG
GLASS BALL - 60.5 FLOWMETER - 270

0900 XCL 7-22
MAIN REC WT - 6,403 gms
COND WT - 227 gms LT ORG - 116 H_2O - 111 gms
GLASS BALL - 61.0 FLOWMETER - 276
HPDTM - 487.31 @ 75° + 1.5 PSIG

0900 Start sampling behind barricade Sample AG-2
Flow = 2.15 Pump # 237
0955 Start personal monitor - Stratton Capiotis AG-3 Pump #236 Flow = 2.1

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0945 ANNIN VALVE OVER-PUMPING, RAISED SETPOINT TO 50

1000 XCL7-23
MAIN REC WT - 5,612 gms
COND REC WT - 247 gms
LT. ORG. - 178 gms
H_2O - 69 gms
H.P.D.T.M. - 494.69 @ 76° + 30 PSIG
GLASS BALL - 61.0 FLOWMETER ~~ROTAMETER~~ - 269

1010 LOSING SYSTEM PRESSURE THRU ANNIN VALVE, CLOSED MANUAL VALVE ABOVE ANNIN VALVE

1000 CUTTING PUMP STROKE - WANT 3000 gms/HR

1100 XCL7-24
MAIN REC WT - 3,074
COND REC WT - 181 gms
LT. ORG - 151 gms
H_2O - 30 gms
H.P.D.T.M. - 498.20 @ 76°F + 3.0 PSIG
GLASS BALL - 61.0 FLOWMETER ~~ROTAMETER~~ - 273

1120 BUMPED UP N_2 BANK; ANNIN VALVE NOW CONTROLLING PROPERLY

1200 XCL7-25
MAIN REC WT - 3,013 gms
COND REC WT - 198 gms
LT. ORG. - 141 gms
H_2O - 57 gms
H.P.D.T.M. - 513.27 @ 76°F + 3.0 PSI
GLASS BALL - 61.0 FLOWMETER - 269

1230 COMPLETED CHECK LIST: SYSTEM OK

1300 XCL7-26
MAIN REC WT - 2,831 gms
COND REC WT - 95 gms
LT. ORGANICS - 46 gms
H_2O - 47 gms
H.P.D.T.M. = 52[illegible].78 @ 76° + 2.5 PSIG
GLASS BALL = 61.0 FLOWMETER = 271

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1400 XCL7- 27
MAIN REC. WT. ——— 2991 gms
COND REC. WT. ——— 140 gms
LT. ORG. ——— 57 gms
H_2O ——— 83 gms
H.P.D.T.M.: 539.56 @ 76°F & 2.5 PSIG
GLASS BALL: 61.0 FLOWMETER: 269

1500 XCL7-28
MAIN REC WT ——— 3083 gms
COND. REC. WT. ——— 128 gms
LT. ORG. ——— 43 gms
H_2O ——— 85 gms
HPDTM: 555.06 @ 76° & 2.5 PSIG
GLASS BALL: 60.5 FLOWMETER: 263

1510 STOP SAMPLING PUMPS ON S. CAPKOTIS
1512 " " " BEHIND BARRACADE

1530 C Kayhart & B. Paul on shift

1600 XCL7-29- PG -HP Sample taken @ 45psi
XCL7-29
Main product wT ——— 3180 Gms.
Cond Rec. wT ——— 145.6 Gms
H_2O wT ——— 98.1 Gms
LT org wT ——— 47.5 Gms.
HPDTM: 565.71 @ 76°F & 1.0 psig
Glass Ball 60.0 Flowmeter: 267

1700 XCL7-30
Main Product wT ——— 2783 Gms
Cond Rec wT ——— 145.7 Gms
H_2O wT ——— 91.5 Gms
LT org. wT ——— 54.2 Gms
HPDTM: 578.59 @ 1.0 psig and 75°F
Glass Ball: 59.5 Flowmeter: 265

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1737 Reduced pump Rate to 13

1750 SP. Reactor Aux. Heater Turned off

1900 XCL 7-31

Main product wt — 4644 Gms

Cond Rec. wt — 219.8 Gms

H_2O wt — 83.1 Gms

Lt org. wt — 136.7 Gms

HPDTM: 601.40 @ 1.0 psig and 76°F

Glass Ball: 58.0 Flowmeter 261

2100 XCL 7-32

Main Product wt. — 4453 Gms.

Cond. Rec. wt — 197.2 Gms

H_2O wt. — 67.2 Gms

Lt. org. wt. — 130.0 Gms

HPDTM = 619.49 @ 2.2 psig and 76°F

Glass Ball: 65.0 Flowmeter 271

2300

XCL 7-33

Main Product wt. — 3699 Gms

Cond. Rec wt. — 171.4 Gms

H_2O wt. — 85.8 Gms

Lt. org wt. — 85.6 Gms

HPDTM = 640.53 @ 2.2 psig & 76°F

Glass Ball: 60.0 Flowmeter: 270

2330 ON SHIFT - R. SCHMALDINST & D. SCHCENEBERGER

20 OCT

0015 COMPUTER TERMINAL FIXED PUMP RATE 2400 G/HR

LOWER PUMP SETTING TO 12 mm

0030 GAS SAMPLE XCL7 - 34-PG-HP

0050 PUMP RATE 2200 gm/hr REDUCE PUMP TO 11 mm

0100 XCL 7 - 34

FEED SAMPLE XCL 7-34 FL

MAIN REC PRODUCT WT = 4449 g

TOTAL COND REC. WT = 183.4 g

H_2O WT = 123.9 g

LT ORGANICS WT = 54.5 g

HP DTM = 666.58 @ 2.4 psi @ 74 °F

GLASS BALL 58 FLOWMETER 268

26 OCT 1977 XCL-7

0300 XCL 7 - 35
FEED SAMPLE XCL 7·35 FL
MAIN REC PRODUCT WT = 3578 g
COND REC.'S TOTAL WT = 159.2 g
H_2O WT = ~~159.2 g~~ ERS 97.7 g
LT. ORGANICS WT = 61.5 g
HP DTM = 692.85 @ 2.2 PSI & 75 °F
GLASS BALL = 58 FLOWMETER = 263
LIQUID (CREOSOTE ?) BEING PUSHED THROUGH VENT STACK
0415 NIT SOURCE N_2 FROM COMING BE TO SEEMS · ROTAMETER
DOWN FROM VENT STACK.

0500 XCL 7 - 36
FEED SAMPLE XCL 7 - 36 FL
MAIN REC PRODUCT WT = 3387 g
COND REC.S TOTAL WT = 157.8 g
H_2O WT = 103.2 g
LT. ORGANIC WT = 54.6 g
H.P. DTM = 718.20 @ 8.4 PSI & 75 °F
GLASS BALL = 57 FLOWMETER = 261
0510 DRAIN APPROXIMATELY 14gms FROM H.P CONDEN. DEMISTER. 0 FROM LP DEMISTER

0700 XCL7 - 37
FEED SAMPLE XCL7 - 37FL
MAIN REC PRODUCT WT = 3555 g
COND RECS TOTAL WT = 165.8 g
H_2O WT. = 101.1 g
LT ORGANICS WT. = 64.7 g
H.P. DTM = 745.80 @ 2.4 PSI & 75 °F
GLASS BALL = 60 FLOWMETER = 270

0730 ON SHIFT: S. CAPIOTIS
0830 COMPLETED CHECKLIST, EVERYTHING OK

XCL7-38-PG
30 PSIG

0900 XCL7-38
MAIN REC'D WT — 3,891 gms
COND. REC. WT. — 154 gms
H_2O — 105 gms
LT. ORG. — 49 gms
HPDTM = 773.80 @ 75°F & 2.0 PSIG
GLASS BALL - 60.0 FLOWMETER - 273
0910 CHARGE TANK #2 NOW EMPTY, STARTING RECYCLE LOOP ON TANK #3

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1030 SYSTEM OK

1200 XCL7-39
MAIN REC. WT —— 4,586 gms
COND. REC. WT. —— 231 gms
H_2O —— 154 gms
LT. ORG. —— 77 gms
HPDTM = 804.32 @ 76°F + 2.0 PSIG
GLASS BALL - 60.0 FLOWMETER - 267

1200 FILLING FEED TANK FROM CHARGE TANK #3

1400 XCL7-40
MAIN REC WT. —— 3,956 gms
COND REC WT. —— 164 gms
H_2O —— 93 gms
LT. ORG. —— 71 gms
HPDTM = 822.11 @ 76° + 2.0 PSIG
GLASS BALL = 60.5 FLOWMETER - 270

1400 ALL ~~[illegible]~~S MAIN REC. PRODUCTS ARE NOW BEING PUT INTO A DRUM MARKED "XCL7 PRODUCT", SPECIAL INSTRUCTIONS WILL BE GIVEN CONCERNING ANY SAMPLES TO BE SAVED SEPERATLY

1530 C Keyhart & Del Rel on shift

1600 XCL7-41
Main Product wt —— 3476 gms
Cond. Rec wt —— 146.4 gms
H_2O wt —— 88.0 gms
Lt org wt —— 58.4 gms
HPDTM - 844.33 @ 1.3 psig and 76°F
Glass Ball: 54.0 Flowmeter [illegible]

1630 XCL7-41- PG HP 545 psi

1700 System ok.

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1750 XCL7-42-PC
Main product wt. ——— 2059 Gms
Cond. Rec. wt. ——— 126.4 Gms
H_2O wt. ——— 82.8 Gms
Lt. org. wt. ——— 43.6 Gms
HPDTM = 860.85 @ 1.2 psig and 76°F
Glass Ball 53.0 Flowmeter 263

1740 Raised pump rate to 12.5
[illegible] Raised pump rate to 15.0
1820 SPR auxiliary heater on at 90%
1900 Raised pump rate to 17.0
1950 XCL7-43
Main Product wt. ——— 3283 Gms
Cond. Rec. wt. ——— 159.1 Gms
H_2O wt. ——— 109.3 Gms
Lt. org. wt. ——— 49.8 Gms
HPDTM = 818.65 @ 1.2 psig and 76°F
Glass Ball 59.0 Flowmeter 251
2000 Raised Pump Rate to 20
2000 XCL7-44
Main Product wt. ——— 2530 Gms
Cond. Rec. wt. ——— 121.8 Gms
H_2O wt. ——— 75.2 Gms
Lt. org. wt. ——— 46.6 Gms
HPDTM = 890.85 @ 1.5 psig 76°F
Glass Ball = 57.0 Flowmeter = 261
2055 Feed Rate 3100 Gms/Hour
2100 XCL7-45 ——— Trouble with draining valve
Main Product wt. ——— 3619 Gms Lost system pressure,
Cond. Rec. wt. ——— 44.4 Gms Trouble with pressure
H_2O wt. ——— 35.8 Gms system - 25 minutes to
Lt. org. wt. ——— 8.6 Gms Fill Feed tank
HPDTM = 902.66 @ 10 psig 76°F
Glass Ball: 54.0 Flowmeter = 245
~~[illegible] Feed Rate [illegible]~~
[illegible]
2130 Raised Pump Rate to 23

20 OCT 77 XCL7

2200 XCL7-46

Main Product wt ———— 4748 Gms

Cond. Rec. wt ———— 2834 ml } Nothing coming from LP Cond.

H2O wt ———— 234 Gms } Appears to be clogged.

Lt. org. wt ———— 4.9 Gms

HPDTM = 910.52 @ 3.2 psig 76°F

Glass Ball: 57. Flowmeter = 259

2225 Reduced Pump Rate to 215

2300 XCL7-47

Main Product wt ———— 4597 Gms

Cond. Rec. wt ———— 47.7 Gms

H2O wt ———— 44.2 Gms

Lt. org wt ———— 3.5 Gms

HPDTM = 922.97 @ 2.5 psig 76°F

Glass Ball = 56.5 Flowmeter = 255

2300 Drain valve inoperative, Dumping by hand — Switch over to

2325 Creosote. — GAS SAMPLE XCL7-48 PG

2330 ON SHIFT R.D. SCHMALDINST & D.R SCHOENEBERGER SWITCH TO CREOSOTE FROM NO. 1 FEED TANK AFTER G/L SEPARATOR OVERFILLED AND LP CONDENSOR RECEIVER BECAME PLUGGED. V23 IS BLOCKED.

0730 ON SHIFT. S. CAPIOTIS

0800 ABLE TO DRAIN L.P. COND. REC.

0840 TURNING OFF REACTOR HEATER + ALL OTHER HEATERS

08–