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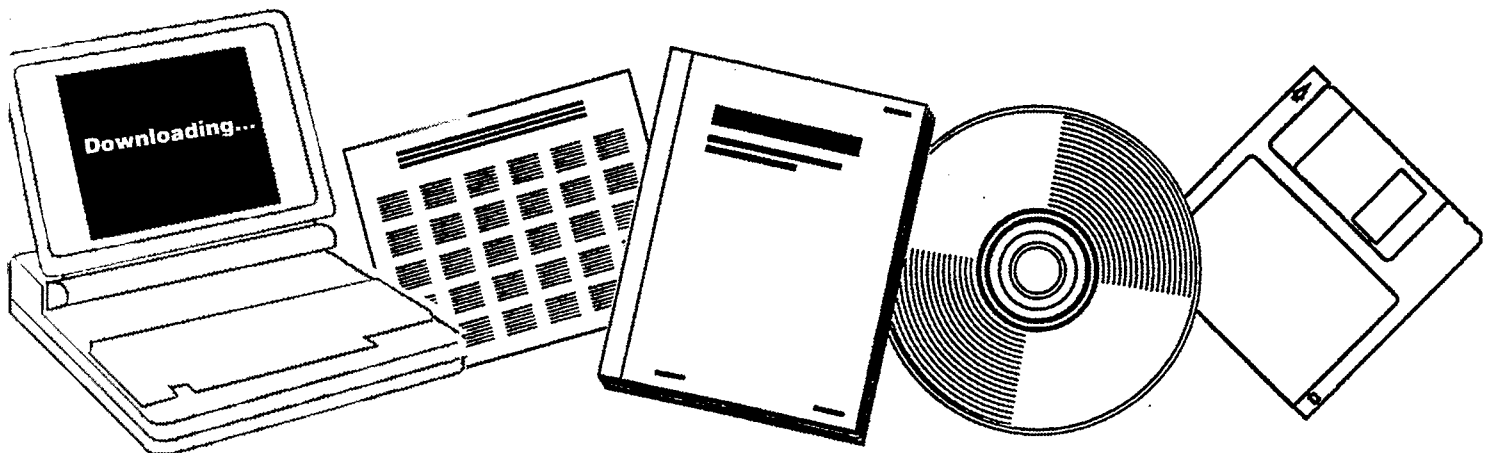
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**GAS/SLURRY FLOW IN COAL-LIQUEFACTION  
PROCESSES (FLUID DYNAMICS IN 3-PHASE-FLOW  
COLUMN). QUARTERLY TECHNICAL PROGRESS  
REPORT, 1 APRIL 1981-30 JUNE 1981**

**AIR PRODUCTS AND CHEMICALS, INC.  
ALLENTOWN, PA**

**NOV 1981**



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(Fluid Dynamics in 3-Phase Flow Column)

Quarterly Technical Progress Report  
For Period 1 April 1981 - 30 June 1981

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## Summary

This is the Quarterly Report under Contract Number DE-AC22-79ET14801, "Gas/Slurry Flow in Coal Liquefaction Processes." This work covers the period 1 January to 31 March 1981. This work is a continuation of studies initiated by Air Products and Chemicals, Inc. on the fluid dynamics of three-phase flow to support the design of the 6000 T/D dissolver for the SRC-I demonstration plant which began in July 1978. DOE supported these three-phase flow studies under the Bridging Task program from 1 July 1979 to 30 September 1979 at the start of the current contract. A background of information developed at Air Products prior to DOE support was included in the first Quarterly Report.

The 6000 T/D SRC-I demonstration plant will employ vertical tubular reactors feeding slurry and gas concurrently upward through these vessels. In the SRC-I design this reactor is essentially an empty vessel with only a distributor plate located near the inlet. Because the commercial plant represents a considerable scale-up over either Wilsonville or Ft. Lewis, this program is addressing the need for additional data on the behavior of three phase systems in large vessels. Parameters being investigated in this program are being studied at conditions that relate directly to the projected demonstration plant operating conditions. Air/water/sand three-phase flow systems in both a 5-inch diameter and a 12-inch diameter column is used under Tasks 2, 3 and 4 in this cold-flow simulator study program.

Tetralin was the organic fluid chosen to be investigated under Task 5 of this program. The objective of using Tetralin and glycol mixtures as organic fluids was to study the effect of fluid properties on the three-phase system. Surface tension and viscosity were the main physical properties of interest in this study. During this quarter gas holdup, solids distribution and axial liquid dispersion coefficients were measured.

Results show that the gas holdup is not changed considerably by the presence of solids; only a slight drop in gas holdup in their presence is noticed. The gas holdup was also affected by the fluid properties such as liquid viscosity and surface tension.

Results show consistently for ethylene glycol mixtures that the ratio of particle terminal velocity to the dispersion coefficient ( $V_p/E_{zp}$ ) is higher with larger particle size. These results were confirmed in both water and tetralin. The results also show that  $V_p/E_{zp}$  is lower with an increase in liquid viscosity. This is mainly due to the decrease in the solids terminal velocity. In the continuous run, i.e., with liquid flow it was observed that the solid distribution profile was less steep with a higher viscosity fluid and is also less steep with smaller particle size.

Axial liquid dispersion coefficients were measured in tetralin using a batch mode. The coefficients were found to increase with gas velocity. This result is consistent with air/water data.

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The major element of a coal dissolution section of any liquefaction plant is the dissolver. Although a considerable amount of liquefaction will occur in the preheater, a major amount of necessary chemical change will occur in the dissolver, namely sulfur removal, oil and distillate formation and solvent rehydrogenation.

Vertical tubular reactors are employed in all of the major processes currently under consideration for commercial liquefaction of coal. In all of these processes, Solvent Refined Coal (SRC), EDS and H-Coal, slurry and gas are concurrently fed upward through these vessels. In the EDS and SRC processes, the reactors are essentially empty vessels, whereas in the H-Coal process, a bed of ebullating catalyst is maintained in the reactor. The major difference between the EDS, SRC-I and SRC-II processes in dissolver operation are the composition of the feed streams and reactants within the dissolver. Other hardware differences such as distributor plates, draft tubes or recycle loops can also cause differences in the behavior of slurries in these vessels. A requirement necessary to any design that will be technically feasible and cost effective is an understanding of the physical behavior of three phase systems in tubular columns.

All of the major processes under development require the understanding of a backmixed three phase system. Each process employs at least a portion of its dissolver volume in a backmixed mode. As the design of the 6000 T/D SRC-I plant progresses, the increased vessel size (and other considerations) may dictate the use of reactors in series, which would decrease the overall backmixed characteristic of the commercial plant.

The SRC-I demonstration plant dissolver will represent considerable scale-up over the Wilsonville and Ft. Lewis dissolver. To intelligently make good design decisions, more information is needed on the flow properties of the three phase systems in large vessels. More important from the standpoint of slurry behavior is the difference in gas and

liquid superficial velocities. This difference can have considerable impact on the process because the gas and liquid superficial velocities have a strong effect on (a) gas void volume, (b) actual solids concentration in the dissolver and (c) the relative degree of backmixing. As velocity through the dissolver increases, the tendency for solids to remain behind diminishes causing a decrease in the actual concentration of ash particles in the reactor. Those particles that do remain behind will tend to be larger in size. Since considerable evidence points to a definite catalytic effect of the reactor solids, the larger particles will have decreased surface areas exposed and, therefore, will have diminished catalytic activity. Knowing that particle sizes can accumulate under commercial flow conditions will give an indication of size of dissolver solids that should be examined for catalytic activity.

Considerable work on the behavior of gas/liquid mixtures flowing through vertical columns has been reported in the literature. Information on three phase (gas/liquid/solid) systems is far less extensive. Detailed background information was presented in the first quarterly report (1). Under this contract, work is being conducted in a 5-inch diameter by 5-foot tall Plexiglas column and a 12-inch diameter by 25-foot tall glass column with are located at the contractor's site. The physical dimensions, auxiliary equipment, and some of the experimental techniques employed in this study were extensively discussed in the first quarterly report. This report contains experimental results from runs conducted during the 1 April-30 June 1981 reporting period.

## 2.0 Technical Progress

### 2.1 Task 5 - Organic Fluid Phases

The objectives of this task are as follows:

- o Study the effect of gas velocity, solids concentration and liquids velocity on gas holdup in a recycle mode for tetralin



- o Study the effect of gas velocity, liquid velocity and particle size on solids concentration in a recycle mode for tetralin
- o Study the effect of gas velocity on the liquid dispersion coefficient of tetralin
- o Investigate gas holdup in different solutions of ethylene glycol
- o Study the effect of gas velocity, particle size, ethylene glycol and water mixture concentration on axial solids distribution
- o Study the effect of gas velocity, liquid velocity and particle size on solids distribution in ethylene glycol mixtures with a recycle mode.

### 3.0 Experimental Section

#### 3.1 Cold Flow Model Equipment

Both the 5-inch diameter and 12-inch diameter column used in these cold flow studies were described in detail in the first Quarterly Report (January 1980). The distributor used in the 12-inch column for all these runs was No. 1 distributor (December 1980). The enclosure for the 12-inch column to protect against volatile fumes was described in detail in the last Quarterly Report (February 1981).

#### 3.2 Experimental Procedure

Several experimental procedures were normally used in this Program depending on the type of experimental run. In this Section, a brief description of the experimental procedure for the runs conducted in this quarter are given.

##### 3.2.1 Gas Holdup

Gas holdup was measured in the 12-inch diameter column in both the absence and presence of fluid flow. Effects of solid particles on gas holdup were also investigated in the presence of fluid flow. The organic fluids used were tetralin and ethylene glycol. In the absence of liquid flow, the experiments were performed by completely filling

the column with liquid and then passing the gas through the liquid at specified rates. Excess liquid exited the column at the top through a side opening. A waiting period of five minutes was used to ensure that steady state was achieved. The bottom valve was then closed to shut off the gas input. The final liquid level was measured, and the difference between the initial and the final levels represented the gas holdup at that particular gas flow rate.

### 3.2.2 Liquid Dispersion

Liquid dispersion experiment in tetralin/nitrogen and sand/tetralin/nitrogen systems were investigated in the 12-inch and 5-inch diameter columns using a tracer detection method. The use of tetralin as the liquid phase required the development of a new experimental method. A method was developed and was tested in the 5-inch diameter column in two liquid dispersion runs. After the successful demonstration of the method in the 5-inch diameter column, several experiments were conducted in the 12-inch diameter column. A brief description of the method and its development is present in this section.

The use of tetralin as a liquid phase for liquid-dispersion experiments created some special problems that were not present in our earlier air/water experiments. In the case of air/water experiments, sodium chloride was used as the tracer and conductivity probe mounted at the exit-line of the column monitored the ion conductivity of the solution continuously. Towards the end of each experiment excess liquid was required to flow continuously into the column until the concentration of the tracer in the exit of the column drops to zero. This procedure required the use of enormous amount of liquids. For example, a typical liquid dispersion run operating at a liquid velocity 0.05 ft/sec would last approximately thirty minutes; this would require about 550 gallons of liquid material. In the case of water, a fresh supply was not a major problem. However, only a limited supply of tetralin is available due to the cost and safety aspects. Hence a decision was made to conduct liquid dispersion runs in a batch mode

(with no liquid flow). Since earlier results clearly showed that the liquid velocity has no effect on the liquid dispersion coefficient, batch experiments would provide us with all the necessary information.

A blue dye soluble in tetralin was chosen as the tracer for liquid dispersion experiments using tetralin; the change in absorbance of the liquid at a given wavelength was monitored using colorimetry. Colorimetry was chosen after carefully considering several methods. The choice of tracer for experiments using tetralin should meet the following conditions:

- o the tracer should be detectible in very small concentration
- o the tracer should be miscible with tetralin
- o the tracer should be removable from tetralin

The third condition was the most difficult one to meet but was expendable. Hence it was decided not to remove the tracer from tetralin but use sufficiently small quantities in such a way that the cumulative amount of tracer added after several experiments would not alter the physical properties of tetralin. Since tetralin has a slight pale yellow tint, the dye should be chosen in such a way that the colored solution exhibits a maximum absorbance at a wavelength far different than the wavelength at which tetralin would exhibit maximum adsorbance. A blue dye was chosen to meet this requirement.

In a typical experimental procedure, the column was filled with tetralin and gas was passed through the column at a desired rate. A concentrated solution of the dye in tetralin was then injected at the bottom of the column and sample were taken at a point 20 feet from the injection port for every 20 seconds. The total amount of sample liquid withdrawn during the course of the experiment is negligible compared to the amount of liquid in the column. (Continuous monitoring

of the dye concentration in the column failed due to the interference of gas bubbles in the liquid.) The adsorbance of the samples collected from the column are measured immediately. However, in the case of experiments using solid particles, the samples were analyzed after the solid particles settled at the bottom. Suspended particles scattered light and gave erroneous readings on the colorimeter. Adsorbance versus time plots obtained from these experiments were transformed to dimensionless curves and tracer curves were prepared. The axial dispersion coefficients were determined from these non-dimensional curves.

### 3.2.3 Solids Distribution

Experiments were conducted in this quarter to study the effect of liquid velocity on the solids distribution in the 12-inch column using tetralin and ethylene glycol-water solutions. Batch experiments were also conducted for the ethylene glycol-water solution. Distributor #1 which was described earlier (FE-14801-12) was used in all these experiments. The experiments were conducted in the recycle mode partially because of the safety aspects to handle tetralin and ethylene-glycol. Both of the #140 minus sand and #60/80 sand were used in the tetralin recycle runs, whereas only the #60/80 mesh sand was used for the glycol runs in this quarter. To achieve recycle conditions the exit from the column was returned to the feed tank thereby creating a closed loop. The recycle loop was allowed to operate for several hours in order to achieve steady state. Solids concentration profiles were measured from slurry samples withdrawn from sampling ports located along the axis of the column. Usually two samples are withdrawn at each axial position: one from the center of the 12-inch column and one from the side of the tube near the wall.

## 4.0 Results and Discussion

### 4.1 Gas Holdup

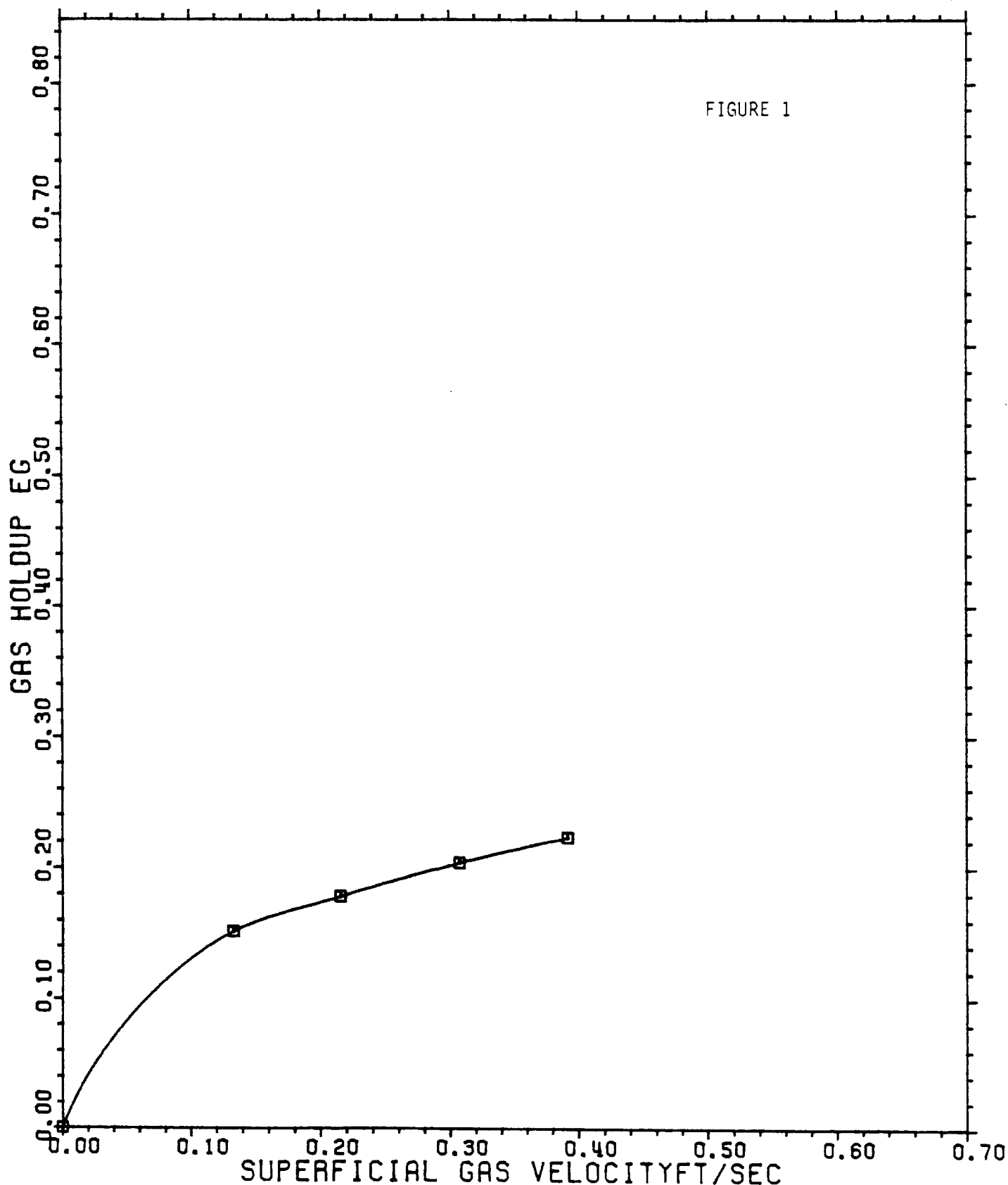
#### 4.1.1 Effect of Gas Velocity and Particle Size on Gas Holdup (Tetralin)

Figures (1) and (2) show the results of gas holdup in tetralin as a function of gas velocity in the presence of 60/80 mesh and 140 mesh minus sand particles, respectively. Comparing these values with the gas holdup of pure tetralin with no solids (DOE-TIC 14801-18) it is apparent that there is no significant change in the gas holdup for the presence or absence of solids. The gas holdup is slightly lower for the 140 mesh minus sand than that for the 60/80 mesh sand, however. One possible explanation is the higher available surface area of the 140 mesh minus particles (for equal volume of the 60/80 mesh), thereby requiring more of the turbulent energy to overcome the frictional work on the particle interfacial surface. Hence the turbulent activity of the liquid available for breaking up gas bubbles was decreased, resulting with slightly larger bubble size (average). Therefore the gas holdup is slightly lower in the presence of the 140 mesh minus particles.

The air/water gas-holdup data shows similar trends to the ones obtained here with no appreciable change in gas-holdup in the presence of solids. In addition, the air/water/sand gas holdup data show a slight decrease in gas holdup in the presence of fine particles (140 mesh minus).

#### 4.1.2 Effect of Liquid Velocity in the Presence of Solids (Tetralin)

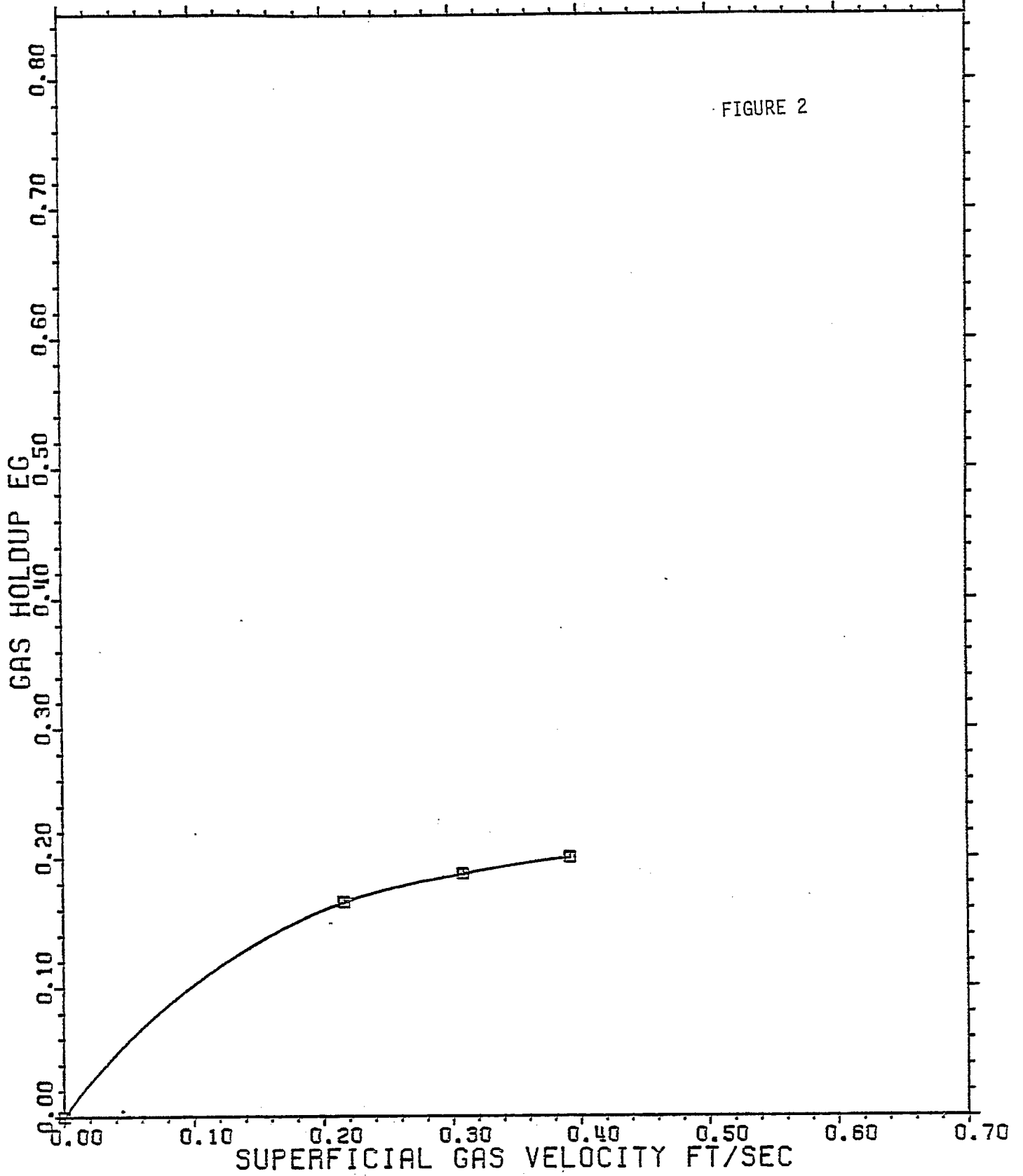
The liquid velocity has essentially no effect on the gas-holdup as shown by Figures (3) and (4). Gas holdup values at a fixed gas velocity of 0.39 ft/sec were plotted as function of liquid velocities in the presence of both large (60/80 mesh) and fine (140 mesh minus) sand particles. With an almost five-fold change in liquid velocity, the gas holdup was practically unaffected.



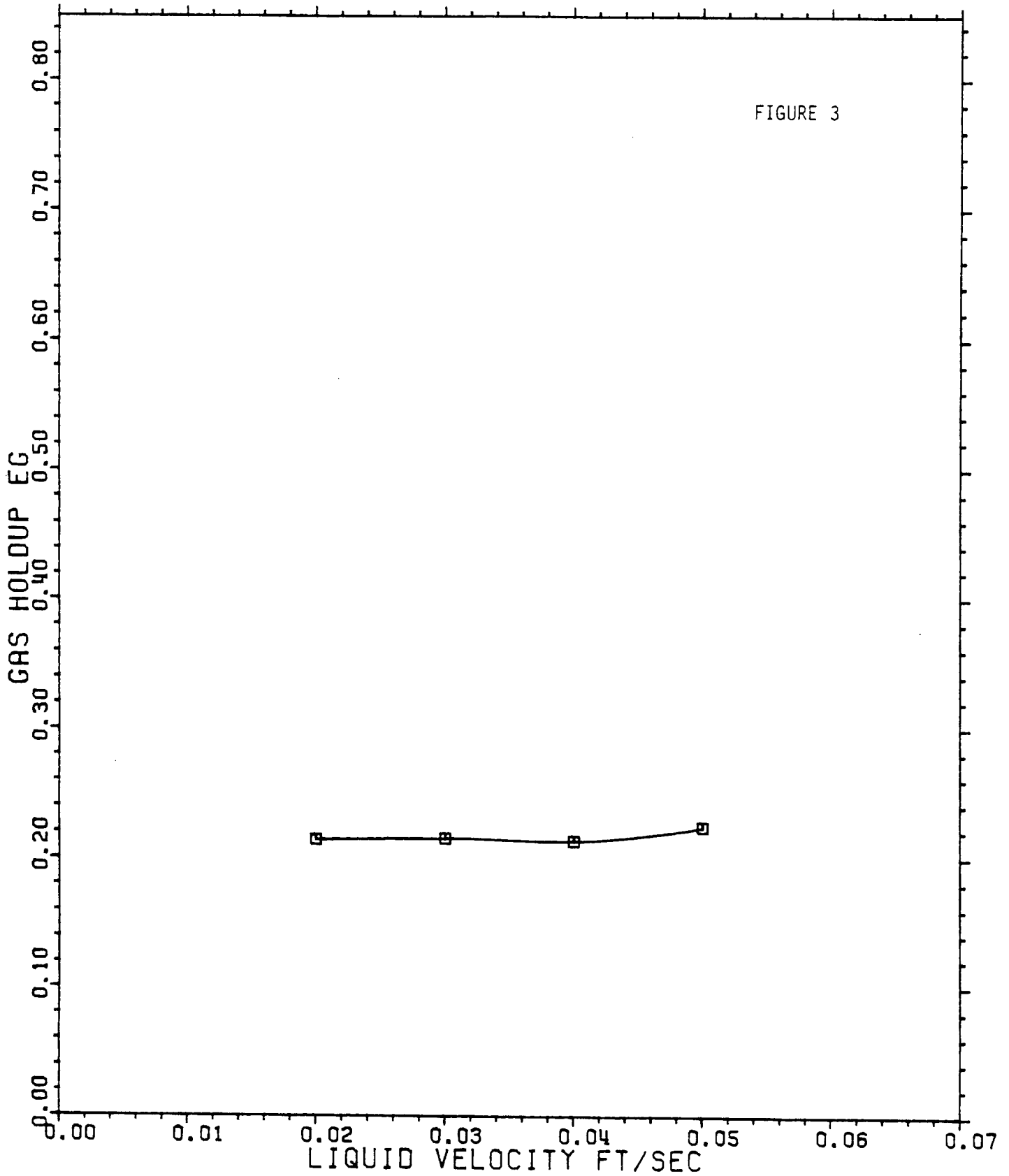
EFFECT OF VG ON EG 12" COL.

EFFECT OF GAS VELOCITY ON GAS HOLDUP  
 •60/80 MESH SAND (RECYCLE MODE)  
 VL=.05 FT/SEC

□



E,	
EFFECT OF GAS VELOCITY ON GAS HOLDUP	
*140 MINUS SAND (RECYCLE MODE)	
VL=.05 FT/SEC	9



EFFECT OF VL ON EG (12" COL.)

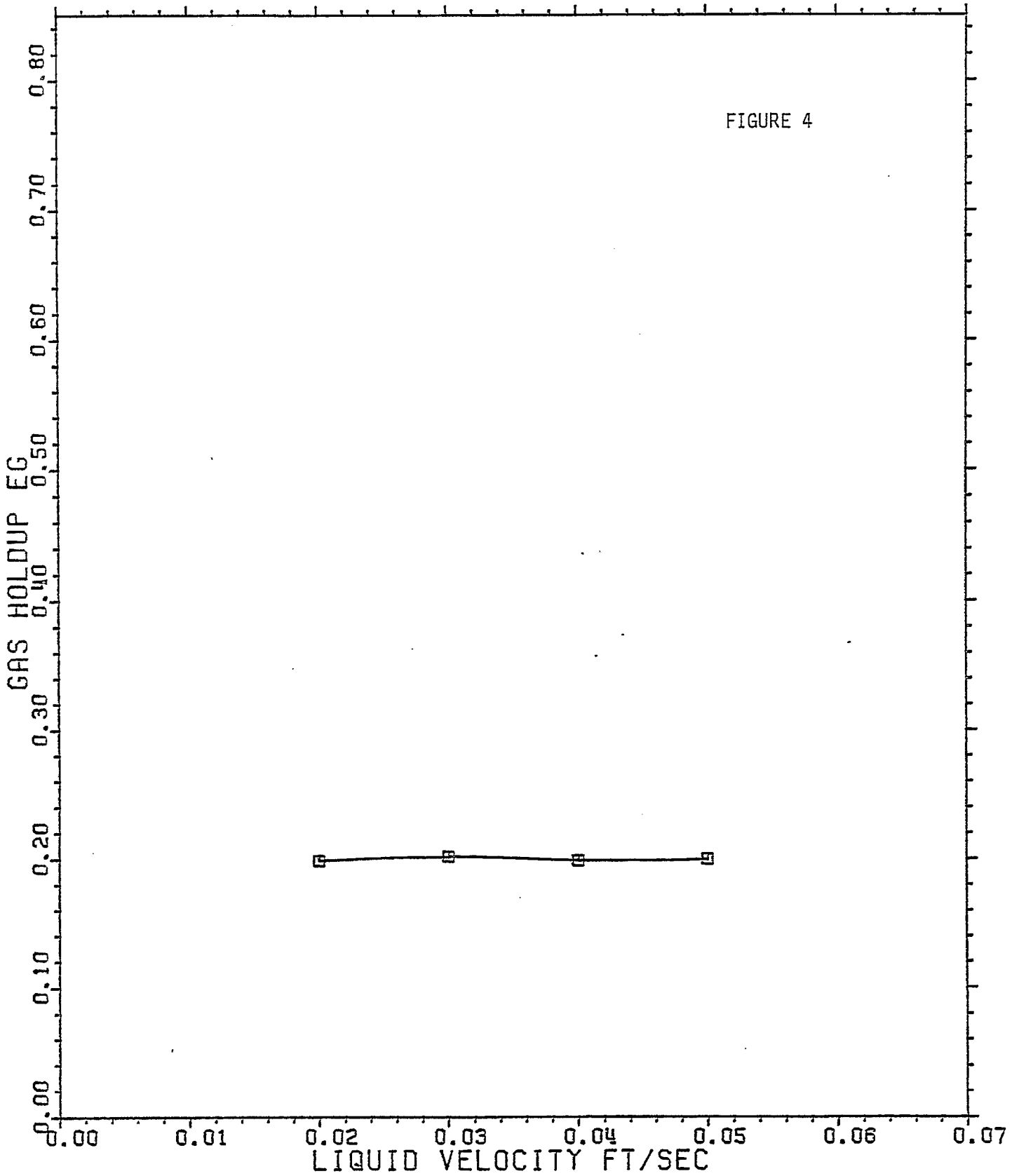
EFFECT OF LIQUID VELOCITY ON GAS HOLDUP

•80/80 MESH SAND (RECYCLE MODE)

VG- .392 FT/SEC

□





EFFECT OF VL ON EG (12" COL.)

EFFECT OF LIQUID VELOCITY ON GAS HOLDUP

\*140 MINUS MESH SAND (RECYCLE MODE)

VG- .392 FT/SEC

11

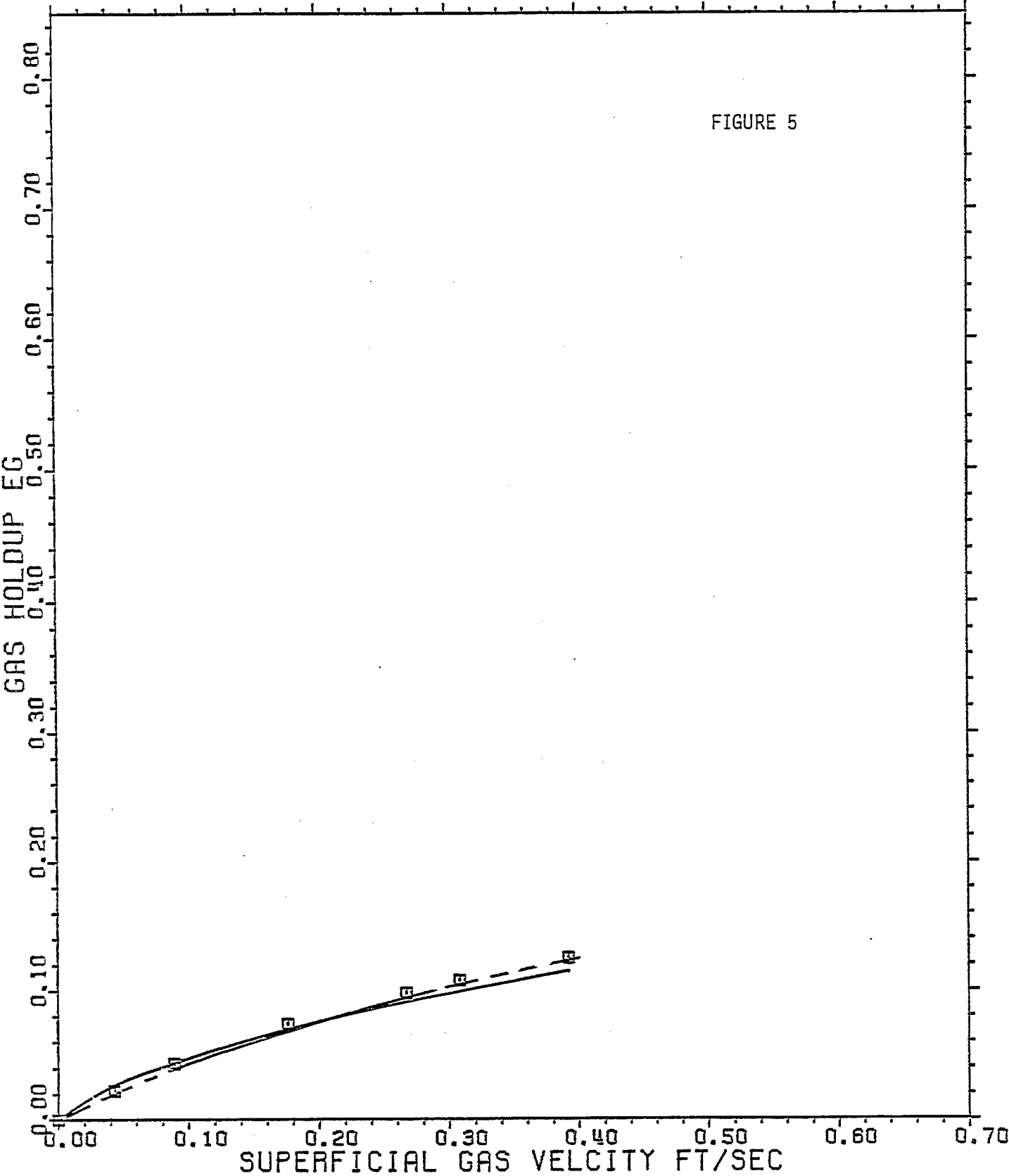
□

#### 4.1.3 Effect of Gas Velocity (Ethylene Glycol-Water Mixtures)

Gas-holdup in different ethylene glycol-water mixtures (100 wt%, 90 wt%, 70 wt% and 50 wt% glycol) was measured. Figures (5) through (8) show the effect of gas velocity on gas-holdup in these mixtures. In these figures, the square symbol represents experimental data which were compared with predictions from two different correlations. The solid line represents Pilhofer's correlation [1] and the broken line represents Akita and Yoshida's correlation [2]. These correlations have been used to fit the air/water and nitrogen/tetralin data (FE-14801-3 and TIC-14801-18). Both correlations are independent of the column diameter but the latter is a function of surface tension. Both correlations are functions of the kinematic viscosity ( $\nu_L$ ) of the continuous phase. Pilhofer's correlation has the gas-holdup proportional to  $(\nu_L)^{-0.23}$  and Akita and Yoshida used a proportionality of  $(\nu_L)^{-0.166}$ . Akita and Yoshida's correlation was used with an empirical constant value of 0.2 for all the comparisons assuming that all these organic mixtures are not highly polar. Table (1) shows the physical properties of the four glycol-water mixtures used in this study. Both correlations model the data reasonably well in the 12-inch column.

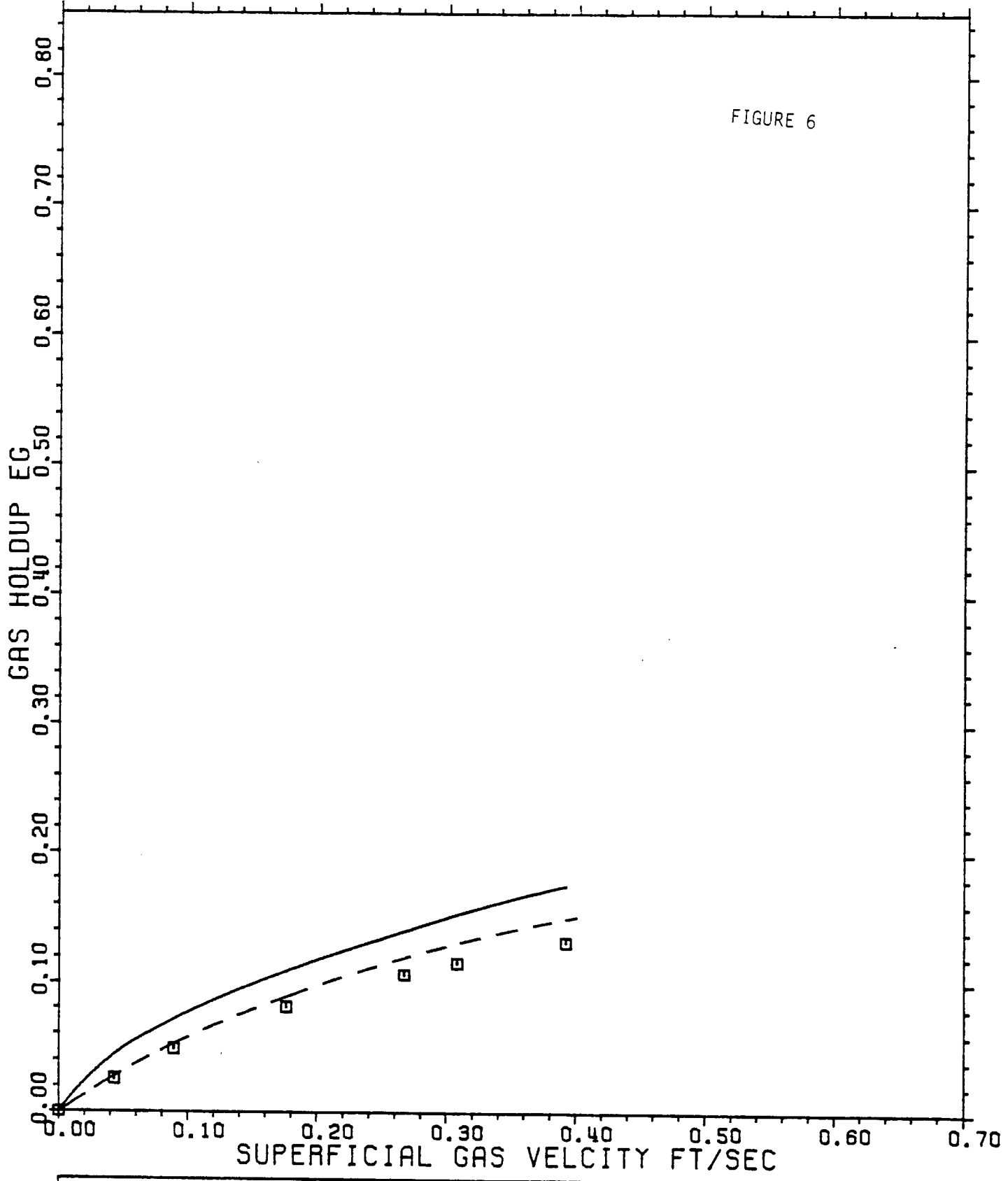
It is interesting to note that, the gas-holdup in the 100 wt% and 90 wt% glycol is practically the same knowing that the viscosity is reduced by about 1.5 times between the 100% to 90% mixture and the surface tension stays practically the same. In the case of the 90% mixture the correlation of Akita and Yoshida is better. However, use of the empirical constant value of 0.2 has underpredicted the values of gas holdup in the 70 wt% and 50 wt% mixtures using Akita's correlation. If a higher value 0.25 by assuming polar solution was used, the fit of the gas-holdup data was improved. Table (2) shows the comparison of the gas-holdup values using the different correlations for the 70 wt% and 50 wt% mixtures. In general, the Pilhofer's correlation fits the tetralin and the glycol gas holdup data better.

FIGURE 5



GAS HOLDUP	
100% GLYCOL GAS HOLDUP IN 12" COL.	<ul style="list-style-type: none"><li>□ EXPERIMENTAL VALUES OF EG</li><li>— PILHOFER ET AL. CORRELATION</li><li>- - AKIDA AND YOSHIDA CORRELATION</li></ul>

FIGURE 6

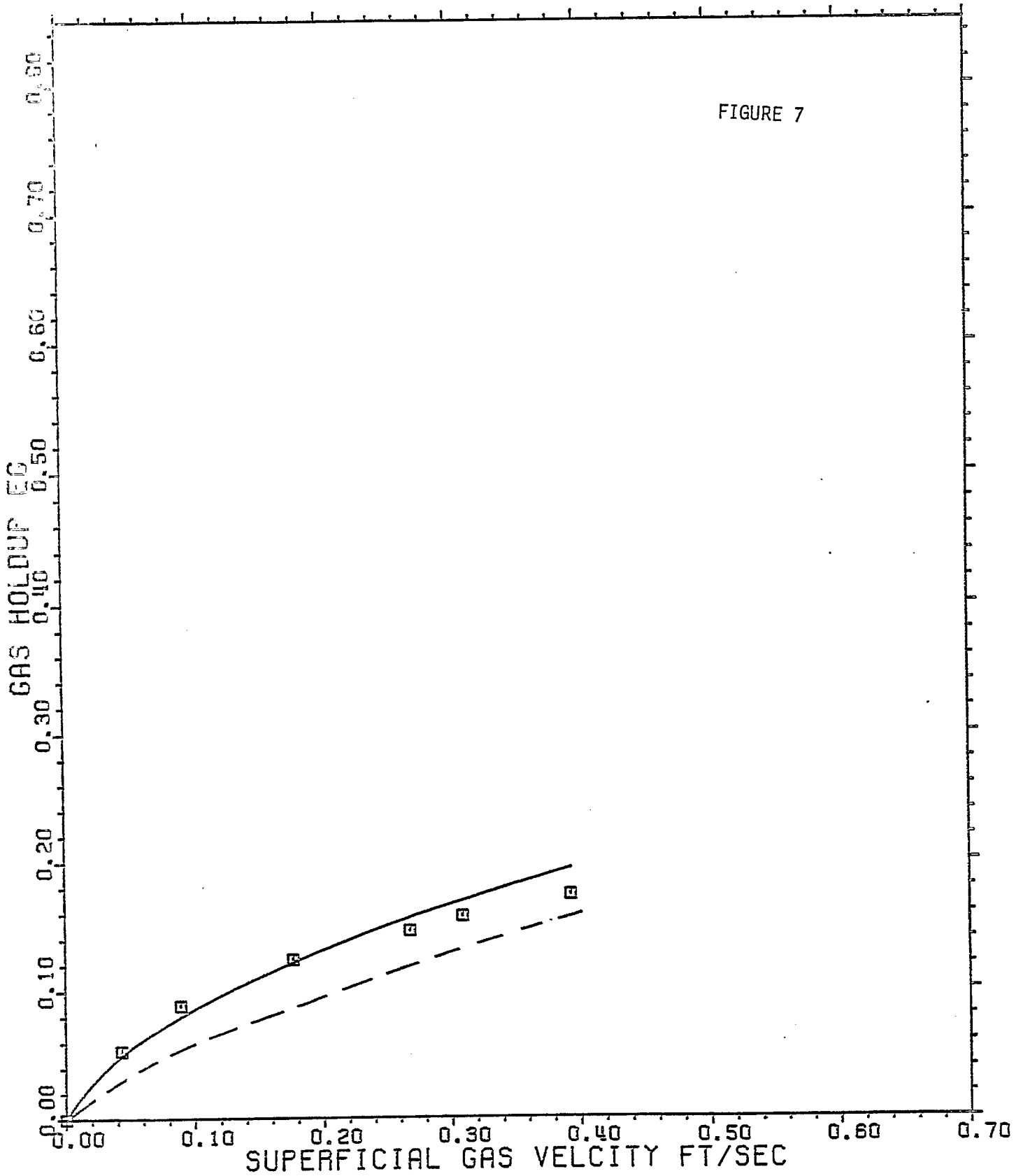


GAS HOLDUP

90% GLYCOL GAS HOLDUP IN 12" COL.

□ EXPERIMENTAL VALUES OF EG  
— PILHOFFER ET AL. CORRELATION  
- - AKIDA AND YOSHIDA CORRELATION

FIGURE 7

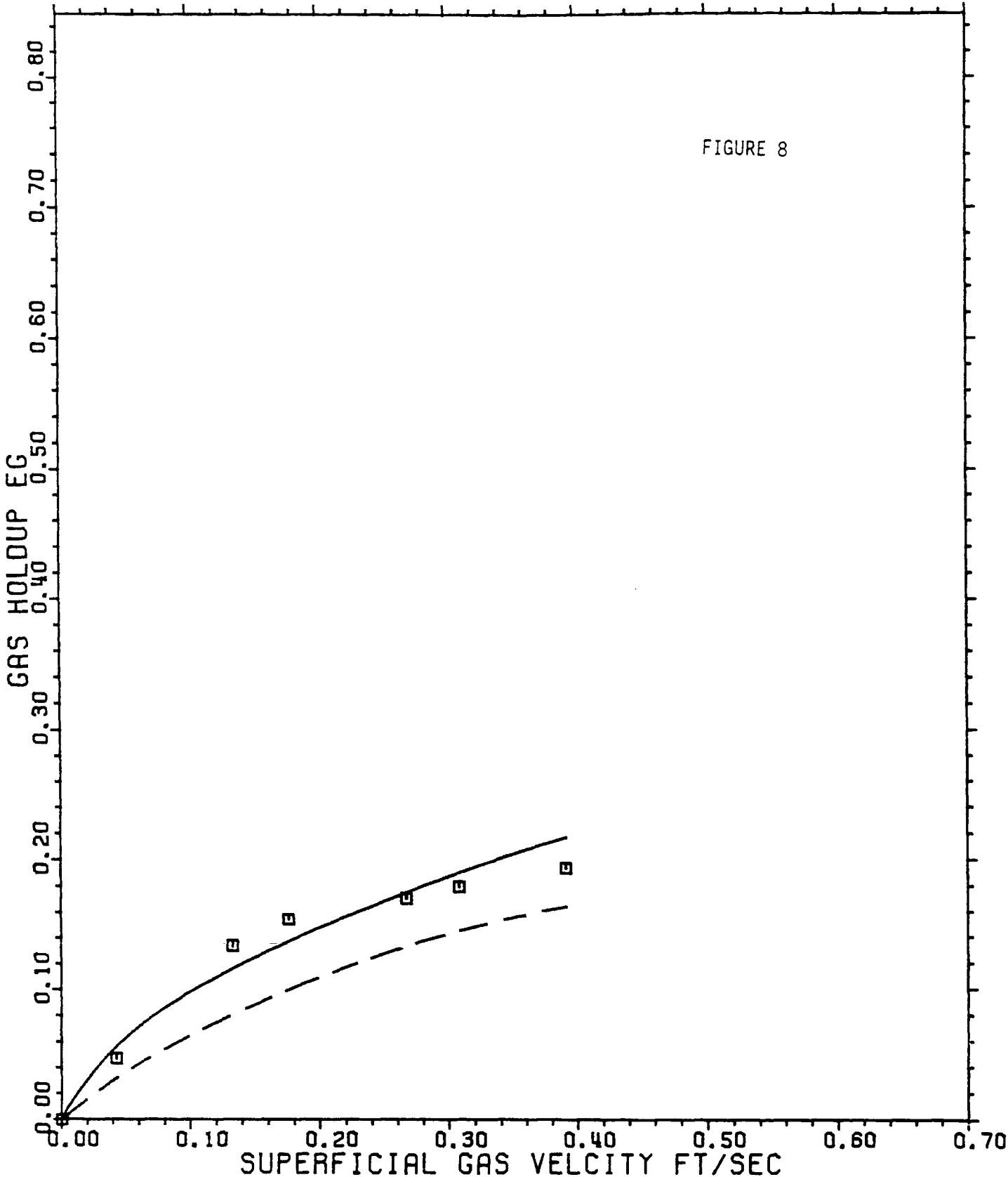


GAS HOLDUP

70% GLYCOL GAS HOLDUP IN 12" COL.

- EXPERIMENTAL VALUES OF EG
- PILHOFER ET AL. CORRELATION
- - AKIDA AND YOSHIDA CORRELATION

FIGURE 8



GAS HOLDUP	
50% GLYCOL GAS HOLDUP IN 12" COL.	<ul style="list-style-type: none"><li>□ EXPERIMENTAL VALUES OF EG</li><li>— PILHOFER ET AL. CORRELATION</li><li>- - AKIDA AND YOSHIDA CORRELATION</li></ul>

Table 1  
Physical Properties of Different  
Ethylene Glycol and Water Mixture

<u>Mixture</u>	<u>Density gm/ml</u>	<u>Viscosity, Cps</u>	<u>Surface Tension dynes/cm</u>
100% Glycol	1.170	19.1	48.2
90% wt. glycol	1.107	13.08	45.9
70% wt. glycol	1.091	6.40	48.8
50% wt. glycol	1.078	4.10	56.7

Table 2

Comparison of Correlations and Experimental  
Gas Holdup for 70 wt% and 50 wt% Glycol Solutions

70 wt% Glycol				
<u>Vg</u> ft/sec	<u>Pilhofer</u>	Akita <u><math>C_1 = 0.2</math></u>	Akita <u><math>C_1 = 0.25</math></u>	<u>Exp.</u>
0.043	0.050	0.031	0.038	0.054
0.176	0.122	0.096	0.111	0.124
0.392	0.195	0.160	0.181	0.175

50 Glyco by wt. 1				
<u>Vg</u> ft/sec	<u>Pilhofer</u>	Akita <u><math>C_1 = 0.2</math></u>	Akita <u><math>C_1 = 0.25</math></u>	<u>Exp.</u>
0.043	0.056	0.032	0.039	0.047
0.176	0.137	0.100	0.115	0.158
0.392	0.217	0.165	0.185	0.194



Additional data were obtained in the 5-inch column using the 100% glycol and 90% solutions. The results are shown in Figures (9) and (10). The gas holdup in these two solutions are practically identical as shown previously in the 12-inch column. However, the values of gas-holdup in the 5-inch column are slightly higher than those of the 12-inch column. The difference tend to increase as the superficial velocity increases. At this point in time, little can be said about this different or to assert if there is any definite effect of diameter on gas-holdup for glycol. Other gas-holdup runs in air/water and nitrogen/tetralin show no appreciable effect.

#### 4.1.4 Effect of Liquid Velocity and Solid Particles (50 wt% Glycol Solution)

Liquid flow has no effect on gas-holdup. Figure (11) shows no appreciable change in gas holdup with a five-fold increase in liquid velocity. This is consistent with the findings in water and tetralin systems. The presence of solids slightly decreases the gas holdup consistent with tetralin and water data.

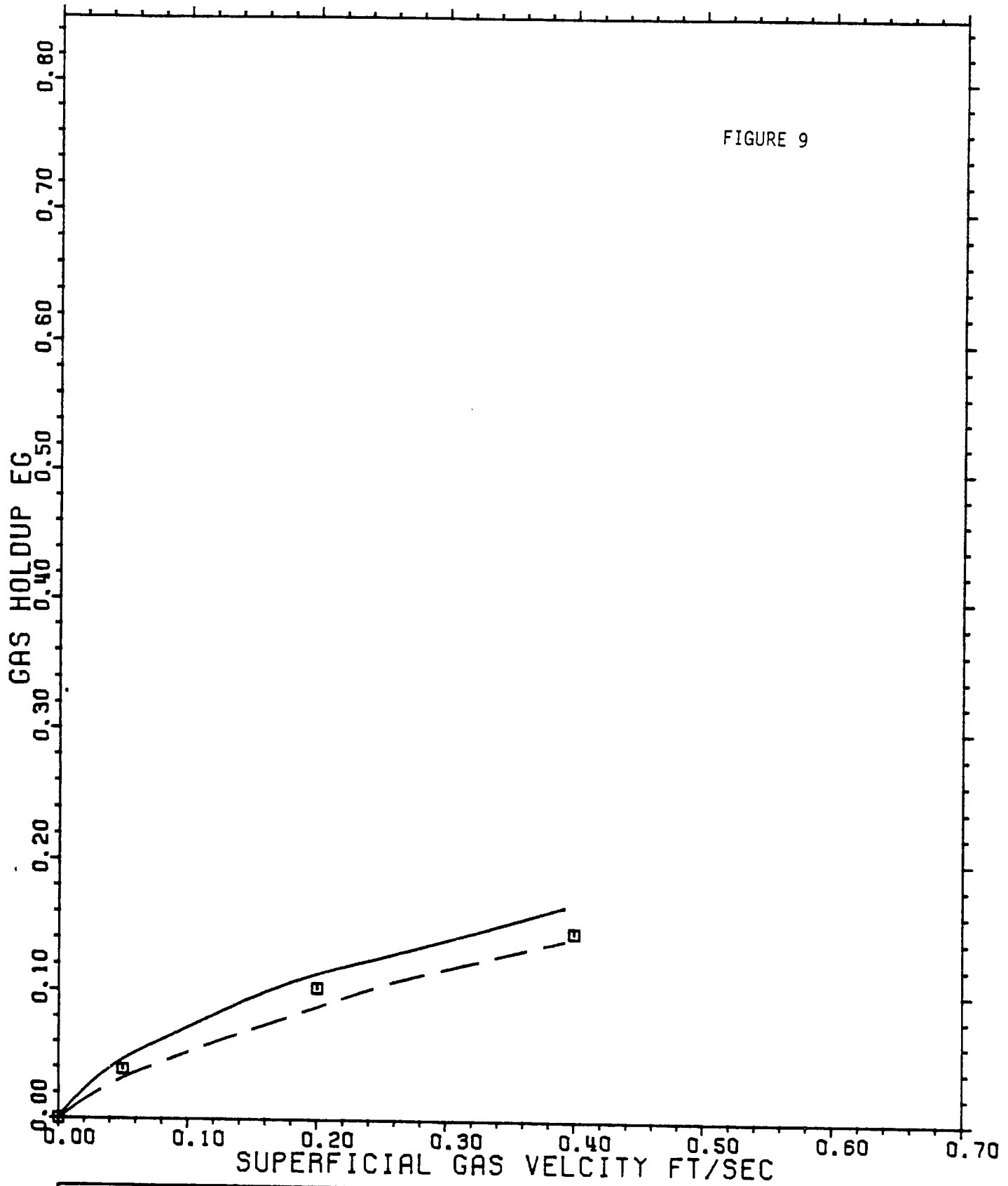
#### 4.2 Solids Distribution

##### 4.2.1 Solids Distribution in Tetralin (140 Mesh Minus and 60/80 Mesh Sand)

Typical solids concentration at various column heights as a function of time are shown in Figures (12) and (13) for two particle sizes. Undoubtedly steady state has been achieved in three hours after experiment start-up. In the data presented below, the average of these values at each level of column was used.

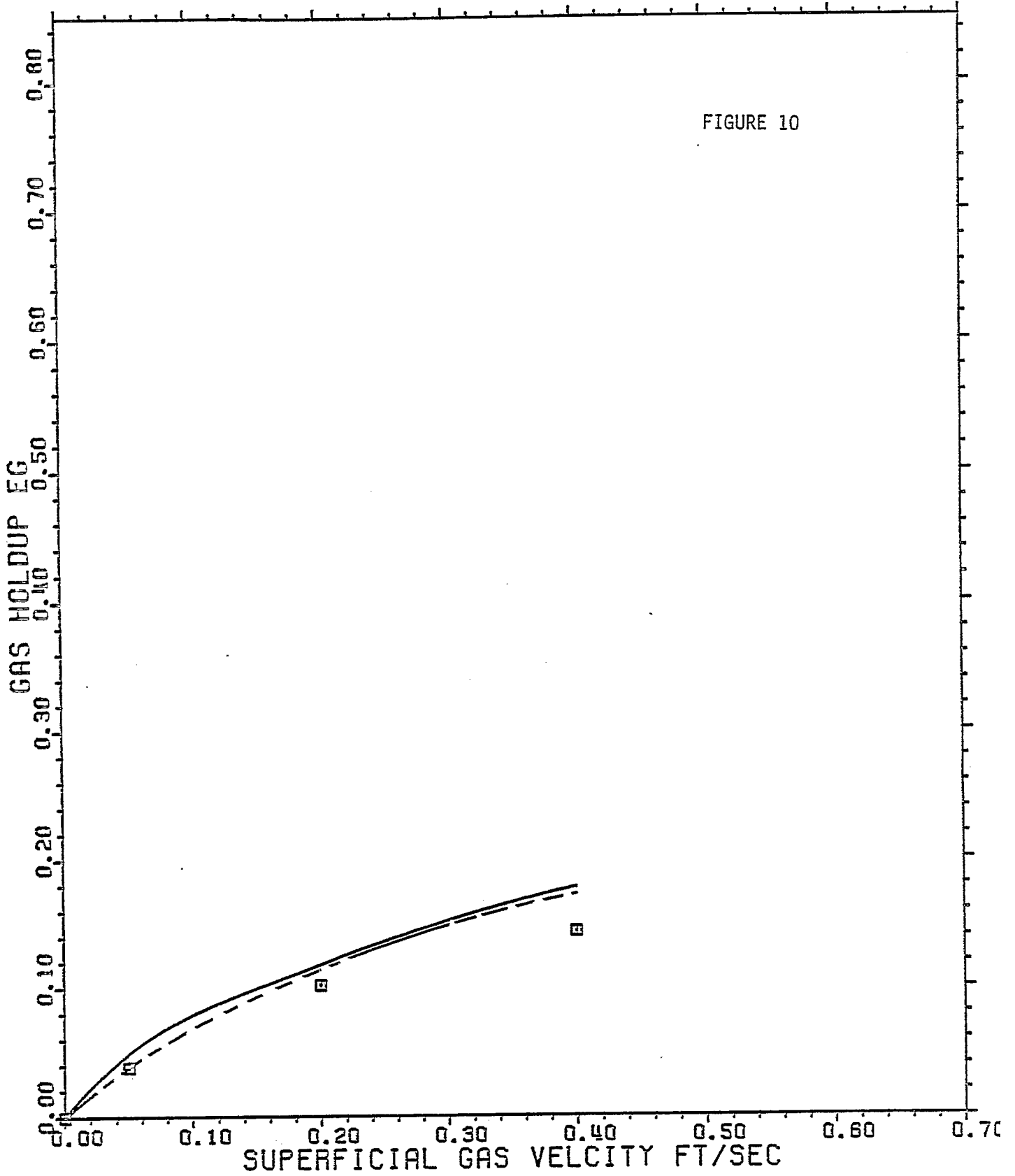
Effect of gas and liquid velocities on the distribution of 140 mesh minus sand particles in tetralin are seen in Figures (14) and (15), respectively. Both figures showed rather flat solid concentration profiles for the entire range of flow rates investigated. This observed flat profiles are very similar to those found in air/water/sand systems. Due to the low settling velocities of these fine particles, the solids are almost homogeneously mixed. Therefore, the

FIGURE 9



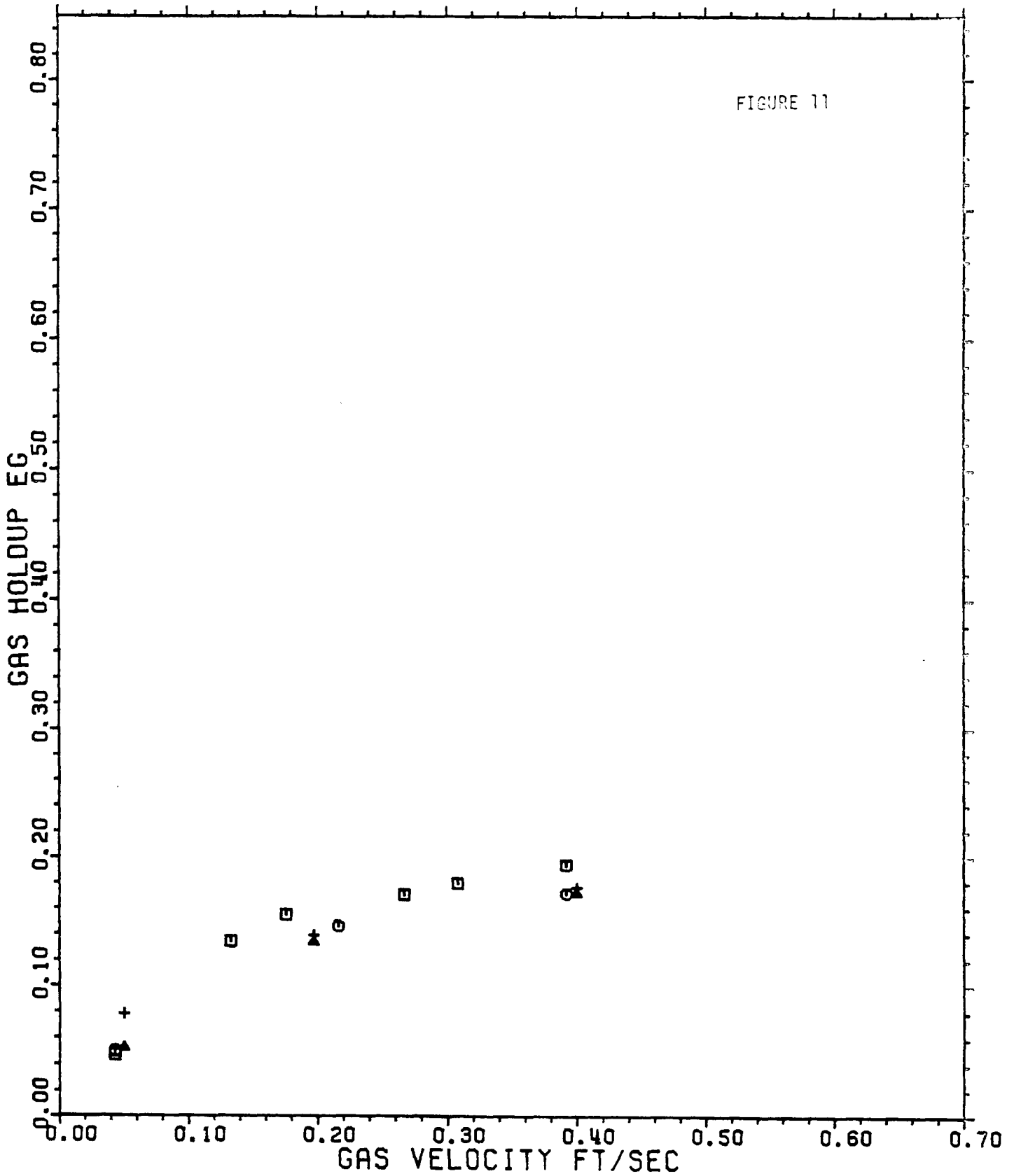
GAS HOLDUP	
100% GLYCOL GAS HOLDUP IN 5" COL.	<ul style="list-style-type: none"><li>□ EXPERIMENTAL VALUES OF EG</li><li>- - FILHOFFER ET AL. CORRELATION</li><li>- - AKIDA AND YOSHIDA CORRELATION</li></ul>

FIGURE 10



GAS HOLDUP	
50% GLYCOL GAS HOLDUP IN 5" COL.	<ul style="list-style-type: none"> <li>□ EXPERIMENTAL VALUES OF EG</li> <li>— PILHOFER ET AL. CORRELATION</li> <li>- - - ARIDA AND YOSHIDA CORRELATION</li> </ul>

FIGURE 11

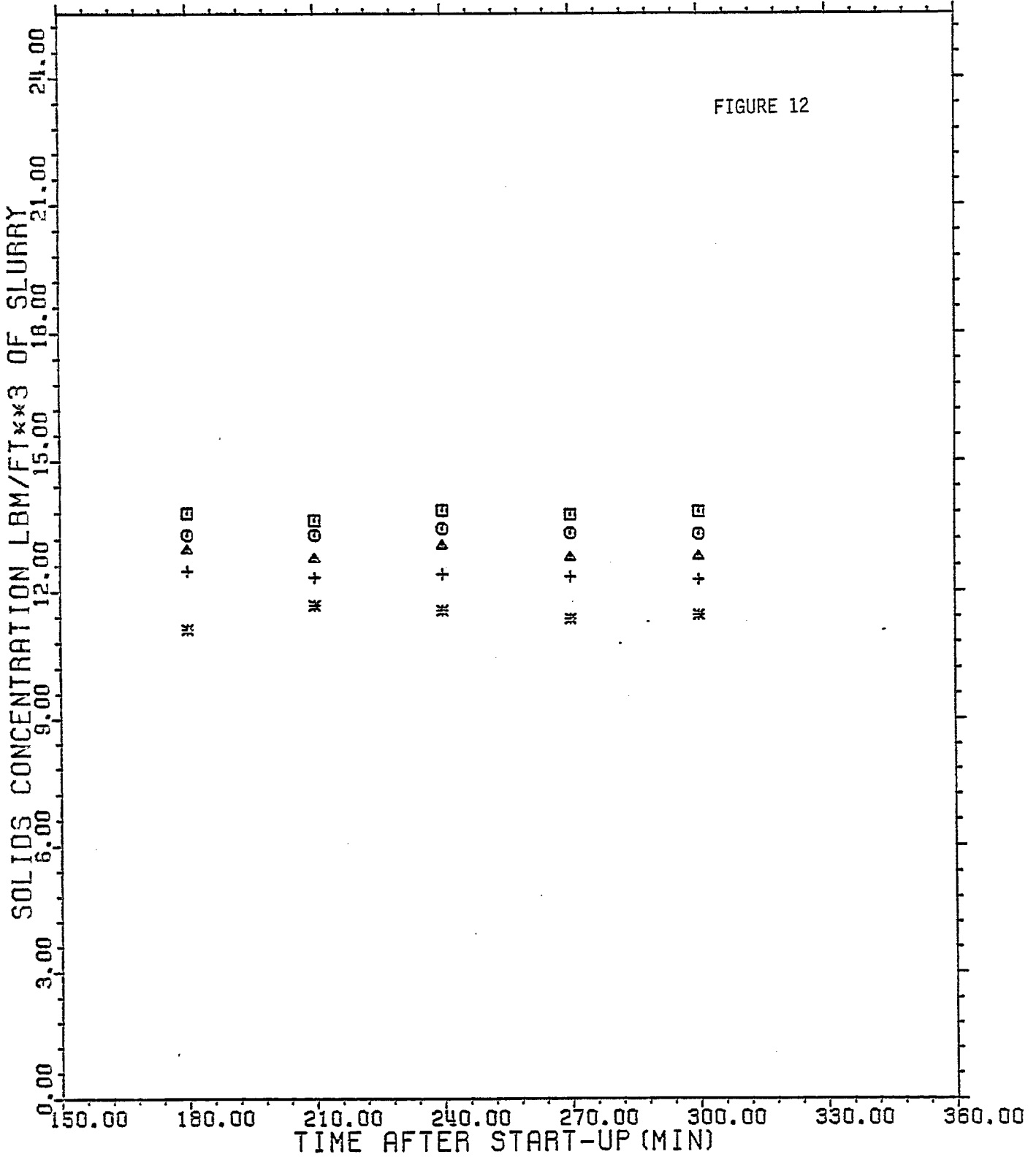


**EFFECT OF VL AND SOLIDS ON G**

EFFECT OF SOLIDS AND VL ON EG  
 12" COLUM, 50% GLYCOL #80/80 MESH SAND

- NO SOLIDS PRESENT
- VL = .01-.015 FT/SEC, #80/80 MESH SAND
- ▲ VL = .03 FT/SEC, #80/80 MESH SAND
- + VL = .05 FT/SEC, #80/80 MESH SAND

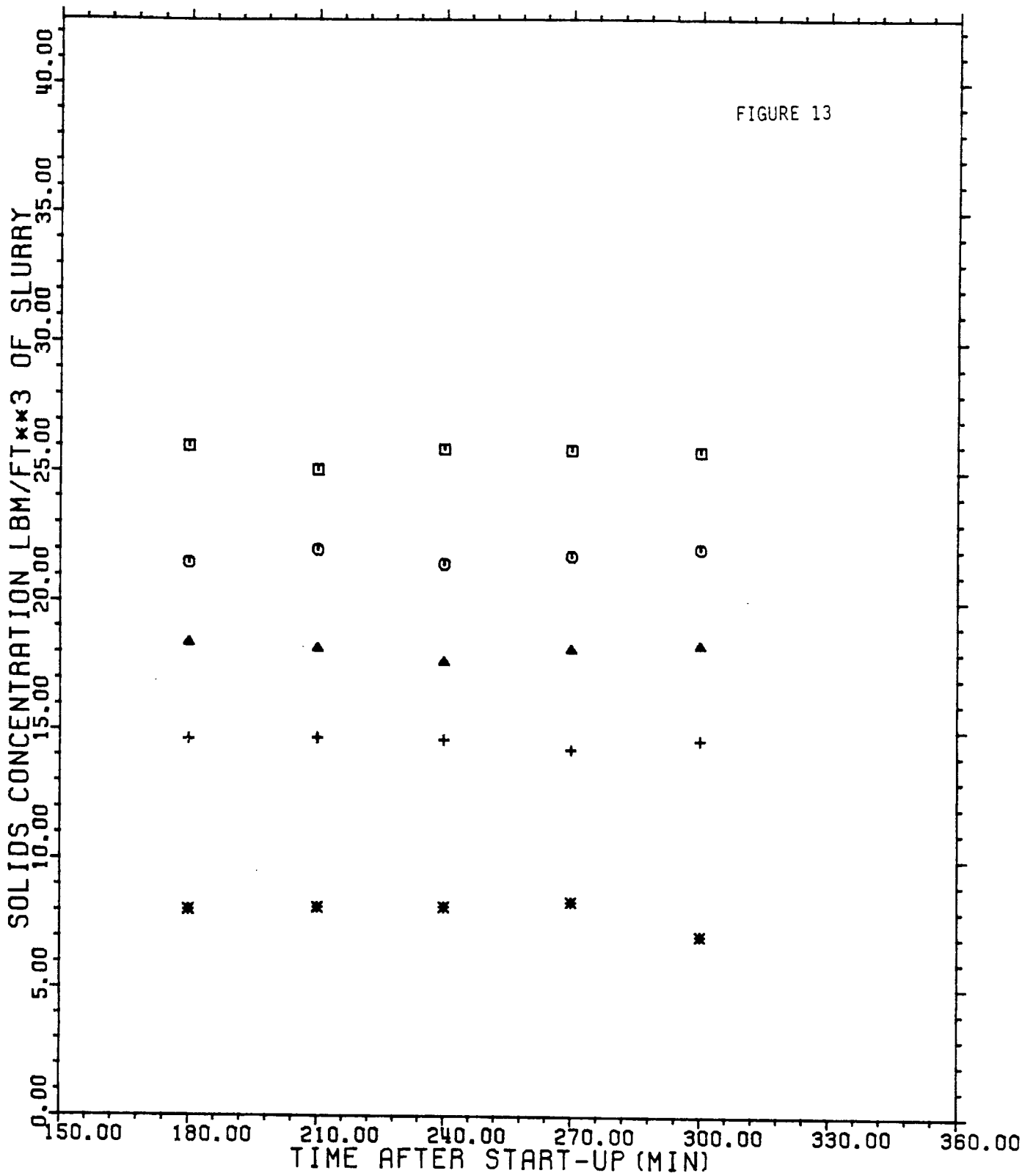
FIGURE 12



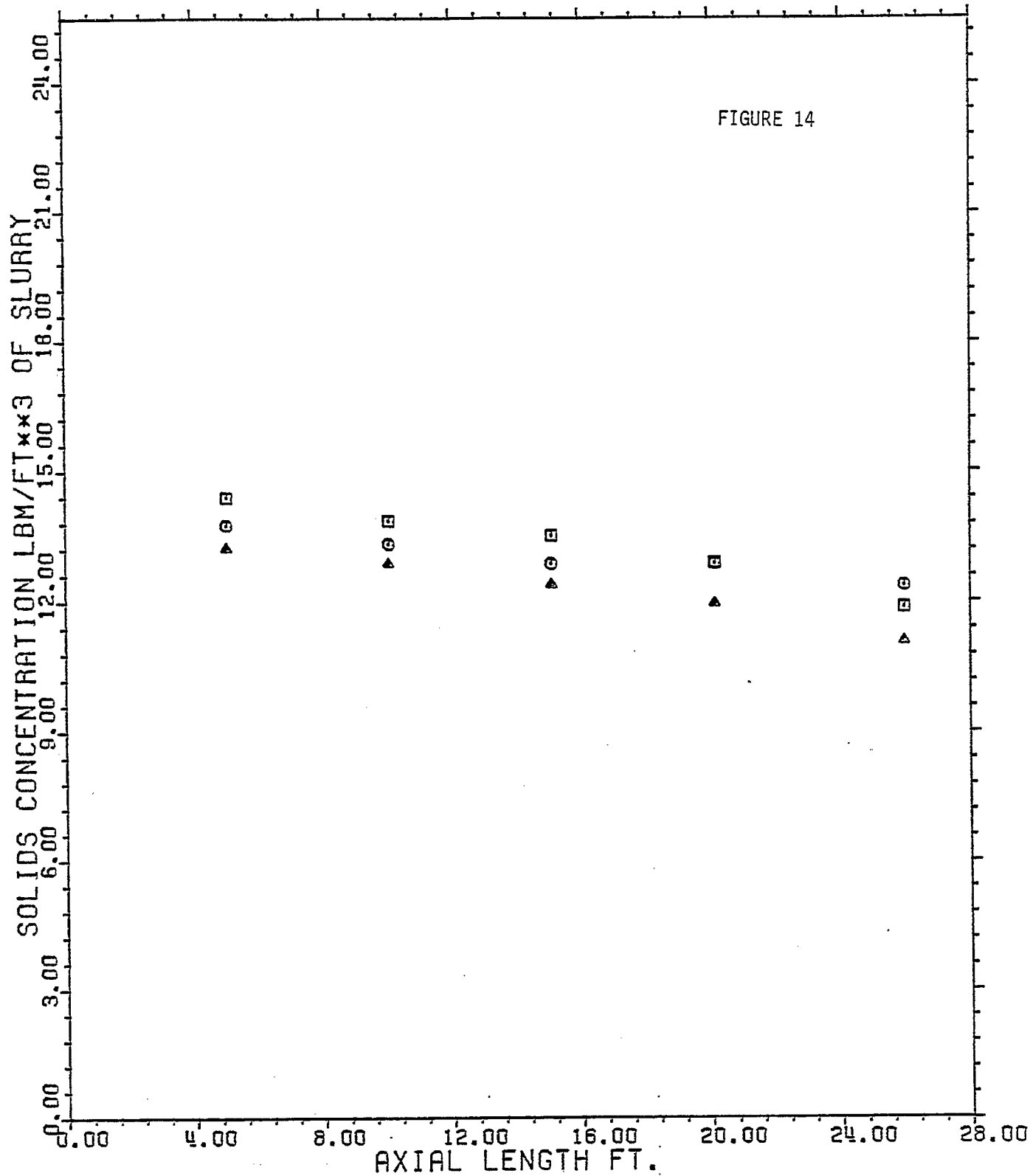
SOLIDS VARIATION WITH TIME

C. VS. T IN A 12" COL. (RECYCLE)  
 \*140 MESH SAND VL=.05 FT/SEC, VG=.308 FT/SEC  
 LIQUID USED IS TETRALIN

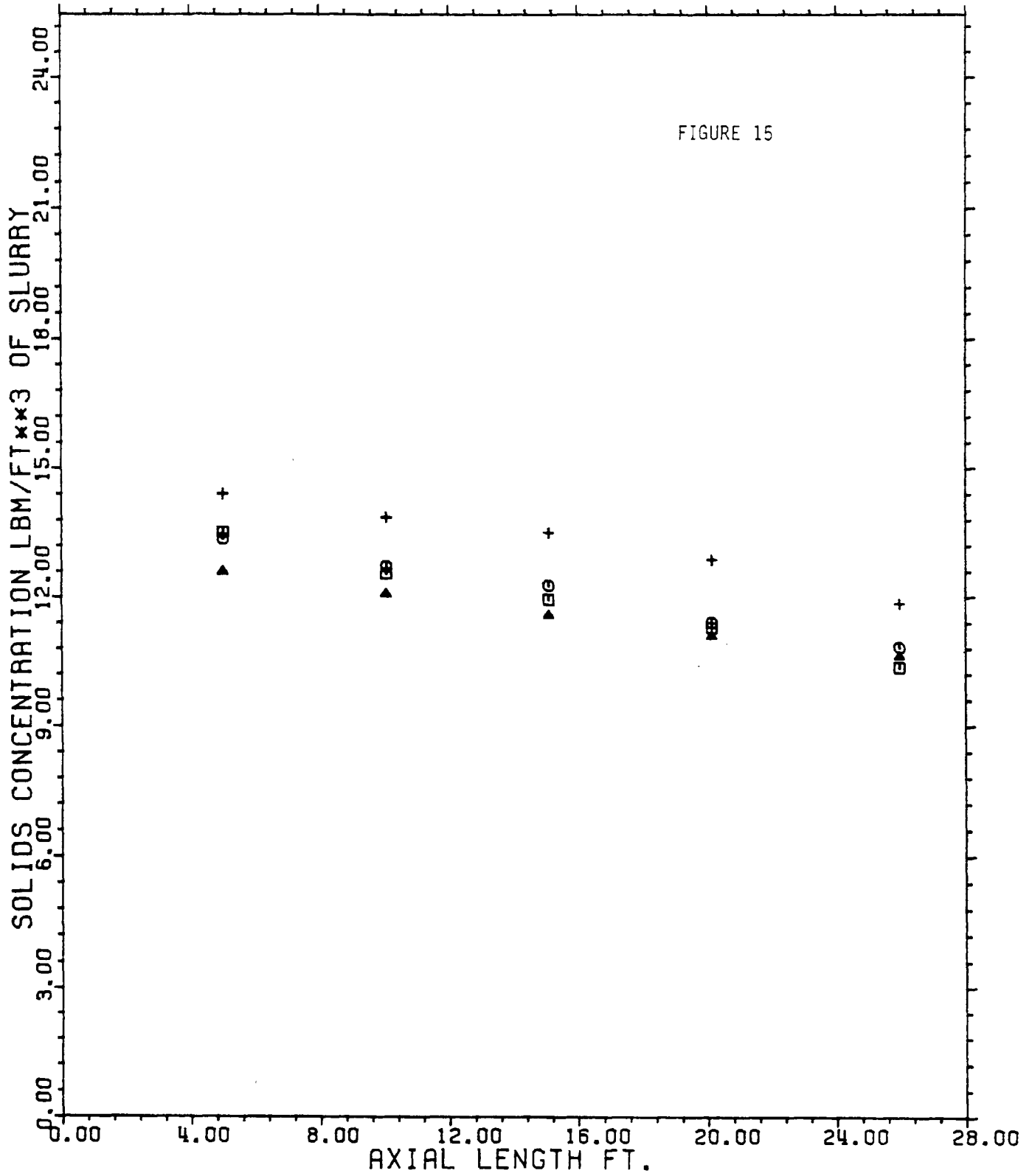
□ L=5.00 FT  
 ○ L=10.05 FT  
 △ L=15.09 FT  
 + L=20.13 FT  
 \* L=25.96 FT



<b>SOLIDS VARIATION WITH TIME</b>	
C VS. T IN A 12" COL. (RECYCLE) #60/80 MESH SAND VL=.05 FT/SEC, VG=.133 FT/SEC LIQUID USED IS TETRALIN	
□ L=5.00 FT ○ L=10.05 FT ▲ L=15.09 FT + L=20.13 FT * L=25.98 FT	24



SOLIDS CONC. VS. LENGTH	
C VS. L IN A 12" COL #140 MESH MINUS SAND (RECYCLE MODE) VL=.05 FT/SEC	
□ VG=.392 FT/SEC ⊙ VG=.308 FT/SEC △ VG=.216 FT/SEC	.25



SOLIDS CONC. VS. LENGTH

C VS. L IN A 12" COL (RECYCLE MODE)

#140 MINUS SAND

VG=.392 FT/SEC

□ VL=.02 FT/SEC  
 ◻ VL=.03 FT/SEC  
 ▲ VL=.04 FT/SEC  
 + VL=.05 FT/SEC

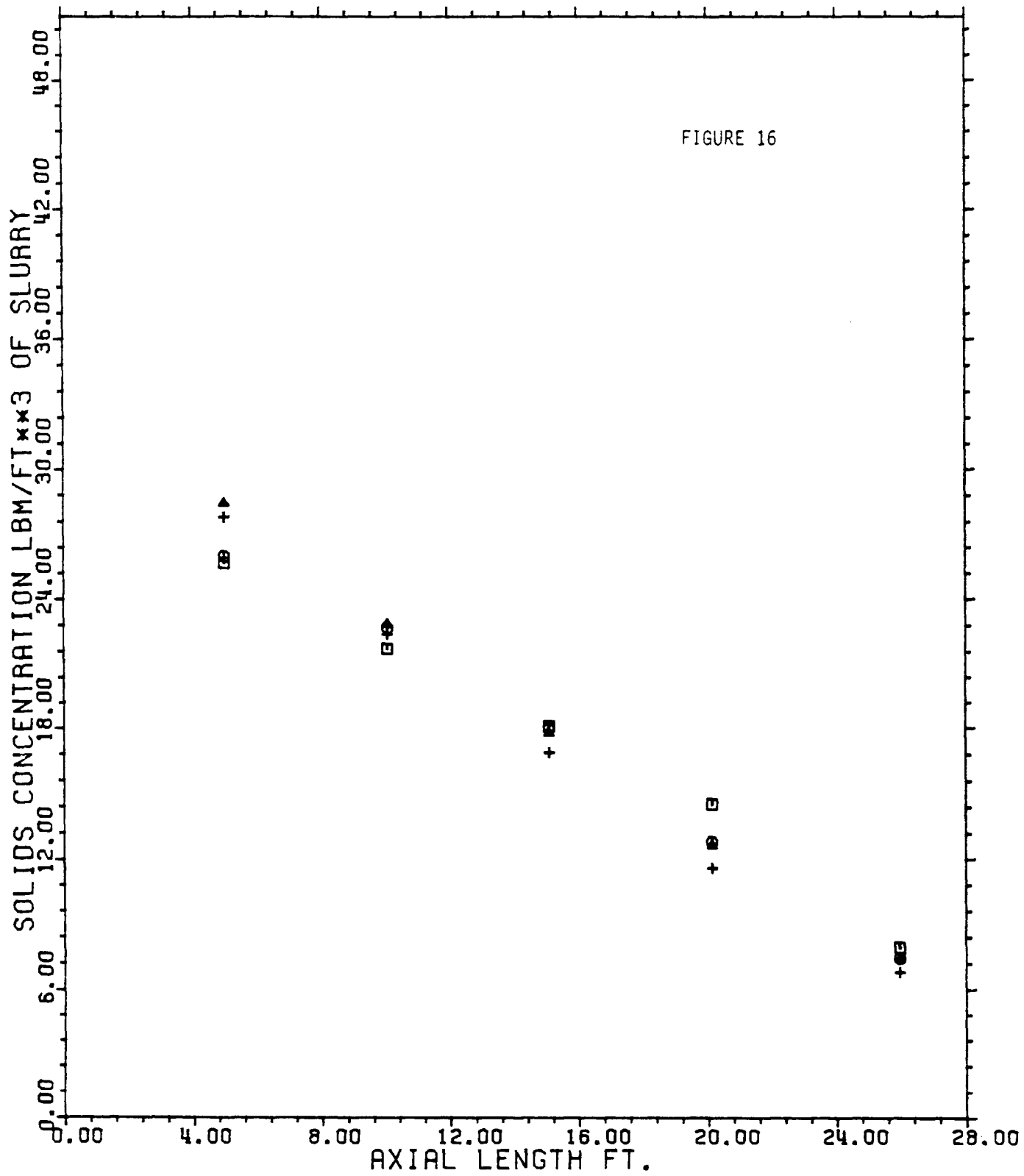


effects of gas and liquid velocities cannot be easily distinguished. However, careful inspection of the profiles shown in Figure (12) indicated no gas velocity effect on the distribution of fine particles. The effect of liquid velocity is not so obvious. Apparently the effect will be small, if there is any.

When larger particles (60/80 mesh sand) were used, steeper solid concentration profiles were observed as shown in Figures (16) and (17). These steeper profiles are primarily due to the higher settling velocities of the large particles. Again the gas velocity has negligible effect on the concentration profile. However, the effect of liquid velocity is quite definite; the solid accumulation increases with decreasing liquid velocity.

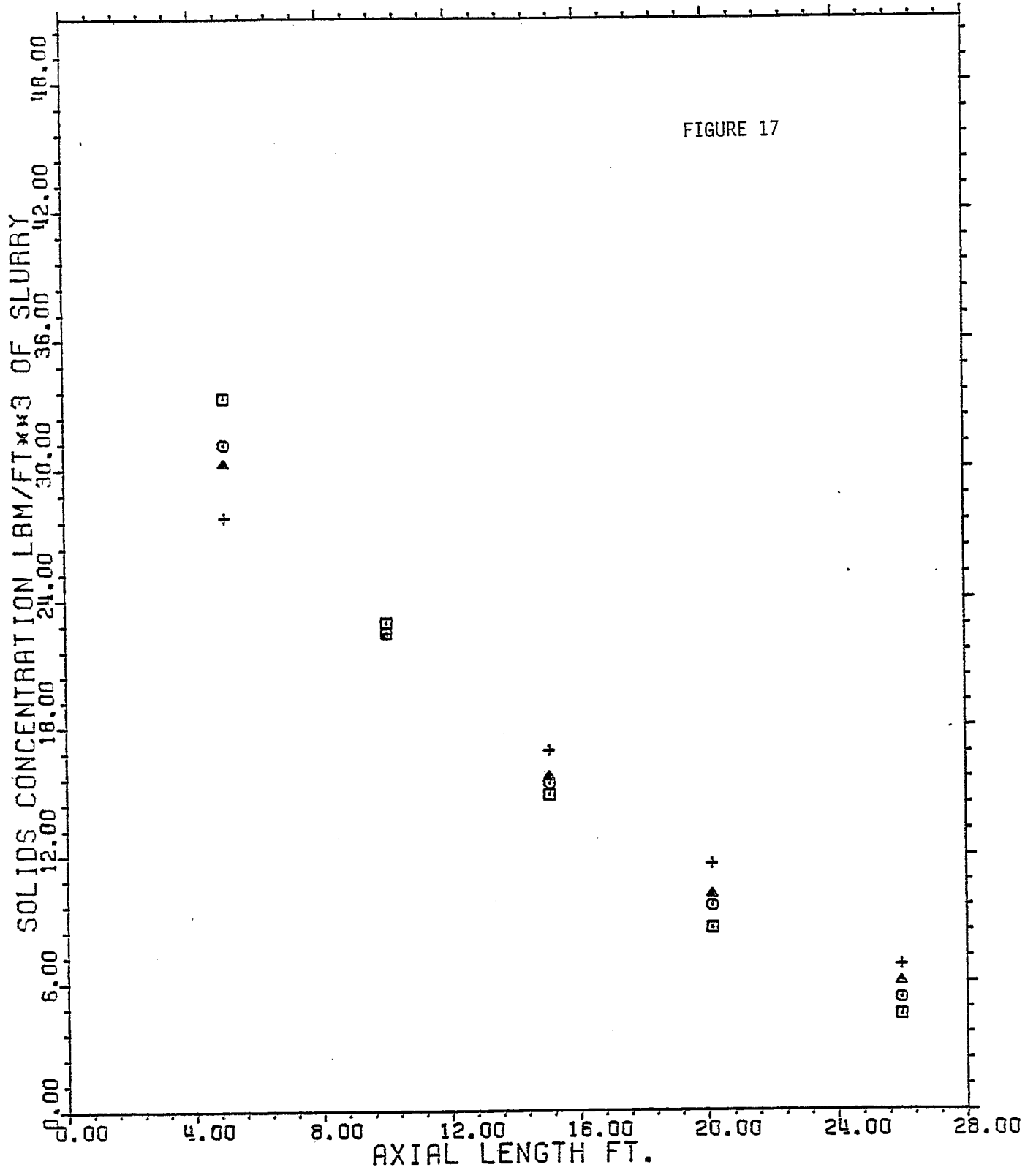
To quantify the difference in behavior between the 140 mesh and 60/80 mesh sand particles, a non-dimensional number is introduced as the ordinate due to the fact that in the recycle mode it is difficult to maintain the same feed concentration for all the runs. By dividing the average concentration by the feed concentration, a dimensionless number can be obtained to quantify the amount of solids retained in the column at the different gas and liquid velocities. The feed concentration is determined by measuring the average concentration in the mixing tanks. The average concentration of the large particles (60/80 mesh sand, which can settle out easily) was measured directly by draining the total content retained in the column at the end of each experiment. However, the fine particles (140 mesh minus sand) require a much longer time to settle out. Instead, the average concentration for the #140 mesh size was calculated by integrating the profile over the length of the column and dividing this by the actual volume of slurry in the column.

The effects of the liquid and gas velocities on solids accumulation for 140 mesh minus and 60/80 mesh sand particle sizes are shown in Figures (18) and (19), respectively. Apparently the liquid velocity effect on the accumulation of the #140 mesh sand was small if there is any shown in Figure (18). In the air/water/sand systems the effect of liquid velocity was more apparent; the solid accumulation

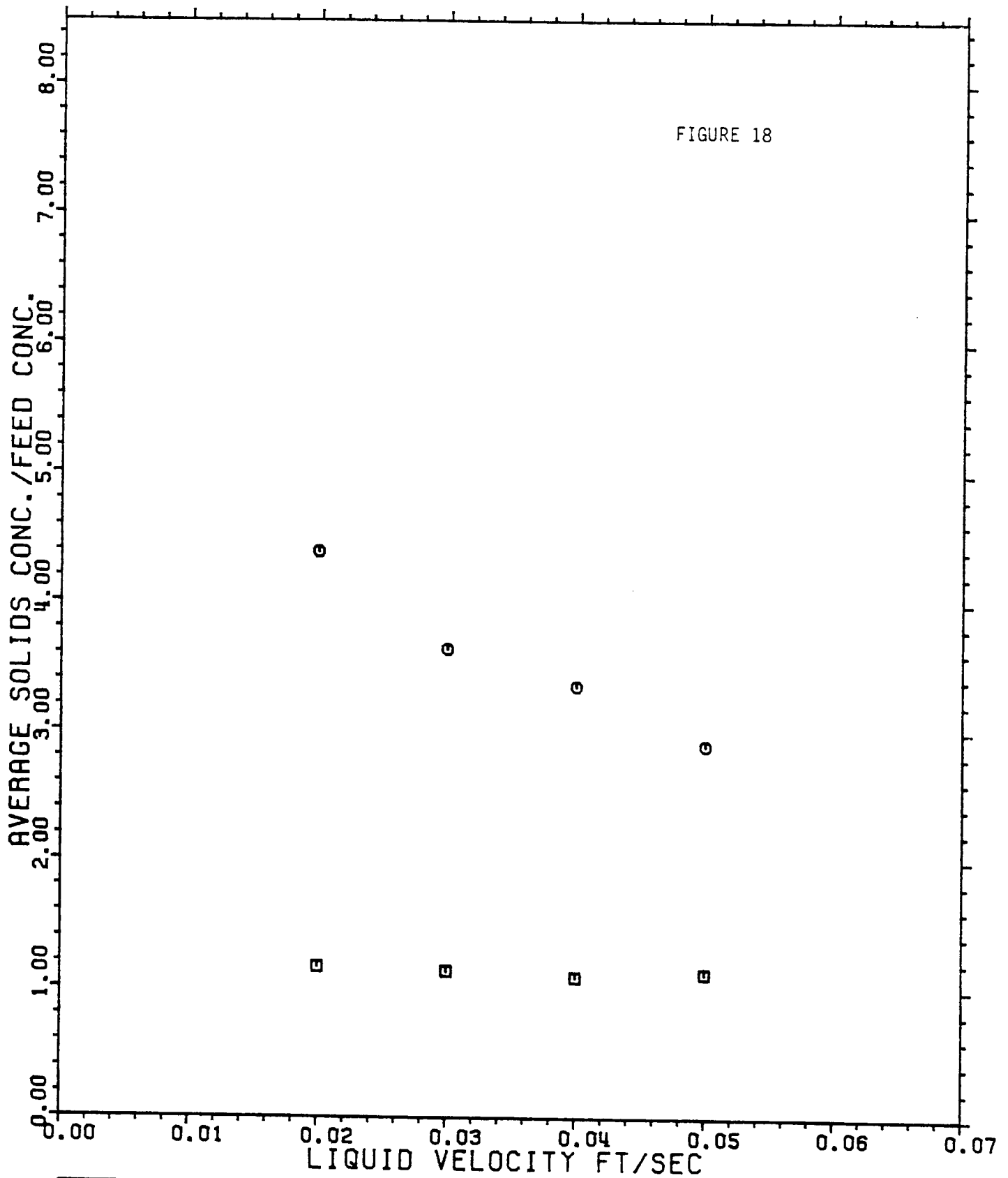


SOLIDS CONC. VS. LENGTH	
C VS. L IN A 12" COL #60/80 MESH SAND (RECYCLE MODE) VL=.05 FT/SEC	
□ VG=.133 FT/SEC ● VG=.218 FT/SEC ▲ VG=.308 FT/SEC + VG=.392 FT/SEC	28

FIGURE 17



SOLIDS CONC. VS. LENGTH									
<p style="text-align: center;">C VS. L IN A 12" COL                      *60/80 MESH SAND (RECYCLE MODE)                      VG=.392 FT/SEC</p>	<table style="width: 100%; border: none;"> <tr> <td style="padding: 2px;">■</td> <td style="padding: 2px;">VL=.02 FT/SEC</td> </tr> <tr> <td style="padding: 2px;">●</td> <td style="padding: 2px;">VL=.03 FT/SEC</td> </tr> <tr> <td style="padding: 2px;">▲</td> <td style="padding: 2px;">VL=.04 FT/SEC</td> </tr> <tr> <td style="padding: 2px;">+</td> <td style="padding: 2px;">VL=.05 FT/SEC</td> </tr> </table>	■	VL=.02 FT/SEC	●	VL=.03 FT/SEC	▲	VL=.04 FT/SEC	+	VL=.05 FT/SEC
■	VL=.02 FT/SEC								
●	VL=.03 FT/SEC								
▲	VL=.04 FT/SEC								
+	VL=.05 FT/SEC								
29									

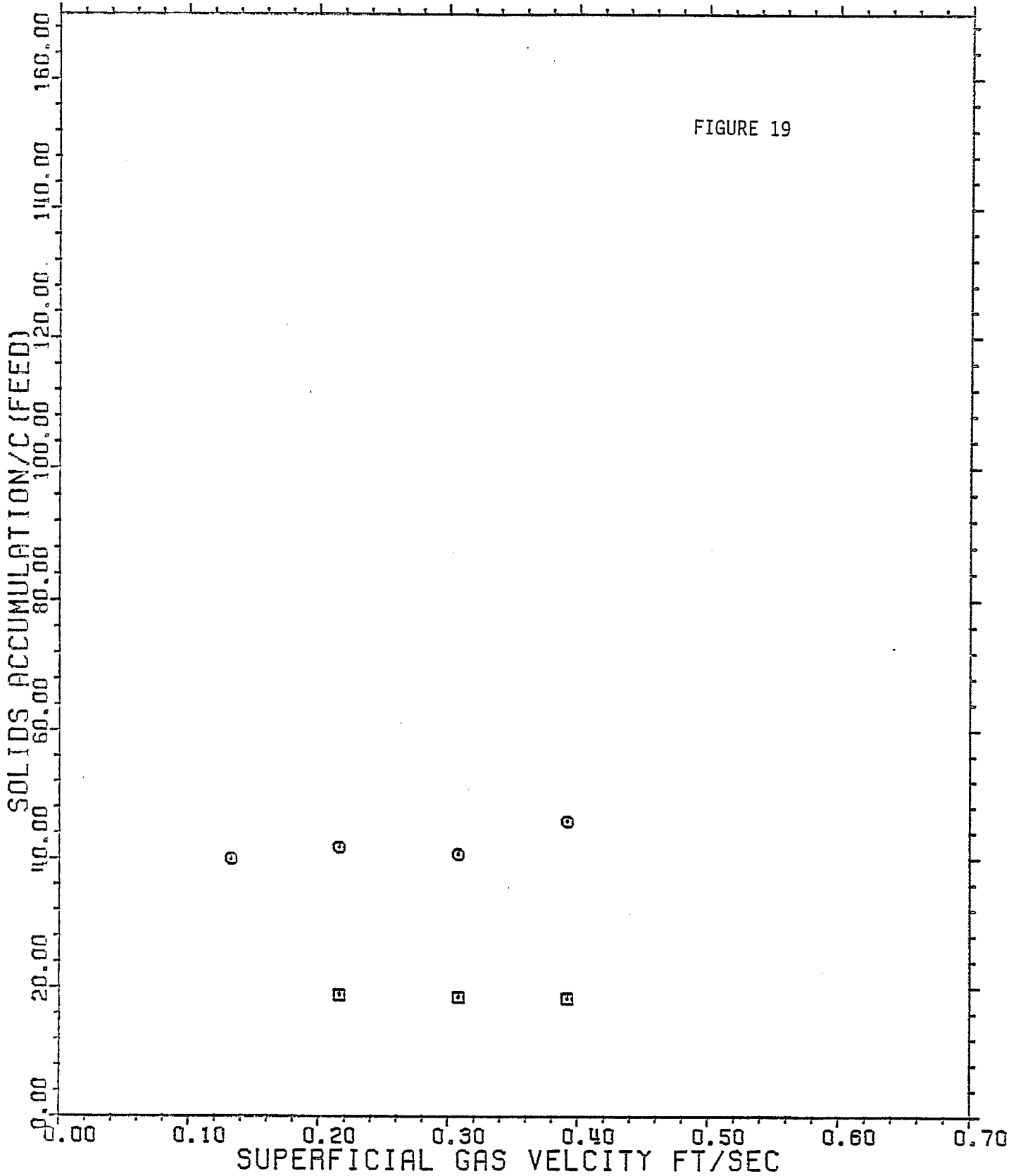


**EFFECT OF VL ON CAV/CF**

EFFECT OF VL ON SOLIDS ACCUMULATION

12" COLUMN, TETRALIN,  $V_G = .392$  FT/SEC

□ •140 MINUS MESH SAND  
 ○ 60/80 MESH SAND



SOLIDS ACCUMULATION	
EFFECT OF VG ON SOLIDS ACCUMULATION	□ #140 MINUS MESH SAND (TETRALIN) ○ #60/80 MESH SAND (VL=.05 FT/SEC)

increased with decreasing liquid velocity. This difference in liquid velocity effect on the fine particles is probably due to the higher viscosity in tetralin (tetralin 2.67 cps versus 1.00 cps water). The particle settling velocity is lower in a more viscous medium. Therefore the effect of liquid velocity in tetralin is less apparent. For the large particles (#60/80 mesh sand) the effect of liquid velocity in both water and tetralin follows the same trend, although the magnitude could be different. A decrease in the solids accumulation with an increase in liquid velocity is shown by Figure (18). It is interesting to note that the dependence on liquid velocity is linear similar to that observed earlier in the aqueous system.

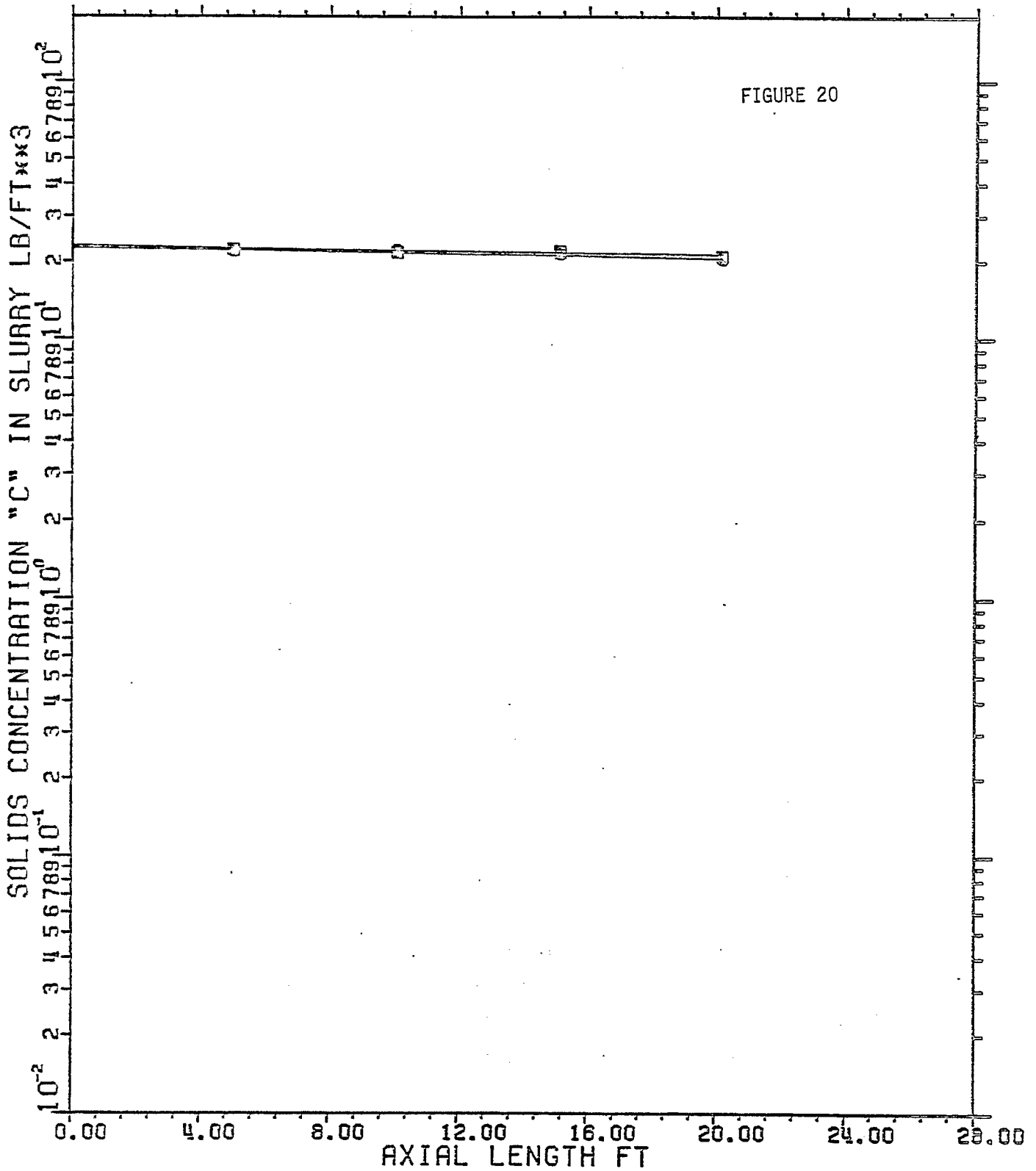
There is no gas velocity effect on the accumulation of both large (60/80 mesh) and fine (140 mesh minus) particles as shown in Figure (19). This result agrees with the earlier findings in the air/water/sand system.

#### 4.2.2 Solids Distribution in Ethylene Glycol-Water Solution (140 Minus Mesh and 60/80 Mesh Sand)

The solids concentration distribution in two different glycol-water solutions (90 wt% and 70 wt% glycol) was conducted in batch operation in the 12-inch column during this quarter using 140 mesh minus and 60/80 mesh sand.

The fine solids distribution (140 mesh minus sand) was similar to those observed in the aqueous and tetralin systems. The fine particles were nearly homogeneous well mixed as shown in Figures (20) and (21). The ratio of particle settling velocity to the dispersion coefficient ( $V_p/E_{zp}$ ) can be determined from the linear relationship of  $\ln C$  (concentration) versus axial length  $L$  plots as discussed in previous reports (TIC-14801-12). Table (3) shows the results of the  $V_p/E_{zp}$  obtained for the #140 minus mesh sand in two glycol-water solutions.

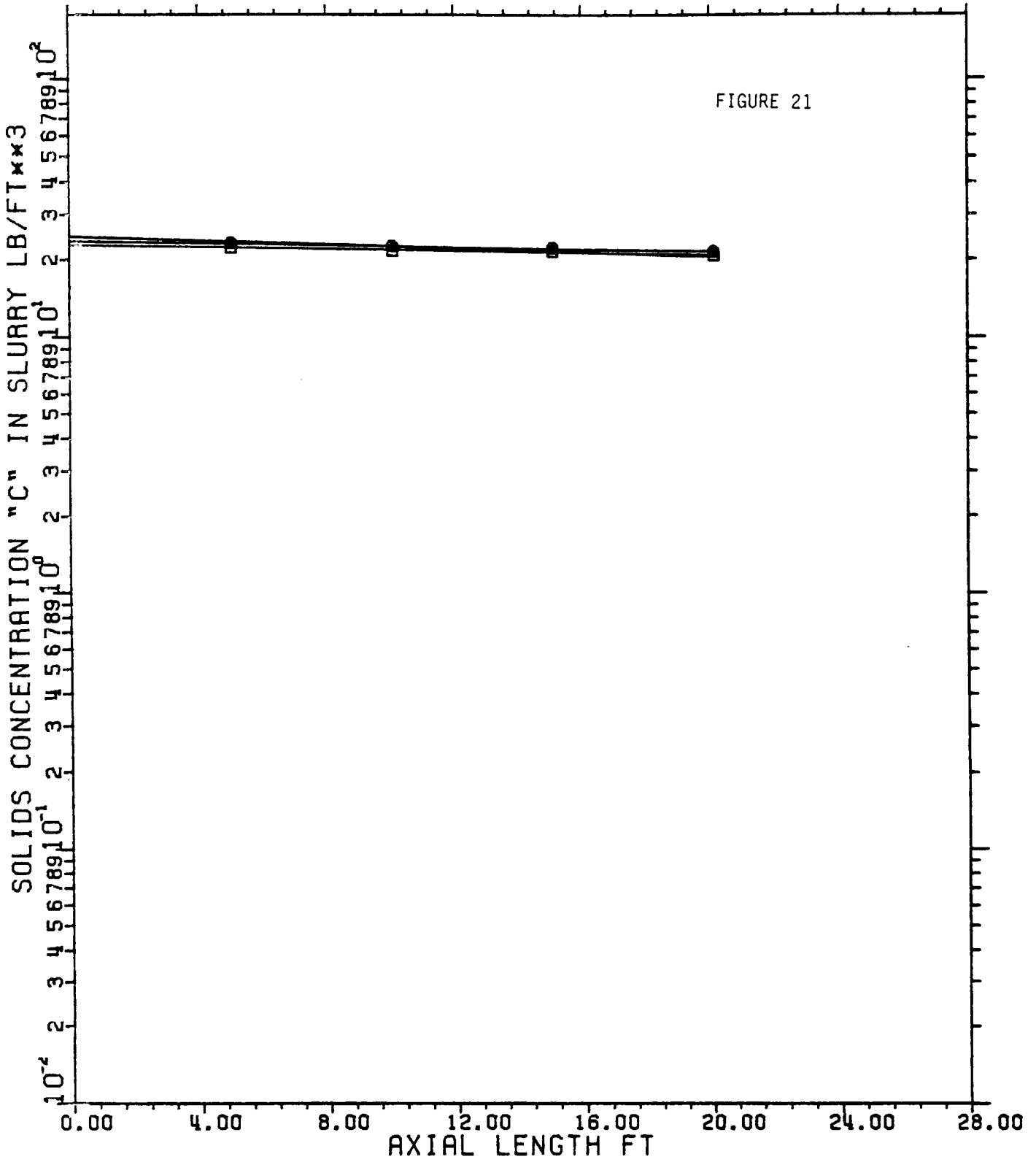
The  $V_p/E_{zp}$  values are slightly and consistently higher in the 70 wt% glycol solution. This slight difference is due to viscosity effect. The 70 wt% glycol has a lower viscosity as shown in Table (3).



EFFECT OF VG ON SOLIDS DISTR

SOLIDS DISTRIBUTION C VS AXIAL LENGTH  
 12" COLUH, 90% GLYCOL #140 MINUS MESH SAND

VG <sub>H</sub>	.043 FT/SEC,	CAVE=20.758 LB/FT
VG <sub>F</sub>	.216 FT/SEC,	CAVE=22.230 LB/FT
VG <sub>L</sub>	.392 FT/SEC,	CAVE=22.930 LB/FT



EFFECT OF VG ON SOLIDS DISTR

SOLIDS DISTRIBUTION C VS AXIAL LENGTH  
 12" COLUM, 70% GLYCOL #140 MINUS MESH SAND

- VG= .043 FT/SEC, CAVE=21.43 LB/FT\*\*3
- VG= .218 FT/SEC, CAVE=23.51 LB/FT\*\*3
- ▲ VG= .392 FT/SEC, CAVE=24.30 LB/FT\*\*3



Table 3

Values of  $V_p/E_{zp}$  for #140 Minus Mesh Sand in Glycol

<u>Gas Velocity ft/sec</u>	<u><math>V_p/E_{zp}</math> (90% glycol)</u>	<u><math>V_p/E_{zp}</math> (70% glycol)</u>
0.043	0.0030	0.0044
0.216	0.0043	0.0050
0.392	0.0040	0.0046
Viscosity (Cps)	13.08	6.20

Therefore the particle settling velocity in this liquid medium is higher, thereby resulting with a higher  $V_p/E_{zp}$  value. However, the differences shown in Table (3) between 70 wt% and 90 wt% glycol solutions are very small. Perhaps the  $E_{zp}$  values are also affected by liquid viscosity in the same direction as  $V_p$  is affected, so that the difference in  $V_p/E_{zp}$  values become small. Table (4) shows a comparison of different values of  $V_p/E_{zp}$  in water, tetralin and glycol. The table also indicates that there is a significant influence of viscosity on the values of  $V_p/E_{zp}$ .

The behavior of large size particles is different from fine particles. The concentration gradient is much steeper than that for fine particles as shown in Figures (22) and (23).

The particle settling velocity is expected to be much higher for the 60/80 mesh sand thereby resulting with higher values of  $V_p/E_{zp}$  ratio which characterizes the steepness of the profile of  $\ln c$  versus axial length plot. Table (5) summarizes the  $V_p/E_{zp}$  values for two different glycol-water solutions.

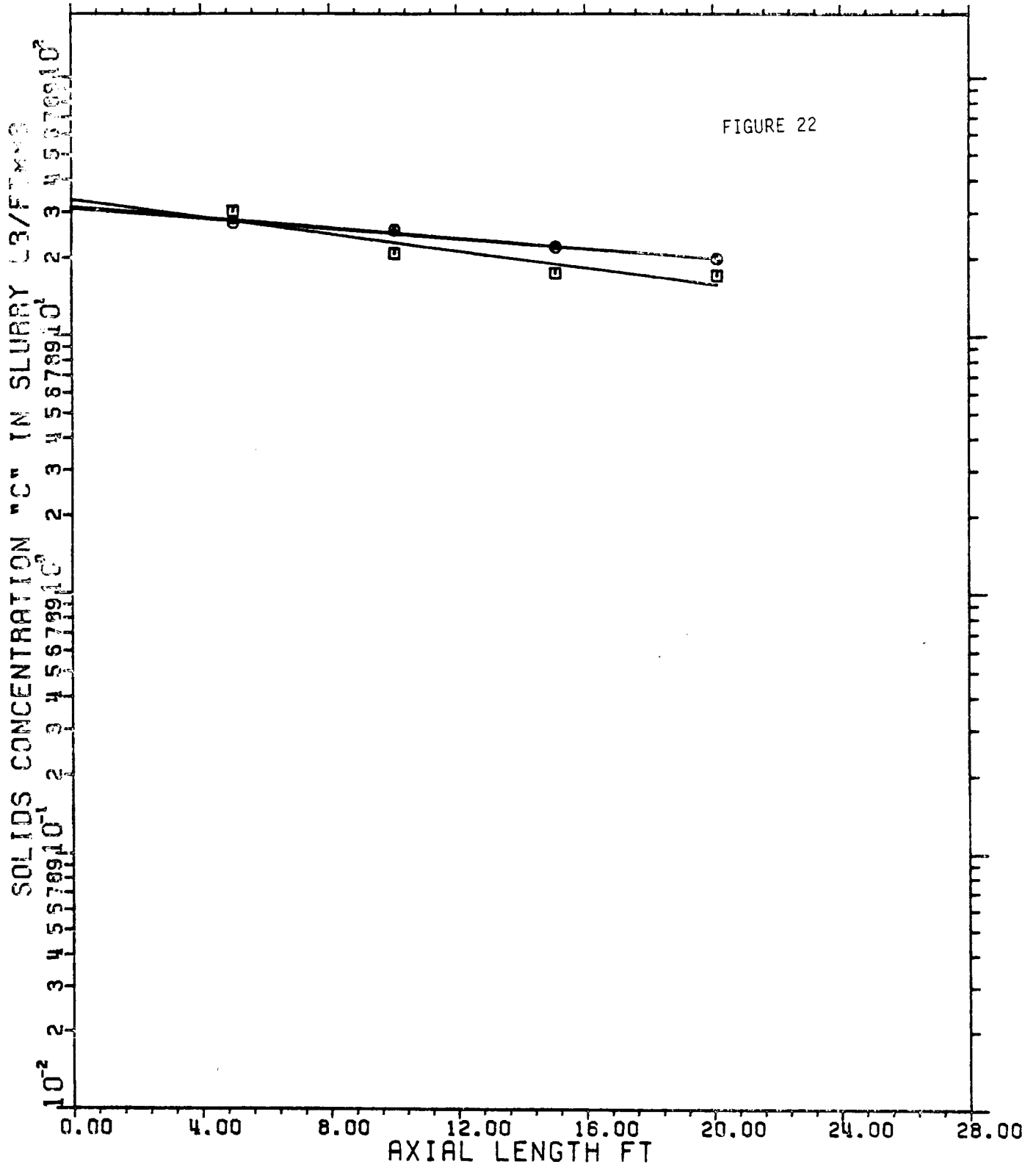
In Table 6 one notes rather higher values of  $V_p/E_{zp}$  at the low gas velocity of 0.043 ft/sec as opposed to the other gas velocities. At this low gas velocity complete suspension was not achieved thereby dramatically altering the values of  $V_p/E_{zp}$ . A better illustration is shown in Figures (22) and (23) which indicate an unusual profile at low gas velocity. Consistently the viscosity plays an important role in the  $V_p/E_{zp}$  value. The  $V_p$  of the large particles was dramatically reduced in a high viscous liquid (90 wt% glycol). Therefore the  $V_p/E_{zp}$  values were much lower in the 90 wt% glycol solution as shown in Table (5). Table (6) shows a comparison of the  $V_p/E_{zp}$  values for different liquids. Again the results show that with an increase in viscosity there is a decrease of  $V_p/E_{zp}$  due primarily to a decrease of  $V_p$ .

Table 4

Comparative Values of  $V_p/E_{zp}$  for the Fine Particles  
#140 Minus Mesh Size in the 12-inch Column for Different Gas Velocities

Concentration $C_s$ lb/ft <sup>3</sup>	water	tetralin	70% glycol	90% glycol
20.758-28.6	0.0050-0.0070	0.0031-0.0052	0.0044-0.0050	0.0030-0.0043
Viscosity (Cps)	1	2.67	6.20	13.08

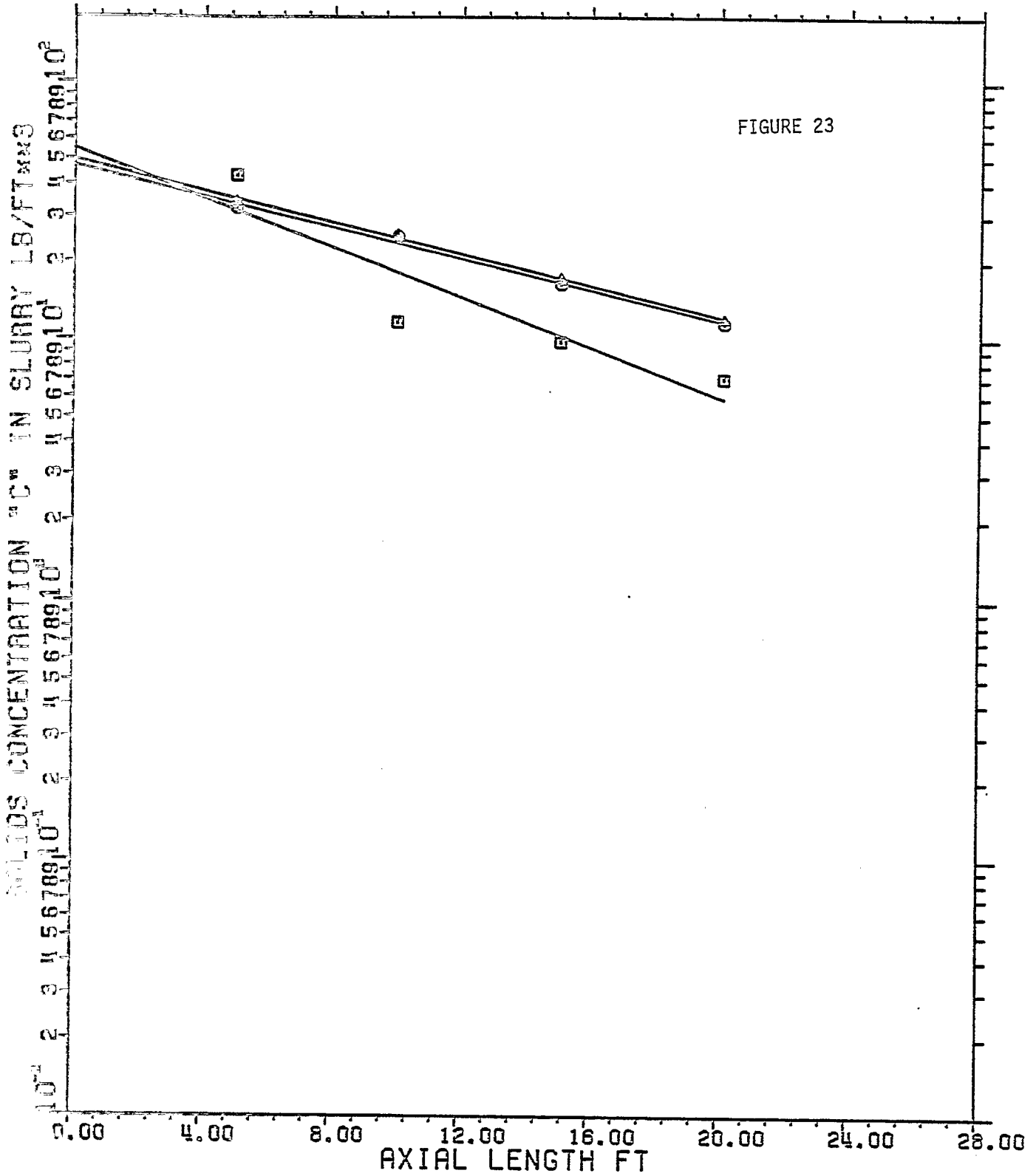
FIGURE 22



EFFECT OF VG ON SOLIDS DISTR

SOLIDS DISTRIBUTION C VS AXIAL LENGTH  
 12" COLUM, 90% GLYCOL #60/80 MESH SAND

□ VG= .043 FT/SEC, CAVE=23.079 LB/FT  
 ○ VG= .218 FT/SEC, CAVE=22.999 LB/FT  
 ▲ VG= .392 FT/SEC, CAVE=22.885 LB/FT



EFFECT OF VG ON SOLIDS DISTR

SOLIDS DISTRIBUTION C VS AXIAL LENGTH  
 12" COLUMN, 70% GLYCOL #60/80 MESH SAND

VG = .043 FT/SEC, CAVE=17.39 LB/FT\*  
 VG = .218 FT/SEC, CAVE=22.45 LB/FT\*  
 VG = .392 FT/SEC, CAVE=23.17 LB/FT\*

Table 5

$V_p/E_{zp}$  Results for 90% and 70% Glycol in the 12-inch  
Column using #60/80 Mesh Sand

Gas Velocity ft/sec	90% glycol	70% glycol
0.043	0.037	0.110
0.216	0.022	0.069
0.392	0.022	0.070
Viscosity Cps	13.08	6.20

Table 6

Comparative Values of  $V_p/E_{zp}$  for the Large Particles #60/80  
Mesh Sand in the 12-inch Column for Different Gas Velocities

Concentration $C_s$ lb/ft <sup>3</sup>	Water	Tetralin	70% Glycol	90% Glycol
17.39-28.66	0.145-0.195	0.0916-0.128	0.069-0.110	0.022-0.037

#### 4.2.3 SOLIDS DISTRIBUTION IN 50%-50% GLYCOL-WATER SOLUTION (60/80 MESH SAND)

Several experiments were conducted to study the axial solids distribution in a continuous recycle mode using 50% glycol solution. Data on the #60/80 mesh sand are presented here and the rest will be presented in the next quarterly.

Figures (24) through (26) show the data of the solids concentration plotted against axial height. Gas velocity does not seem to have a significant effect on solids distribution. This was shown to be true in the aqueous and tetralin slurries. However, liquid velocity has a definite effect on the distribution of solids. An increase in liquid velocity decreases the steepness of the profile due to the larger effect of convection in the system. This observation confirms results obtained using water and tetralin.

In general, the solids accumulation results show a higher solids retention in water followed by tetralin and then glycol. This would be expected since water is the least viscous of the three followed by tetralin and 50% glycol solution ( $\nu_L = 4.2$  cps). Due to the tendency to carry more solids through the column with a higher viscosity, less solids retained in 50% glycol solution is expected.

#### 4.3 AXIAL LIQUID DISPERSION

One experiment was performed in the 5-inch diameter column to measure the tetralin liquid axial dispersion coefficient in a batch operation. All other runs are made in the 12-inch diameter column to measure axial dispersion coefficients at different gas velocities and in the presence of solids. No liquid flow was employed in the measurement of the dispersion coefficient since it was established in the previous water data that liquid velocities do not affect the liquid dispersion coefficient.



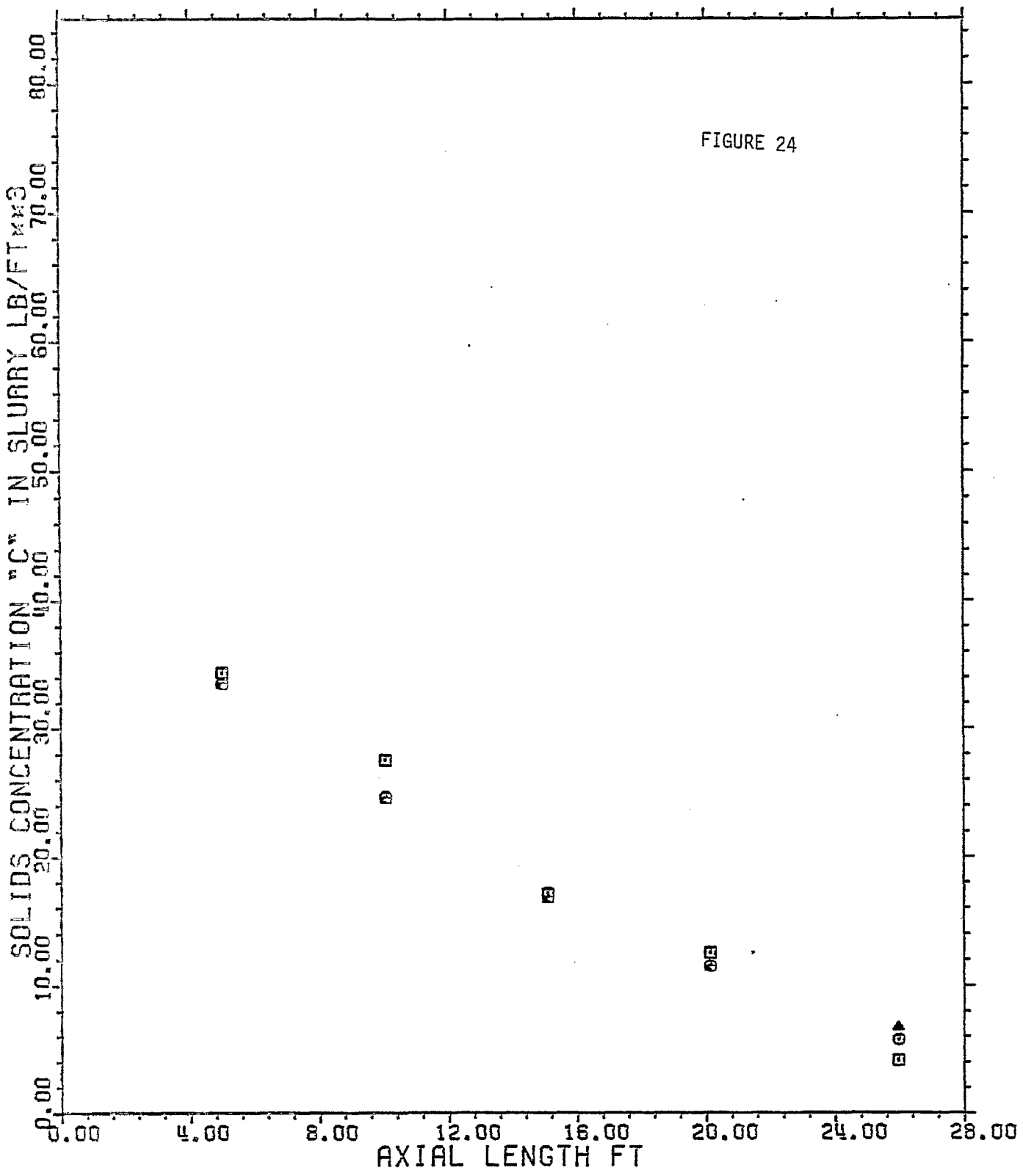
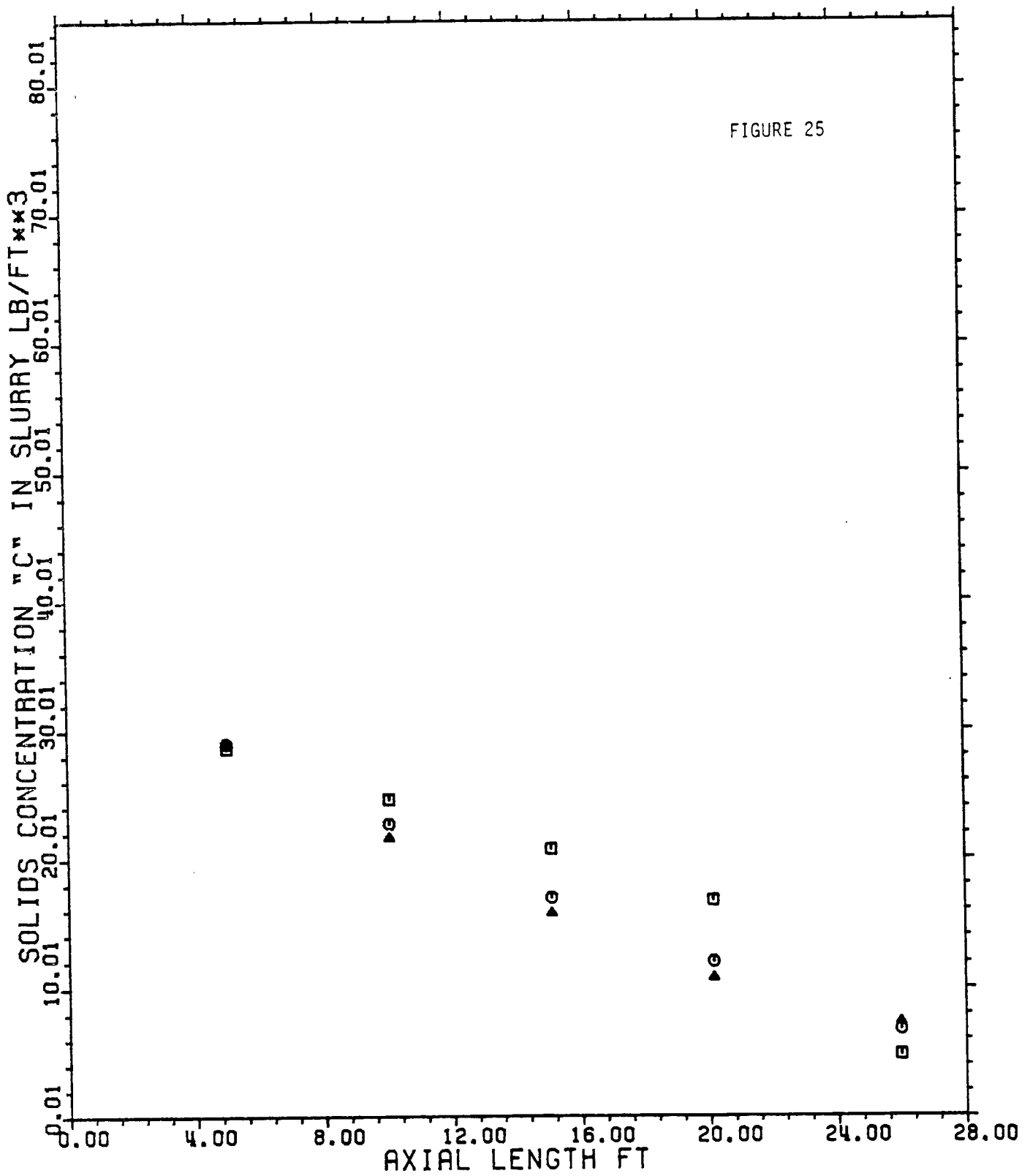


FIGURE 24

EFFECT OF VG ON SOLIDS DISTR

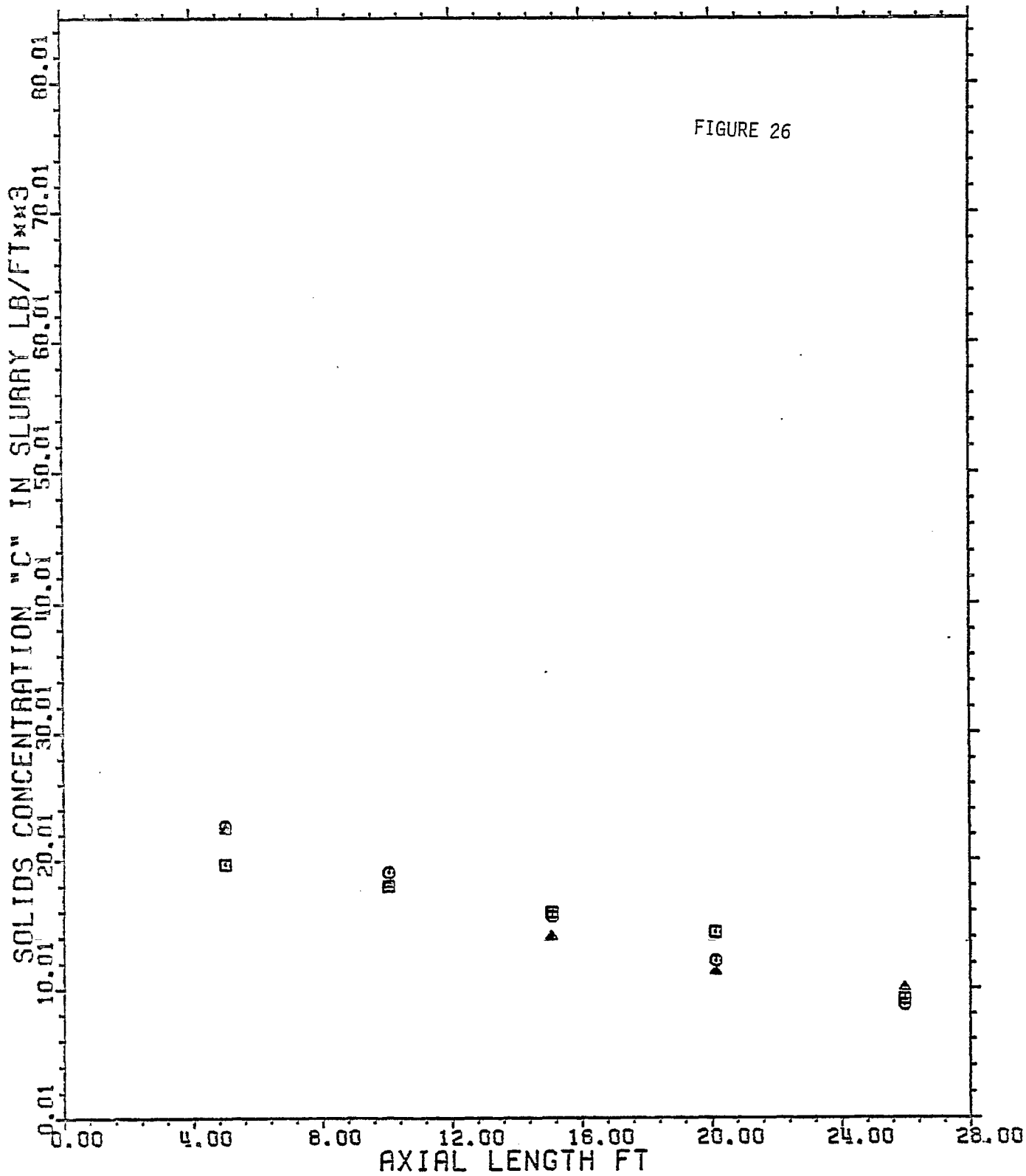
SOLIDS DISTRIBUTION C VS AXIAL LENGTH  
 12" COLUM, 50% GLYCOL #60/80 MESH SAND  
 VL=.01-.015 FT/SEC (RECYCLE)

- VG = .043 FT/SEC
- VG = .218 FT/SEC
- △ VG = .392 FT/SEC



EFFECT OF VG ON SOLIDS DISTR  
 SOLIDS DISTRIBUTION C VS AXIAL LENGTH  
 12" COLUM. 50% GLYCOL #60/90 MESH SAND  
 VL=.03 FT/SEC (RECYCLE)

□ VG= .050 FT/SEC  
 ○ VG= .197 FT/SEC  
 ▲ VG= .392 FT/SEC



**EFFECT OF VG ON SOLIDS DISTR**

SOLIDS DISTRIBUTION C VS AXIAL LENGTH  
 12" COLUMN, 50% GLYCOL #60/80 MESH SAND

VL = .05 FT/SEC (RECYCLE)

□ VG = .043 FT/SEC  
 ○ VG = .197 FT/SEC  
 △ VG = .392 FT/SEC

#### 4.3.1 THEORETICAL BACKGROUND

A basic one dimensional diffusion model was used in treating the data obtained. The equation is as follows according to Yoshihiro Ohki et al (2):

$$\frac{\partial c}{\partial t} = E_{zL} \frac{\partial^2 c}{\partial z^2} \quad (\text{Equation 1})$$

- c - tracer concentration
- z - axial distance
- $E_{zL}$  - liquid axial dispersion coefficient

Assuming an impulse injection, the approximate solution is obtained as follows:

$$C' = 1 + 2 \sum_{n=1}^{\infty} \left[ \left( \cos \frac{\pi n Z}{L} \right) \times \exp \left( -n \frac{2\pi^2}{L^2} E_{zL} t \right) \right] \quad (\text{Equation 2})$$

- $C'$  - non dimensionalized tracer concentration
- L - total length of aerated liquid

This approximate solution is within 1% of the complete analytical solution. Equation (2) was used to obtain the value of  $E_{zL}$ . Hence, by obtaining  $Z/L$  at a particular sampling site and determining  $C'$  as a function of  $t$  from the experiment,  $E_{zL}$  can be calculated. Ideally one point on the curve of  $C'$  and  $t$  should be sufficient to determine  $E_{zL}$ . In actuality three points were selected in the range of  $C'$  between 0.3 and 0.8 from which three values of  $E_{zL}$  were calculated. The average  $E_{zL}$  was reported for that particular gas velocity. A separate method was used to determine  $E_{zL}$  and to compare with the average value obtained from the technique described above. This method involves a least square fit program to optimize the value of  $E_{zL}$  to best fit the entire tracer curve. A comparison of these two methods will be discussed later.

#### 4.3.2 AXIAL LIQUID DISPERSION RESULTS (TETRALIN)

Figure (27) shows the result of two experimental runs in the 5-inch column. The aim was to evaluate  $E_{zL}$  for that gas velocity and to examine any entrance effects regarding whether the injection point was made from the top of the column and sampled from the bottom above the distributor or vice versa. Sample ports A and D referenced in Figure (27) were 0.33 ft and 3.66 ft above the distributor plate, respectively. It is shown from the Figure (27) that the position of the injection port is immaterial.

Table (8) shows a comparison between the calculated average values (three points) and those with the least square fit. The close agreement between the two sets of data suggests that the adequacy of using either method to calculate the liquid dispersion coefficient in batch operation.

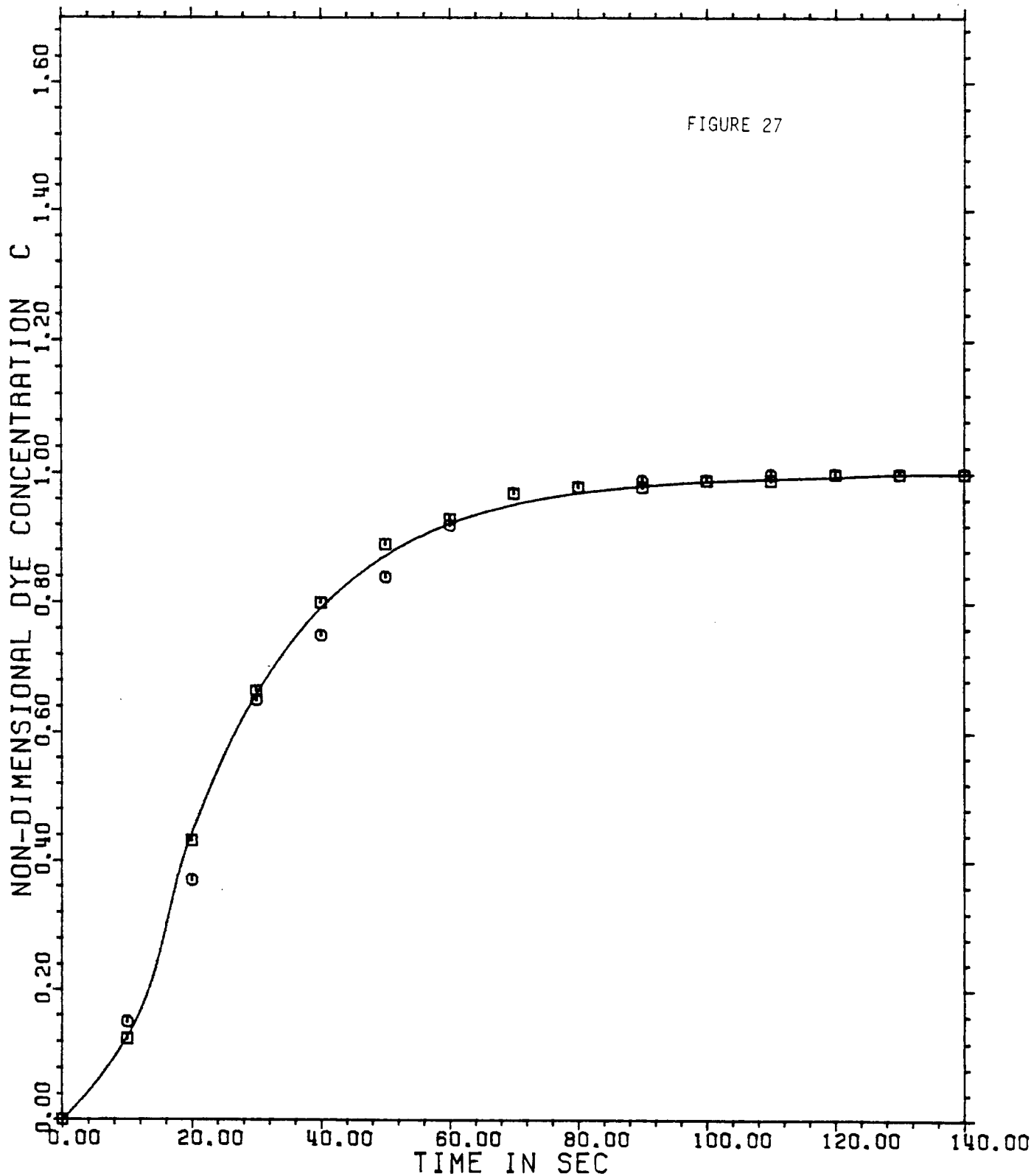
The axial dispersion coefficients for the 12-inch column are shown in Figures (28) through (35) and compared with their fitted model using the average value of the  $E_{zL}$ .

Figure (36) shows a plot of liquid dispersion coefficient ( $E_{zL}$ ) versus gas superficial velocity ( $V_g$ ) for the 12 inch column. The dispersion coefficient is shown to increase with gas velocities. This result is in conformity with our previous air/water results in the 5-inch and 12-inch diameter columns. A more detailed discussion of this behavior will be presented next quarter.

#### 5.0 Future Work

Complete data on solids distribution in a recycle mode for glycol-water solution with liquid flow will be obtained.

The Final Report which will unify all of the results experimentally will attempt to bring out the important features of the behavior of bubbly columns under the different conditions that were tested.



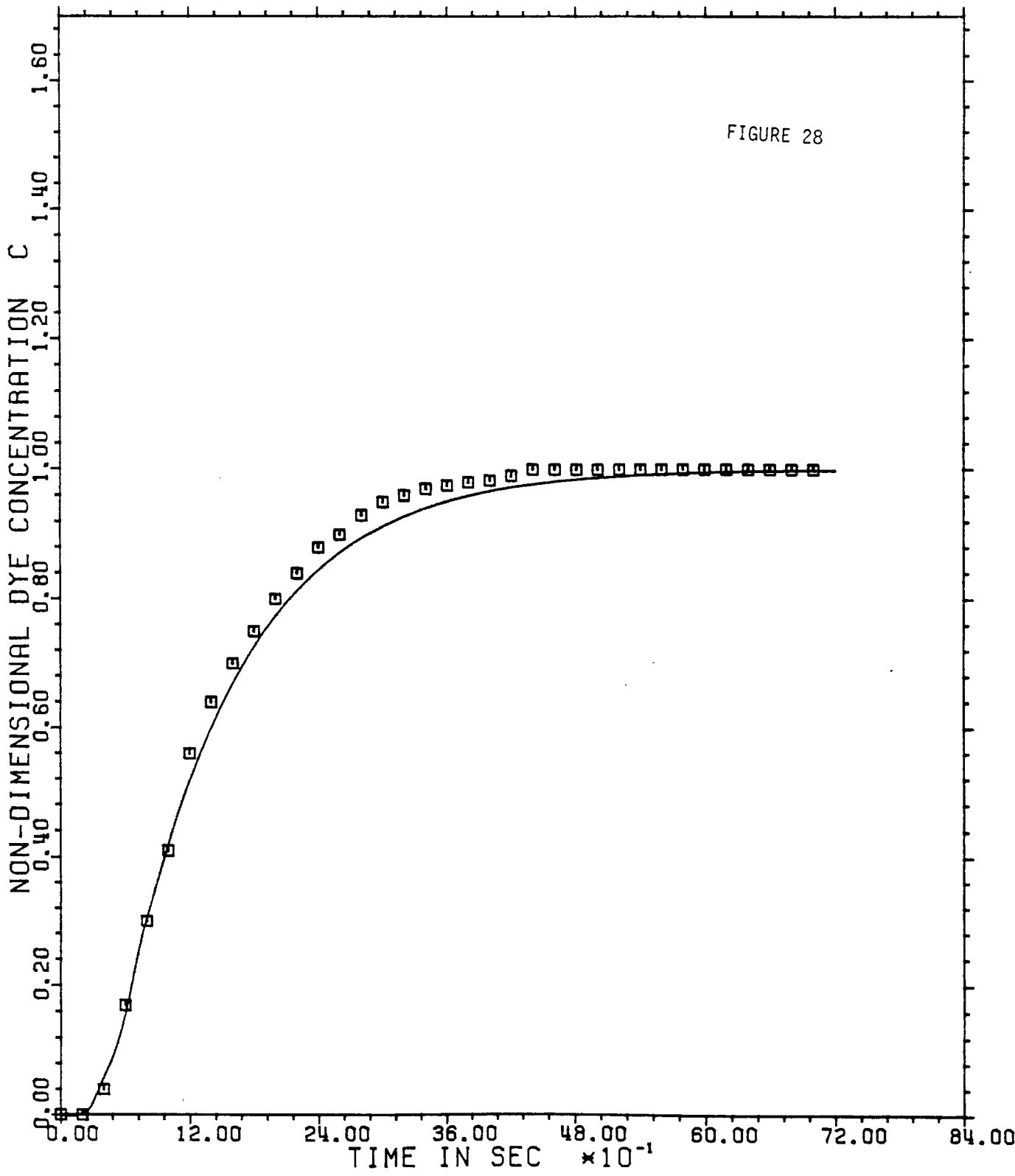
LIQUID DISPERSION MODEL	
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS.	
VG=.05 FT/SEC. EL=.093 FT <sup>2</sup> /SEC	
5" COLUMN AND TETRALIN USED (BATCH)	
48	□ EXPERIMENTAL DATA INJECT AT A SAMP ○ EXPERIMENTAL DATA INJECT AT D SAMP — MODEL PREDICTION EL=.093 FT <sup>2</sup> /SEC

Table 8

Comparison of  $E_{zL}$  by Averaging and Least Square Fitting Technique

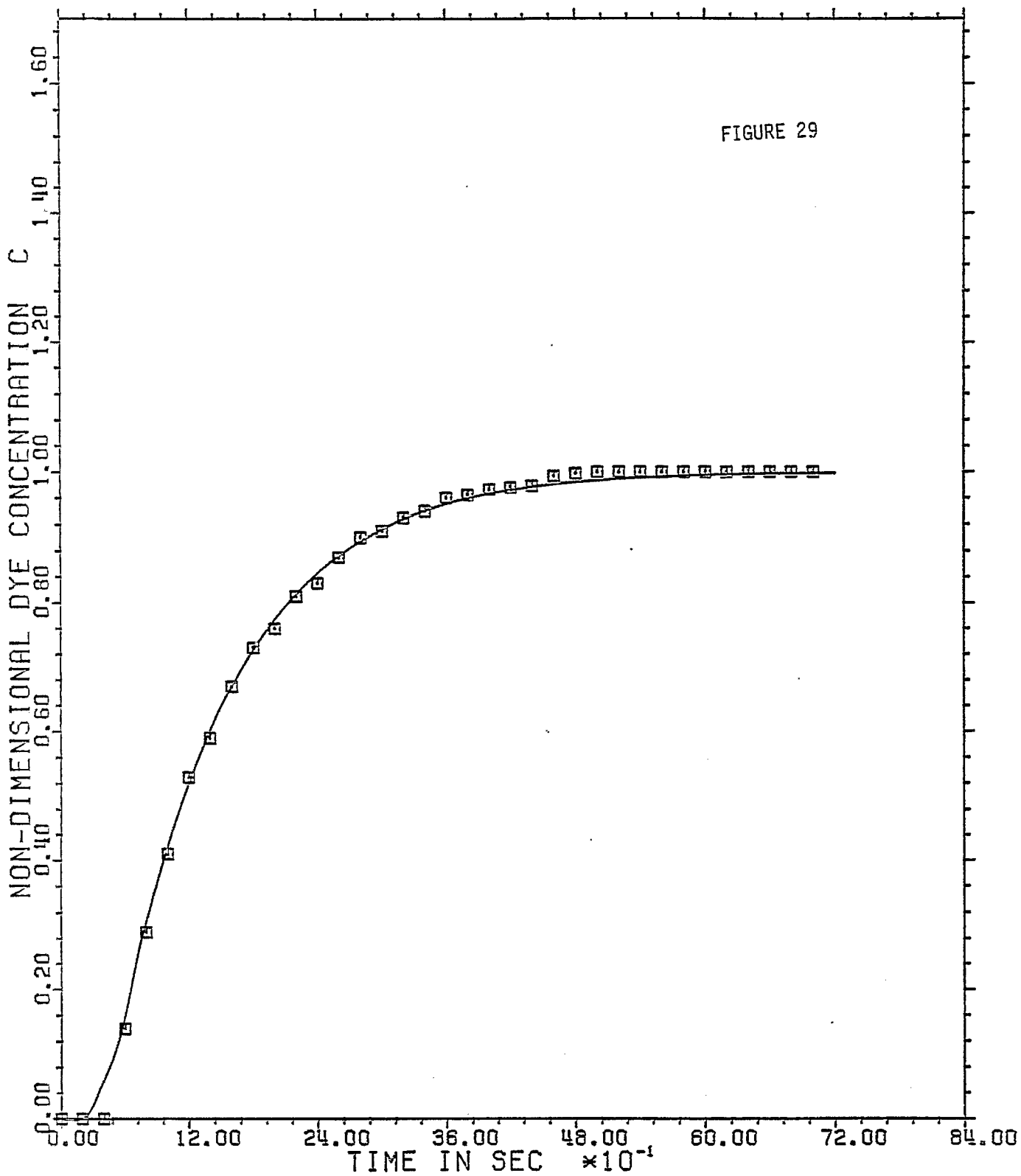
Column Diameter (inches)	$V_g$ (ft/sec)	$E_{zL}$ (least sq.)	$E_{zL}$ (Averaged)	Remarks
5	0.050	0.094	0.093	samples at D
5	0.050	0.088	0.093	sampled at A
12	0.043	0.450	0.450	
12	0.133	0.687	0.650	
12	0.176	0.641	0.651	
12	0.216	0.639	0.651	
12	0.267	0.689	0.641	
12	0.356	0.739	--	averaging was not done
12	0.392	0.864	0.850	no solids
12	0.392	0.781	0.790	#140 mesh sand $C_2 = 5 \text{ lb/ft}^3$
12	0.392	0.723	0.700	#140 mesh sand $C_s = 22 \text{ lb/ft}^3$

FIGURE 28



<b>LIQUID DISPERSION MODEL</b>	
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS. VG=.133 FT/SEC, EL=.650 FT**2/SEC 12" COLUM AND TETRALIN USED (BATCH) 50	□ EXPERIMENTAL DATA — MODEL PREDICTION





LIQUID DISPERSION MODEL

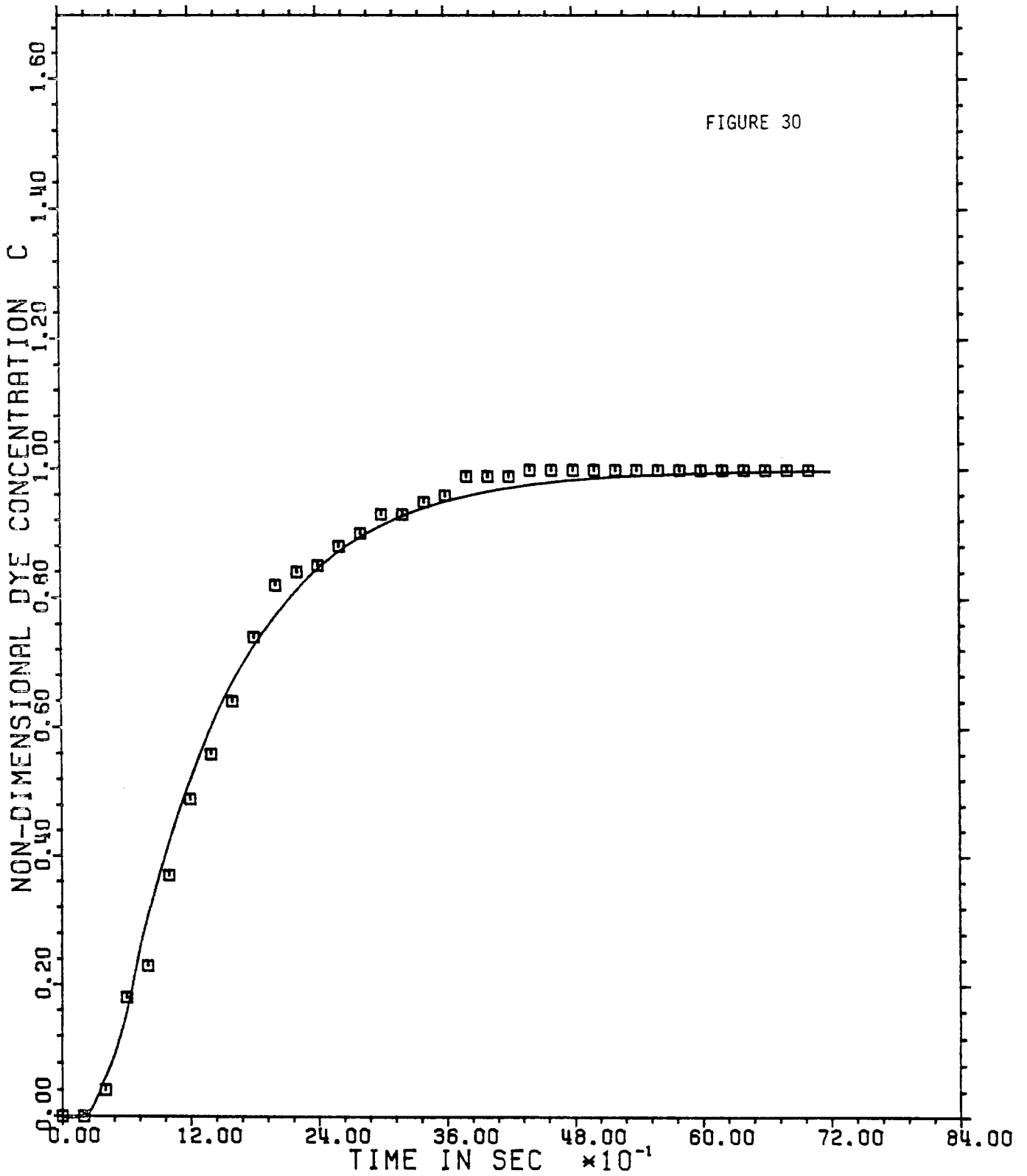
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS.

VG=0.176 FT/SEC, EL=0.651 FT<sup>2</sup>/SEC

12" COLUMN AND TETRALIN USED (BATCH)

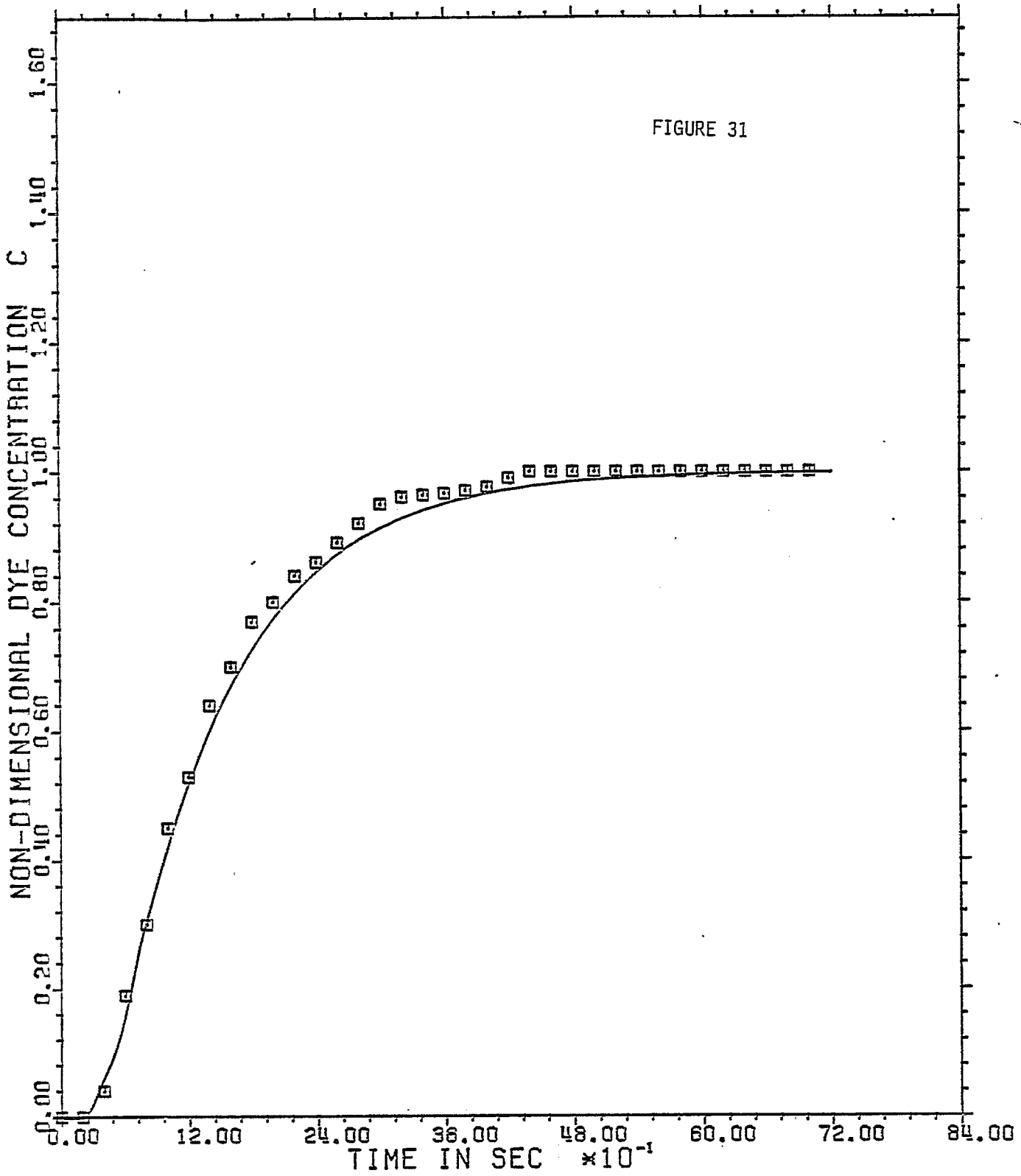
□ EXPERIMENTAL DATA  
 — MODEL PREDICTION

FIGURE 30



<b>LIQUID DISPERSION MODEL</b>	
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS. VG=.218 FT/SEC, EL=.651 FT <sup>2</sup> /SEC 12" COLUMN AND TETRALIN USED (BATCH)	
$\square$ EXPERIMENTAL DATA	— MODEL PREDICTION

FIGURE 31

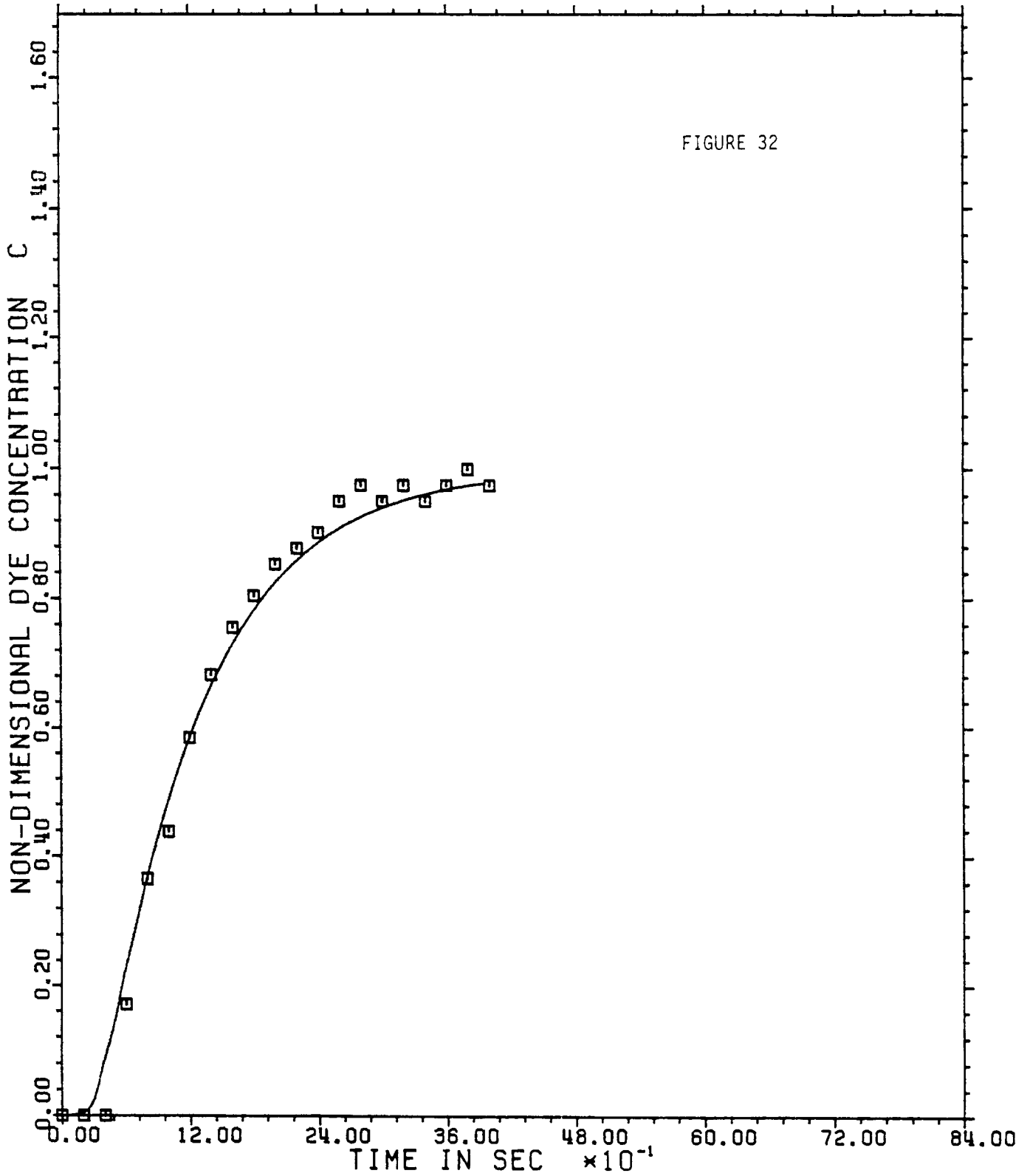


LIQUID DISPERSION MODEL

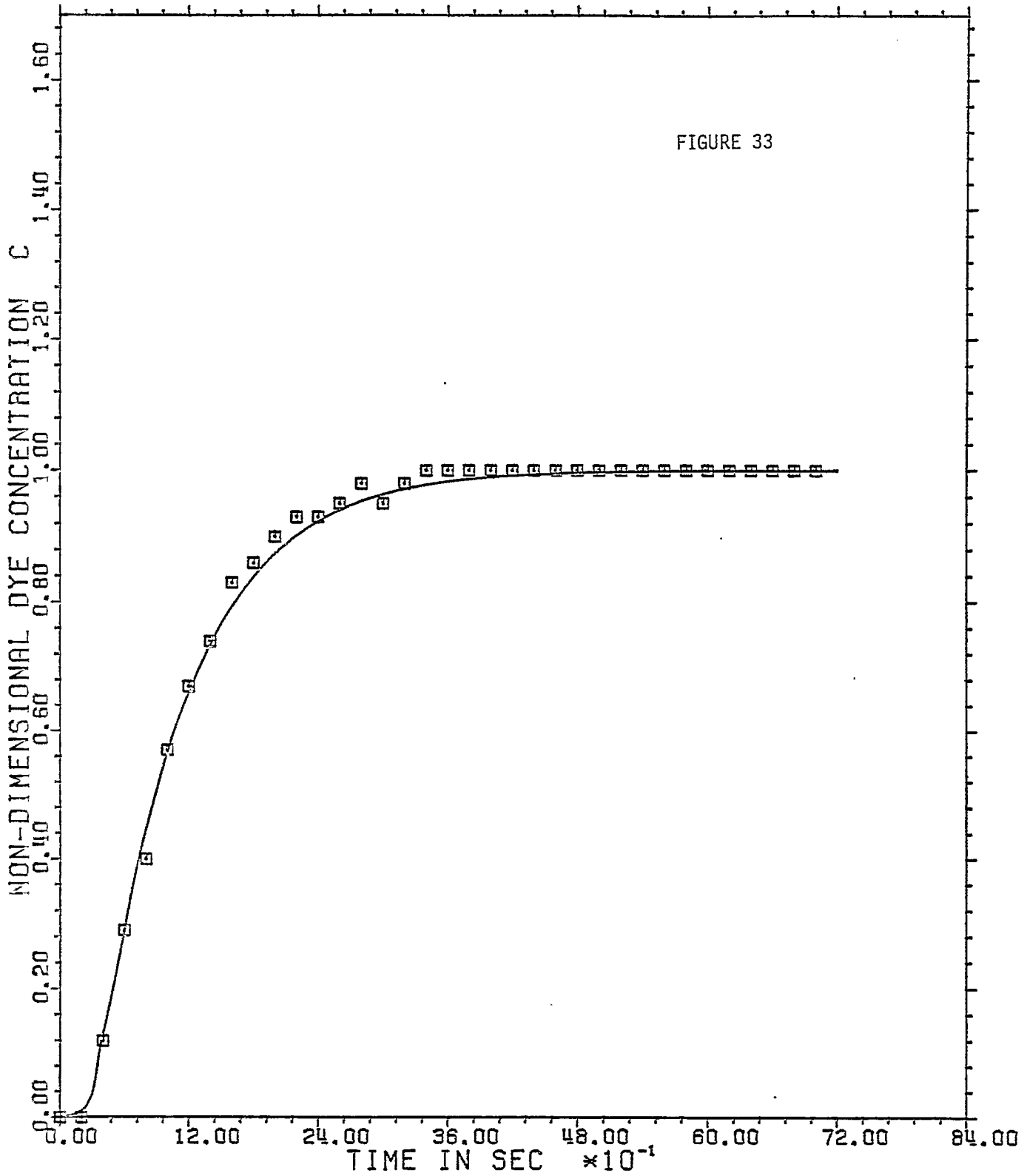
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS.  
 VG=.267 FT/SEC, EL=.651 FT $\times$ 2/SEC  
 12" COLUMN AND TETRALIN USED (BATCH)

□ EXPERIMENTAL DATA  
 — MODEL PREDICTION

FIGURE 32

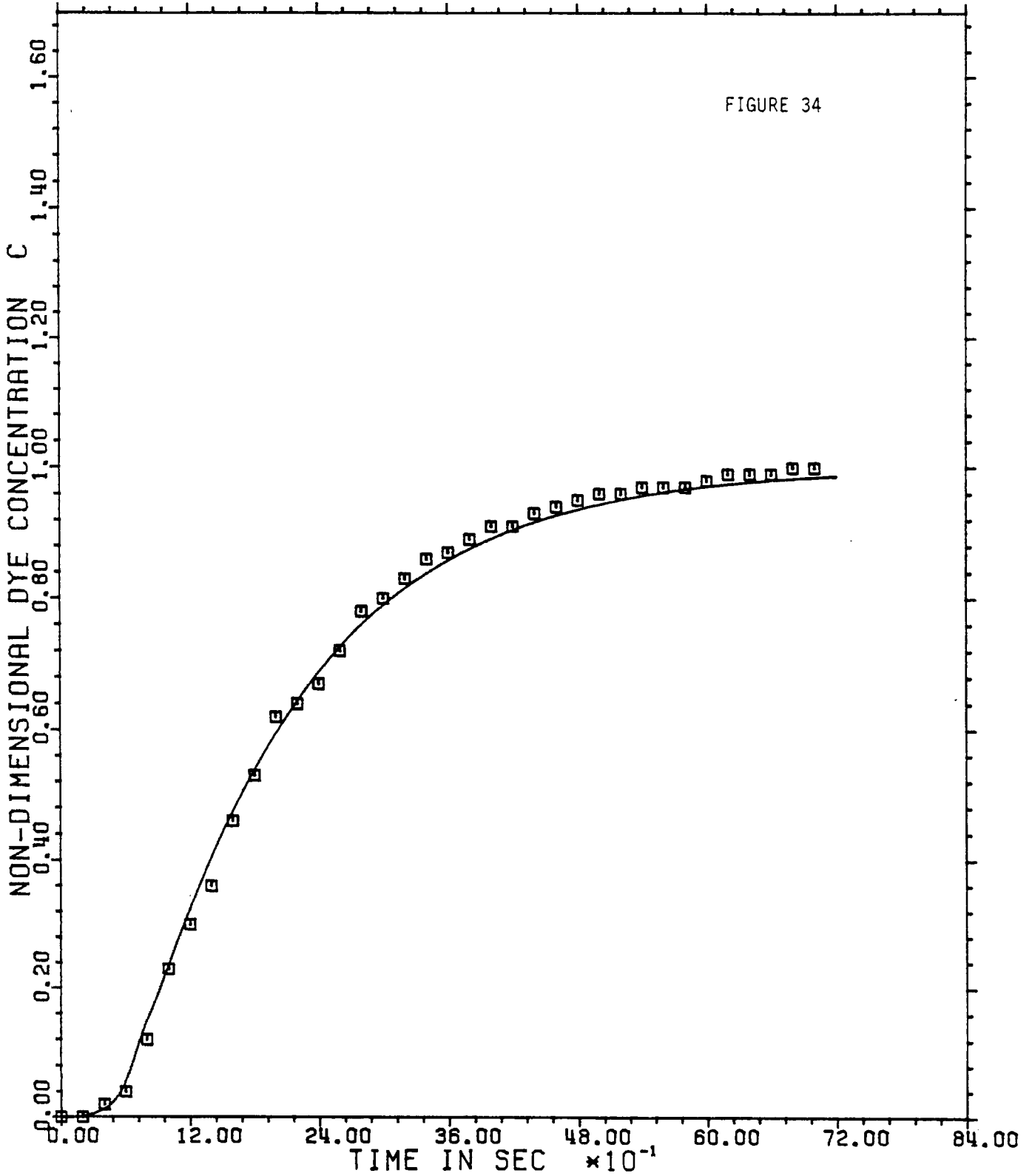


<b>LIQUID DISPERSION MODEL</b>	
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS. VG=.358 FT/SEC. EL=.739 FT**2/SEC 12" COLUM AND TETRALIN USED (BATCH)	
54	□ EXPERIMENTAL DATA — MODEL PREDICTION



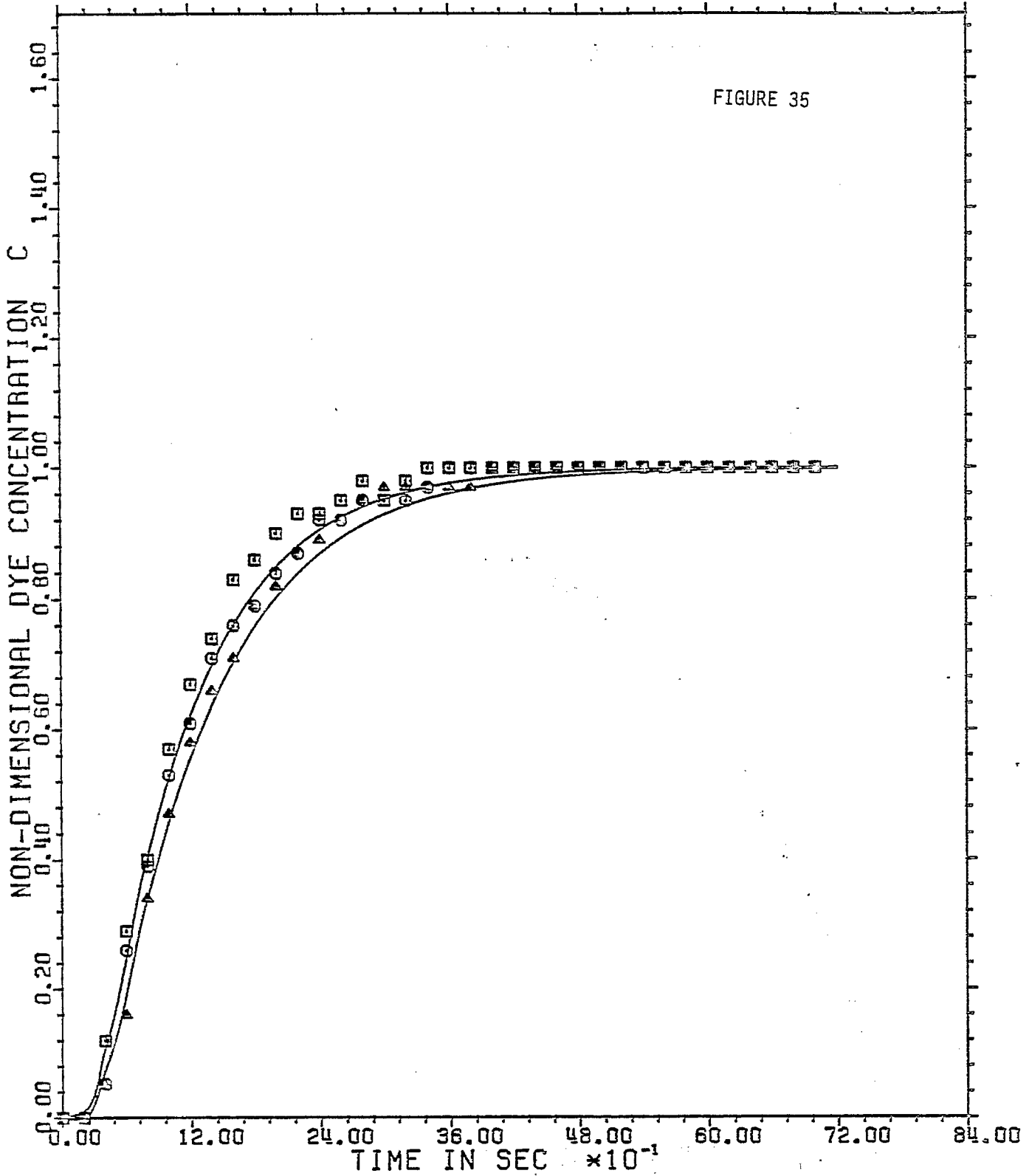
<b>LIQUID DISPERSION MODEL</b>	
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS. VG=.392 FT/SEC, EL=.850 FT <sup>2</sup> /SEC 12" COLUMN AND TETRALIN USED (BATCH)	
55	□ EXPERIMENTAL DATA — MODEL PREDICTION

FIGURE 34



<b>LIQUID DISPERSION MODEL</b>	
COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS. VG = .043 FT/SEC, EL = .450 FT <sup>2</sup> /SEC 12" COLUMN AND TETRALIN USED (BATCH)	□ EXPERIMENTAL DATA — MODEL PREDICTION

FIGURE 35



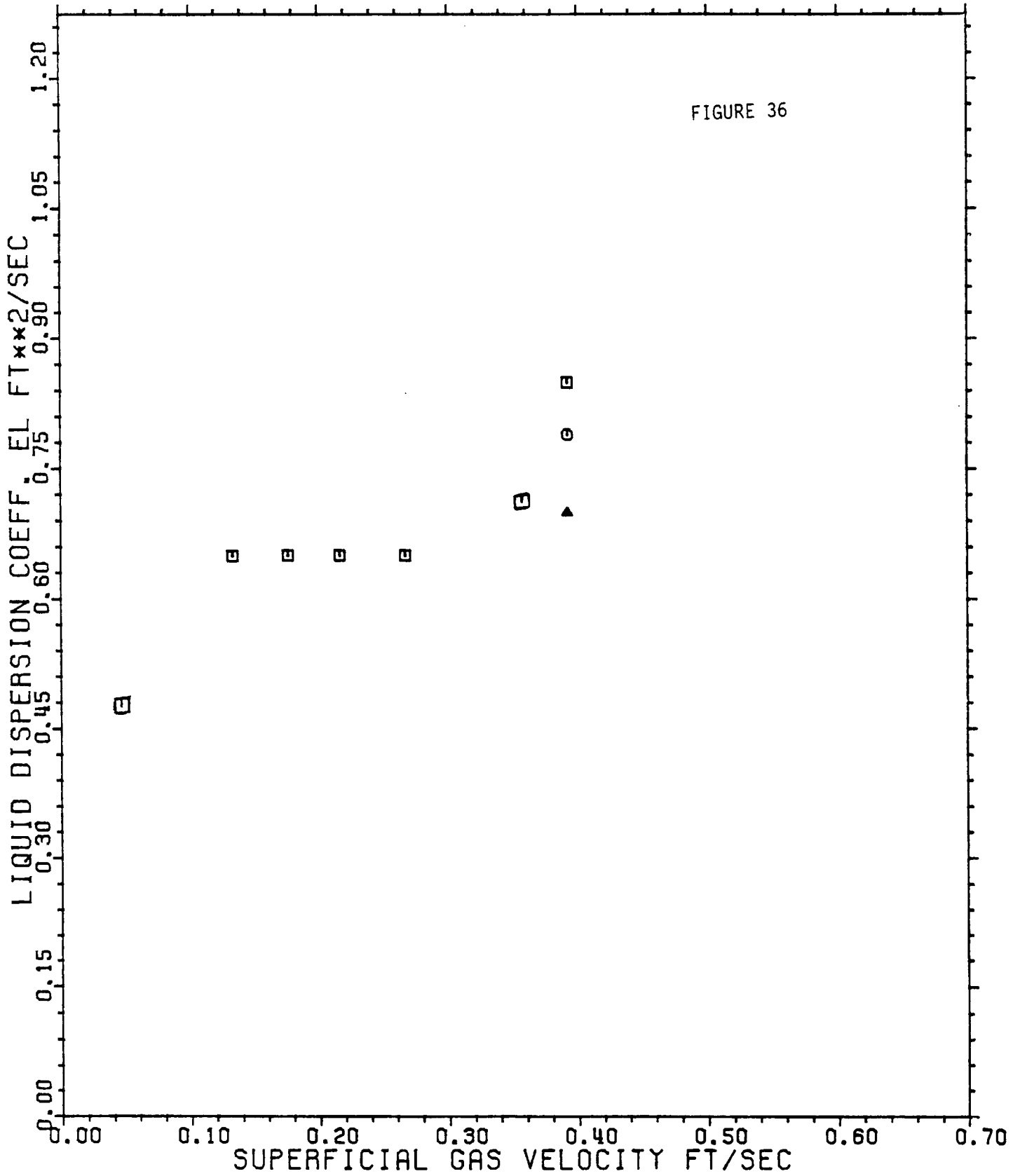
EFFECT OF SOLIDS ON EL

COMPARISON OF THEORETICAL AND EXPERIMENTAL C VS.  
VG-.392 FT/SEC

12" COLUMN AND TETRALIN USED (BATCH) 57

- EXPERIMENT C=0.00 LB/FT<sup>3</sup>
- EXPERIMENT C=6.19 LB/FT<sup>3</sup>
- △ EXPERIMENT C=22.01 LB/FT<sup>3</sup>
- MODEL EL=.79 FT<sup>2</sup>/SEC, C=6.19
- MODEL EL=.70 FT<sup>2</sup>/SEC, C=22.01

FIGURE 36



EFFECT OF VG ON EL	
EFFECT OF GAS VELOCITY ON EL 12 INCH COLUMN, TETRALIN (BATCH)	□ NO SOLIDS PRESENT ○ -140 MESH SAND C=8.01 LB/FT <sup>3</sup> ▲ -140 MESH SAND C=22.01 LB/FT <sup>3</sup>



## References

1. Pilhofer et al, "Determination of Fluid Dynamic Parameters in Bubble Column Design", Chemical Reaction Engineering-Houston, Advances in Chemistry Series, 109, 1972.
2. Akita , K. and Yoshida, F., I&EC Process Design Development 1973, 12 (76).
3. Yoshihiro et al. Chemical Engineering Science Vol. 25 (1970).

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