ORIGINAL

Catalyst and Reactor Development for a Liquid Phase Fischer-Tropsch Process

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ABSTRACT

In October 1980, Air Products and Chemicals, Inc. began a three year contract with the DOE: "Catalyst and Reactor Development for a Liquid Phase Fischer-Tropsch Process". The program contains four major tasks: (1) Project Work Plan, (2) Slurry Catalyst Development, (3) Slurry Reactor Design Studies, and (4) Pilot Facility Design.

This report describes work carried out in the fourth quarter of the contract. In Task 2, the 300 mL and 1000 mL slurry reactors ordered for the contract were constructed and instrumented. Mass transfer tests in the #1 300 mL slurry reactor were incorporated into the baseline catalyst test, because of the low activity that was found in two tests of a ruthenium methanation catalyst in the slurry phase. Two modified "conventional" catalysts were prepared and five gas phase screening tests were carried out, giving additional information on the conditions required to produce an enhanced C_{10}^{+} fraction. Eight supported cluster catalysts were synthesized, and eleven were screened in the gas phase.

In Task 3, measurements of gas holdup and solids dispersion were completed for the 1-5 μm silica/isoparaffin system. Preliminary correlations were derived for gas holdup as a function of gas velocity and slurry density: a near zero-order dependence upon slurry velocity was observed. The column solids profile was constant for the conditions studied. Bubble diameters were measured photographically in the two-phase system.

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1.0 INTRODUCTION

Coal liquefaction will be an important source of transportation fuels in the future, and can be accomplished by both the direct route (hydrogenation of coal in a donor solvent) or by an indirect route (gasification of coal followed by the Fischer-Tropsch reaction).

The product selectivity of the Fischer-Tropsch reaction has been the focus of extensive research for many years, yet still remains a prime target for technical innovation. Fischer-Tropsch technology, as it is currently practiced commercially for liquid fuels production, provides a broad range of hydrocarbon products which require costly downstream refining.

Selectivity can be influenced by variations in the catalyst composition and process conditions. Yet, in spite of the extensive effort devoted to this problem, a suitable catalyst has not previously been developed for producing a narrow range hydrocarbon product such as gasoline or diesel fuel without the coproduction of lighter and heavier undesirable products.

The Fischer-Tropsch reaction is also exothermic, and improved heat transfer would also be expected to have a major beneficial effect on product selectivity. Slurry phase reactor operation improves heat transfer and temperature control, and results in greater selectivity to liquid products, usually through lower methane production. However, considerable differences have been reported in the space-time yield, catalyst life and ease of operation of slurry phase reactors.

In addition to improved product selectivity, slurry phase operation offers the advantages of ease of scale-up and the ability to directly utilize the carbon monoxide-rich synthesis gas produced by coal gasifiers. The full potential of the slurry phase Fischer-Tropsch process has not yet been realized, and its further development is an important part of our country's program to establish viable technology for converting coal to hydrocarbon fuels.

Therefore, Air Products, (APCI) under contract to the DOE has undertaken a program in catalyst and reactor development for a slurry phase Fischer-Tropsch process. This contract spans 36 months and is divided into four major tasks. This report describes the work accomplished during the fourth quarter.

2.0 OBJECTIVE

The overall objective of this program is to evaluate the catalysts and slurry reactor systems for the selective conversion of synthesis gas into transportation fuels via a single stage, liquid phase process.

Task 1 - To establish a detailed Project Work Plan.

Task 2 - To evaluate and test catalysts for their potential to convert synthesis gas to gasoline, diesel fuel, or a mixture of transportation fuels suitable for domestic markets, and to quantify catalyst activity, selectivity, stability and aging with a target process concept involving a single stage, liquid phase reactor system.

Task 3 - To evaluate through the use of cold-flow reactor simulators the flow characteristics and behavior of slurry reactors for the production of hydrocarbons from synthesis gas. This includes (1) defining heat, mass and momentum transfer parameters which affect the design of slurry reactors, (2) establishing operating limits for slurry reactors with respect to system physical parameters, (3) developing or confirming correlations for predicting the flow characteristics and heat/mass transfer of slurry reactors, and (4) defining the necessary requirements for the design of larger scale reactors.

Task 4 - To develop a preliminary design for a pilot-plant slurry phase Fischer-Tropsch reactor.

3.0 SUMMARY AND CONCLUSIONS

3.1 Task 1 - Project Work Plan

This task was completed in the first quarter.

3.2 Task 2 - Slurry Catalyst Development

3.2.1 Sub-Task 2a - Background Studies

A computerized survey of available literature and patents dealing with the conventional and slurry phase Fischer-Tropsch processes and the hydrodynamics of three phase slurry reactors was continued.

3.2.2 Sub-Task 2b - Bench Scale Reactor Set Up

The 300 ml and 1000 ml slurry reactors ordered for the contract were constructed and instrumented.

Two mass transfer tests in the #1 300 ml slurry reactor using a ruthenium methanation catalyst were negated by the low activity of the catalyst in the slurry phase, which was not dependent on using either a gas or slurry phase activation procedure. The determination of reactor mass transfer characteristics is therefore being incorporated into a slurry test of the baseline Fischer-Tropsch catalyst that is currently in progress.

3.2.3 Sub-Task 2c - Catalyst Preparation and Slurry Reactor Testing

This section contains potentially patentable material and has therefore been issued in a supplementary report marked "Not For Publication".

3.2.4 Sub-Task 2d - Metal Cluster Catalyst Preparation and Screening Tests

This section contains potentially patentable material and has therefore been issued in a supplementary report marked "Not For Publication".

3.3 Task 3 - Slurry Reactor Design Studies

Measurements of gas holdup and solids dispersion in the isoparaffin/1-5 μm silica/nitrogen system were completed. For zero solids loading, the data fitted a correlation of Pilhofer and Bach[1], and at 10 wt.% solids was correlated by Akita and Yoshida[2]. However at 30 wt.%, both correlations predicted a higher gas holdup than was observed.

A preliminary correlation was derived by regression techniques for gas holdup as a function of gas velocity, slurry velocity and slurry density. A statistical analysis showed a near zero order dependence on slurry velocity, in agreement with previous published work.

The solids profile in the 5" column was uniform for all solids loadings in the range 10-30 wt.% and all gas velocities in the range 0.05-0.5 ft/sec. However, the 30 wt.% slurry was subject to plugging outside of the column, requiring some equipment modifications.

Bubble diameter measurements were made photographically in the two phase isoparaffin/nitrogen system. The average bubble diameter was 0.108 ± 0.021 inches, and a small decrease was found for increasing gas velocity at both zero and 0.1 ft/sec liquid velocity.

A technique was developed for measuring liquid dispersion by conductivity probe, and the design was drawn up for the 12" cold flow simulator.

4.0 ACKNOWLEDGEMENTS

The contributions to this program by T. G. Dunlap, J. M. LaBar, L. E. Schaffer and E. G. Valagene are gratefully acknowledged.

5.0 RESULTS AND DISCUSSION

5.1 Task 1 - Project Work Plan

This task was completed in the first quarter.

5.2 Task 2 - Slurry Catalyst Development

5.2.1 Sub-Task 2a - Background Studies

Background studies, including a computerized search of current literature and patents, were continued this quarter.

5.2.2 Sub-Task 2b - Bench Scale Reactor Set-Up

Construction and instrumentation of the second 300 ml and the 1000 ml slurry reactors continued. Completion and initial shakedown tests of the 1000 ml reactor are scheduled for next month.

Two mass transfer tests were initiated in the #1 300 ml slurry reactor using a slurry of 5% Ru/Al_2O_3 methanation catalyst. Methanation was chosen as the reaction for studying the mass transfer characteristics of the slurry reactor in order to simplify the product distribution and calculation of conversions. Both tests however were negated by the very low activity of the methanation catalyst in the slurry phase, in spite of two different activation procedures.

In the first mass transfer test (5871-11-MT1), the 300 mL reactor was loaded with 29.7g of 5% Ru/Al_2O_3 (-325 mesh) as 160 mls of a 19.36 wt.% slurry in Edwards No. 16 oil.

The catalyst was activated in the slurry phase at 800 rpm by contacting with H_2 at 845 psig, a GHSV of 218 h^{-1} and a temperature of 348°C for 17.5 hours. However, subsequent reaction at 250-350°C, 800-1500 rpm, 450 psig, and a GHSV of 280-994 h^{-1} with 2:1 H_2/CO produced a maximum CH_4 concentration in the outlet gas of only 0.8%. It was initially thought that this was due to insufficient activation of the catalyst limited by the maximum operating temperature of the reactor, and the test was therefore discontinued.

In the second mass transfer test (5871-27-MT2), the 5% Ru/Al_2O_3 was activated in the gas phase before transfer to the slurry phase under N_2. This procedure did not, however, improve the low catalyst activity that was observed in the previous test (5871-11-MT1), and only low yields of CH_4 (<1%) were obtained at 300°C, 800-1750 rpm, 450 psig, and a GHSV of 300 h^{-1}. The activation procedure was not, therefore, the reason for the low observed conversion, which remains unexplained.

It was therefore decided not to continue any further with the use of methanation for the slurry reactor mass transfer tests, but to incorporate these with the baseline Fischer-Tropsch catalyst test (5871-38-B1) utilizing a commercial sintered Fe_2O_3 promoted with 2-3% Al_2O_3, 0.5-0.8% K_2O and 0.7-1.2% CaO. This test is now successfully in progress and will be reported fully next quarter. The mass transfer

characteristics of the reactor are being determined by observing the effect of stirring speed and space velocity on conversion of a 2:1 H_2/CO mixture at various temperatures.

Preliminary GC analysis of the oil recovered from the reactor after the tests 5871-11-MT1 and 5871-27-MT2 showed no significant thermal cracking at 250-350°C over a period of 120 hours.

5.2.3 Sub-Task 2c - Catalyst Preparation and Slurry Reactor Testing

This section contains potentially patentable material and has therefore been issued in a supplementary report marked "Not For Publication".

5.2.4 Sub-Task 2d - Metal Cluster Catalyst Preparation and Screening Tests

This section contains potentially patentable material and has therefore been issued in a supplementary report marked "Not For Publication".

5.3 Task 3 - Slurry Reactor Design Studies

5.3.1 5" Cold Flow Simulator

(i) Gas Hold-Up in Isoparaffin

Gas hold-up results for the isoparaffin/1-5 μm silica/nitrogen system (including zero weight percent) were completed this quarter and are tabulated in Tables 1, 2, 3, 4. Figures 1 and 2 also plot this data for zero and positive liquid flow rates respectively.

The data is well correlated with Pilhofer & Bach[1] for zero weight percent (Figures 3 and 4), and Akita and Yoshida[2] at nominally ten weight percent solids (Figures 5 and 6). However, at thirty weight percent, both correlations predicted a higher gas hold-up than was observed. This data was statistically analyzed to given the following preliminary correlation, with an R^2 (goodness of fit) of 0.897:

$$\varepsilon_G = 79.08\ V_G^{0.654} V_L^{0.006} / \rho_{SL}^{1.31} \qquad (1)$$

[see nomenclature, section (vi)] where ρ_{SL} slurry density, is described by the equation

$$\rho_{SL} = w(\rho_S - \rho_L) + \rho_L \qquad (2)$$

This correlation is based on 58 experimental runs. Other particle sizes and solid types will, of course, expand the number of variables included in the correlation and will modify the exponents.

It should be noted that the correlation is only a preliminary one. Later correlations will include the slurry viscosity, μ_{SL}, which should increase the goodness of fit. Also, correlations will be presented in both dimensional and dimensionless forms.

The above correlations suggest a very strong probability that V_L is not a significant variable. This is quantatively confirmed by the F statistic test in Table 5 (pr >F), which shows that all variables but the log of the liquid velocity have a 99.99% chance of being significant, whereas the log of the velocity has a 43.3% chance of not being significant. Equation (1) can then be recorrelated to

$$\varepsilon_G = 78.01\ V_G^{0.652} / \rho_{SL}^{1.32} \qquad (3)$$

with an $R^2 = 0.894$. That liquid velocity has no discernable effect on gas hold-up is in agreement with most two- and three-phase work in the literature.

Multiple regressions were performed by taking the natural log of all the variables, and solving for the coefficients, a_i, of the equation

$$\ln \varepsilon_G = a_1 + a_2 \ln V_G + a_3 \ln V_L + a_4 \ln \rho_{SL} \qquad (4)$$

that would minimize the sum of the squares of the residuals. When V_L and W had zero values, it was necessary to change their values to 10^{-4} so that they could be included in the analysis.

Equation (3), also plotted in Figures 1 and 2, over-predicts ε_G at the higher gas velocities compared to the other two correlations:

Akita and Yoshida[2]:

$$\varepsilon_G/(1-\varepsilon_G)^4 = 6.12 \times 10^{-4} V_G \rho_L^{0.37}/(\mu_L^{0.17} \sigma_L^{0.2}) \quad (5)$$

and

Pilhofer and Bach[1]:

$$\varepsilon_G/(1-\varepsilon_G) = 0.34\ V_G^{0.69} \rho_L^{0.23}/\mu_L^{0.23} \quad (6)$$

As these correlations provide a good fit within limited ranges, $\varepsilon_G/(1-\varepsilon_G)^n$ with n = 1,2,3,4 will be tried as the dependent variable in future analyses.

Table 5 gives a summary of the statistical analysis performed. Figure 7 gives an actual vs. predicted plot. Again, the slight curvature in Figure 7 indicates that the correlation predicts too high a ε_G at high gas velocities.

(ii) Solid Dispersion

In the isoparaffin/1-5μm silica system, the solids profiles were uniform for all weight percent in the range of 10-30 wt% and all gas velocities in the range of 0.05-0.5 ft/sec. This result is in agreement with the literature.

(iii) Bubble Diameter

Photographic slides were taken of the isoparaffin-nitrogen system and representative bubble diameters were measured. A given bubble's diameter was measured horizontally, vertically, at 45° and at 135°. The bubble diameter was taken as the average of those four measurements. Table 6 lists the average bubble diameter from each slide (which is not the Sauter mean diameter).

For the most part, bubble diameters were fairly uniform for a given run, especially when liquid was flowing. Figure 8 plots bubble diameter vs. gas velocity for both 0.0 and 0.1 ft/sec liquid velocity, and shows a slight decrease in bubble diameter with increasing gas velocity. The overall average bubble diameter for all runs was 0.108 inches with an absolute standard deviation of 0.021 inches, and a relative standard deviation of 20.0%.

The bubble diameter measurements are in agreement with Calderbank[3] and with some of Deckwer's work using organic liquids[4]. However, Deckwer's measurements using "vestowax", an actual Fischer-Tropsch product from Sasol, gave smaller bubble diameters and this difference is difficult to reconcile. Additional information on the vestowax will be obtained in an attempt to understand the reason for the anomalously small bubble diameter in this medium.

(vi) Liquid Dispersion Development

Liquid dispersion measurements will be carried out in water using an electrolyte as a tracer and measuring its conductivity pulse through the column. The intended conductivity probe, while giving satisfactory results in a batch system, was found to be unsatisfactory for a flow-through system like the 5" column. Therefore, another probe was devised which appears to be satisfactory. The original and modified probe are both shown in Figure 9. The outputs from probes 1 and 2 are shown in Figures 10 and 11. Probe 1 unfortunately gave a fluctuating signal for a constant conductivity. The modified probe 2 gave a constant signal, but with a negative bias resulting from the nitrogen bubbles disrupting the electrical circuit between the two electrodes on the probe. This negative bias noise will be filtered out using mathematical techniques.

(v) Behavior of the 30 wt% 1-5 μm Silica/Isoparaffin Slurry

The 30 wt% 1-5 μm silica/isoparaffin slurry is qualitatively different from both the 8.5 wt.% 1-5 μm silica/isoparaffin slurry and the 29.2 wt% 1-5 μm silica/water slurry. Whereas the latter two appeared to be in a very "fluid" state when dripping or settling in the column, the 30 wt% silica/isoparaffin system takes on a paste-like or mud-like consistency. It is only when nitrogen is flowing through it that the slurry appears as fluid as the other two systems.

The qualitative different fluid behavior necessitated several equipment modifications to prevent continual equipment plugging. A nitrogen bleed line was added at the bottom of the reservoir tank to facilitate pumping the slurry. As the gas flow is monitored at the exit from the column, this bleed nitrogen is accounted for in the total nitrogen flow. It was also necessary for the slurry to exit the column at a steeper angle than before, and the inline bucket, used to measure the slurry velocities, had to be relocated and tilted at a greater angle.

(vi) Nomenclature

a	Constant
V	Velocity, ft/sec
W	Solids weight fraction
ε	Gas hold-up
ρ	Density, lb/ft^3
σ	Surface tension, lb/hr^2
μ	Viscosity, lb/ft hr

Subscripts

G	Gas
L	Liquid
SL	Slurry

5.3.2 12" Cold Flow Simulator

An initial design for the 12" column was completed, and equipment will be ordered shortly.

6.0 EXPERIMENTAL

6.1 Task 1 - Project Work Plan

No experimental work.

6.2 Task 2 - Slurry Catalyst Development

6.2.1 Sub-Task 2a - Background Studies

No experimental work.

6.2.2 Sub-Task 2b - Bench Scale Reactor Set-Up

Construction of the 300 mL and 1000 mL slurry reactors continued with completion of the 1000 mL reactor and initial shakedown tests expected next month.

The first mass transfer test (5871-11-MT1) in slurry reactor #1 was limited by the low activity of the methanation catalyst. The 300 mL reactor was loaded with 29.7g of 5% Ru/Al_2O_3 (-325 mesh) as 160 mls of a 19.36 wt.% slurry in Edwards No. 16 oil.

The catalyst was activated in the slurry phase at 800 rpm by contacting with H_2 at 845 psig, a GHSV of 218 h^{-1} and a temperature of 348°C for 17.5 hours. However, subsequent reaction at 250-350°C, 800-1500 rpm, 450 psig, and a GHSV of 280-994 h^{-1} with 2:1 H_2/CO produced a maximum CH_4 concentration in the outlet gas of only 0.8%.

A second mass transfer test (5871-27-MT2) was therefore begun using 5% Ru/Al_2O_3 activated in the gas phase a higher temperatures. The catalyst was loaded into a tubular reactor and activated with H_2 at 1 atm and a GHSV of 500 h^{-1}, raising the temperature slowly from 350 to 420°C and maintaining 420°C for 3.5 hours. After cooling and

dismantling under N_2, the catalyst was slurried in deoxygenated No. 16 oil and transferred to the slurry reactor under a N_2 purge. This procedure did not, however, result in an increase in activity and this test was also discontinued. Mass transfer tests are now being incorporated with the baseline Fischer-Tropsch catalyst slurry test.

6.2.3 Sub-Task 2c - Catalyst Preparation and Slurry Reactor Tests

This section contains potentially patentable material and has therefore been issued in a supplementary report marked "Not For Publication".

6.2.4 Sub-Task 2d - Cluster Catalyst Preparation and Screening Tests

This section contains potentially patentable material and has therefore been issued in a supplementary report marked "Not For Publication".

6.3 Task 3 - Slurry Reactor Design Studies

6.3.1 5" Cold Flow Simulator

(i) Solid Dispersion Analytical Procedure

While the water/silica system was dried in an oven to obtain solid wt%, such a procedure was deemed unnecessarily hazardous for the isoparaffin slurry. Instead, an aspiration followed by pentane wash technique was developed and is explained below. Unfortunately, this technique was perfected only after a number of the 10 and 20 wt% samples had been erroneously analyzed, and as a result, part of the quarter was spent resimulating various conditions in order to obtain complete solid profiles.

The procedure followed for the aspiration requires a micro-pore filter funnel (Gelman 4200, 47 mm membrane filter) 0.45 μm filter paper, two 500 ml filter flasks, an aspirating line and 100 ml pentane, and is as follows:

Place a 0.45 μm filter paper in the weighed filter funnel and attach to the filter flask labeled "ISOPARAFFIN". Shake this sample bottle to mix the slurry and filter. When the sample appears dry, transfer the filter funnel to the filter flask labeled "PENTANE".

Rinse the sample bottles with pentane into the filter funnel. Wash the sample several times with pentane (approximately 30 ml per wash), and allow to dry thoroughly. Remove the filter paper from the funnel and transfer the contents to a weighing boat. Reweigh the filter funnel and the weighing boat to obtain the total solids weight.

7.0 REFERENCES

1. T. H. Pilhofer, H. F. Bach and K. H. Mangartz, ACS Symp. Ser., 1978, 65, 372.

2. K. Akita and F. Yoshida, Ind. Eng. Chem. Proc. Des. Dev. 1973, 12, 76.

3. P. H. Calderbank, F. Evans, F. Farley, G. Jepson, and A. Poll, "Catalysis in Practice", Symp." Proc., Inst. Chem. Engrs., London, 1963, 66.

4. G. Quicker and W. D. Deckwer, Chem. Eng. Sci., 1981, 36, 9, 1577-1579.

5. H. Hikita and Kikukawa, Chem. Engng. J. 1973, 81, 74.

8.0 FIGURES

FIGURE 1

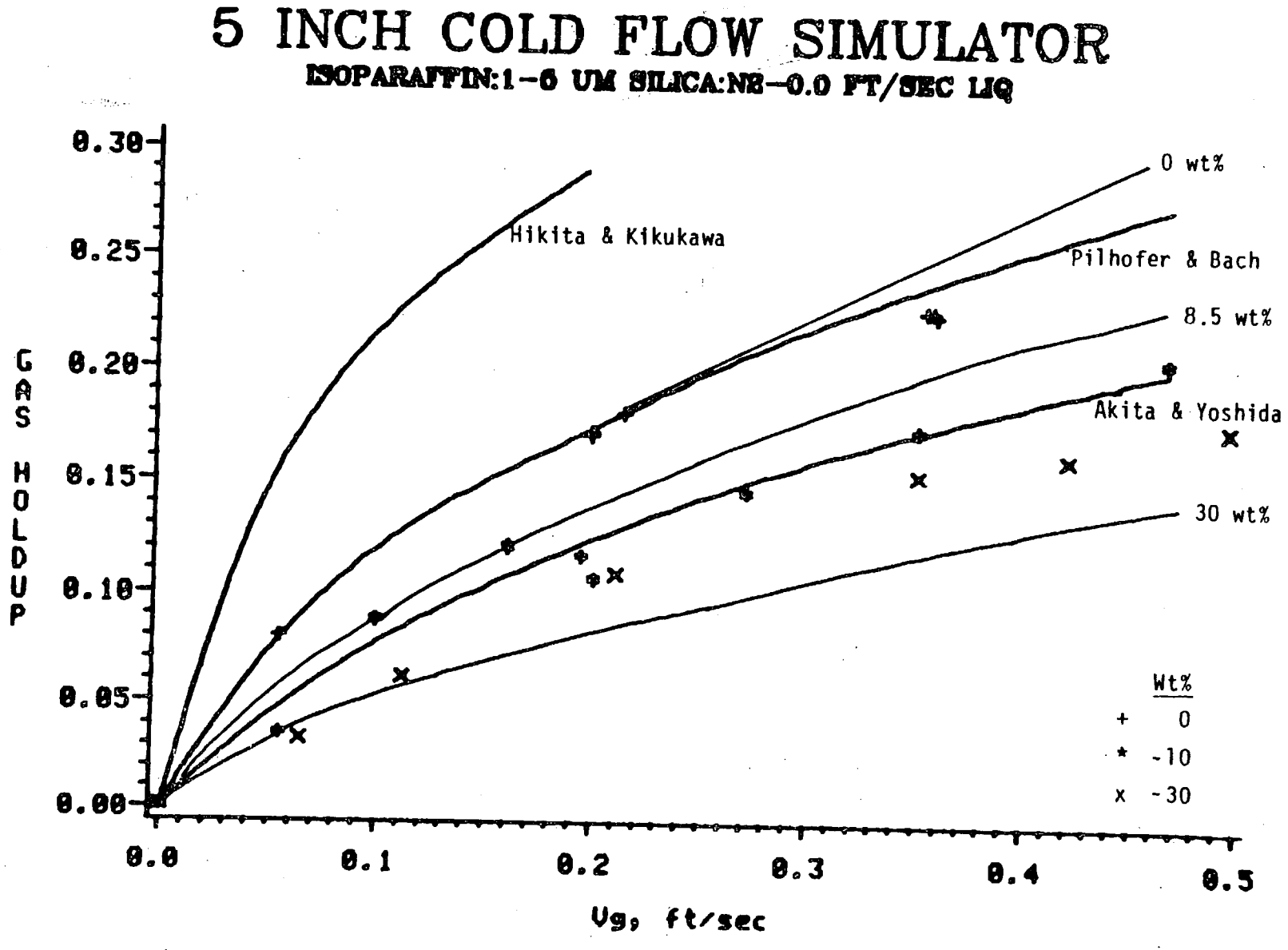

5 INCH COLD FLOW SIMULATOR
ISOPARAFFIN:1-6 UM SILICA:N2-0.0 FT/SEC LIQ
GAS HOLDUP
0.30
0.25
0.20
0.15
0.10
0.05
0.00
0.0
0.1
0.2
0.3
0.4
0.5
Ug, ft/sec
Hikita & Kikukawa
Pilhofer & Bach
Akita & Yoshida
0 wt%
8.5 wt%
30 wt%
Wt%
+ 0
* -10
x -30

FIGURE 2

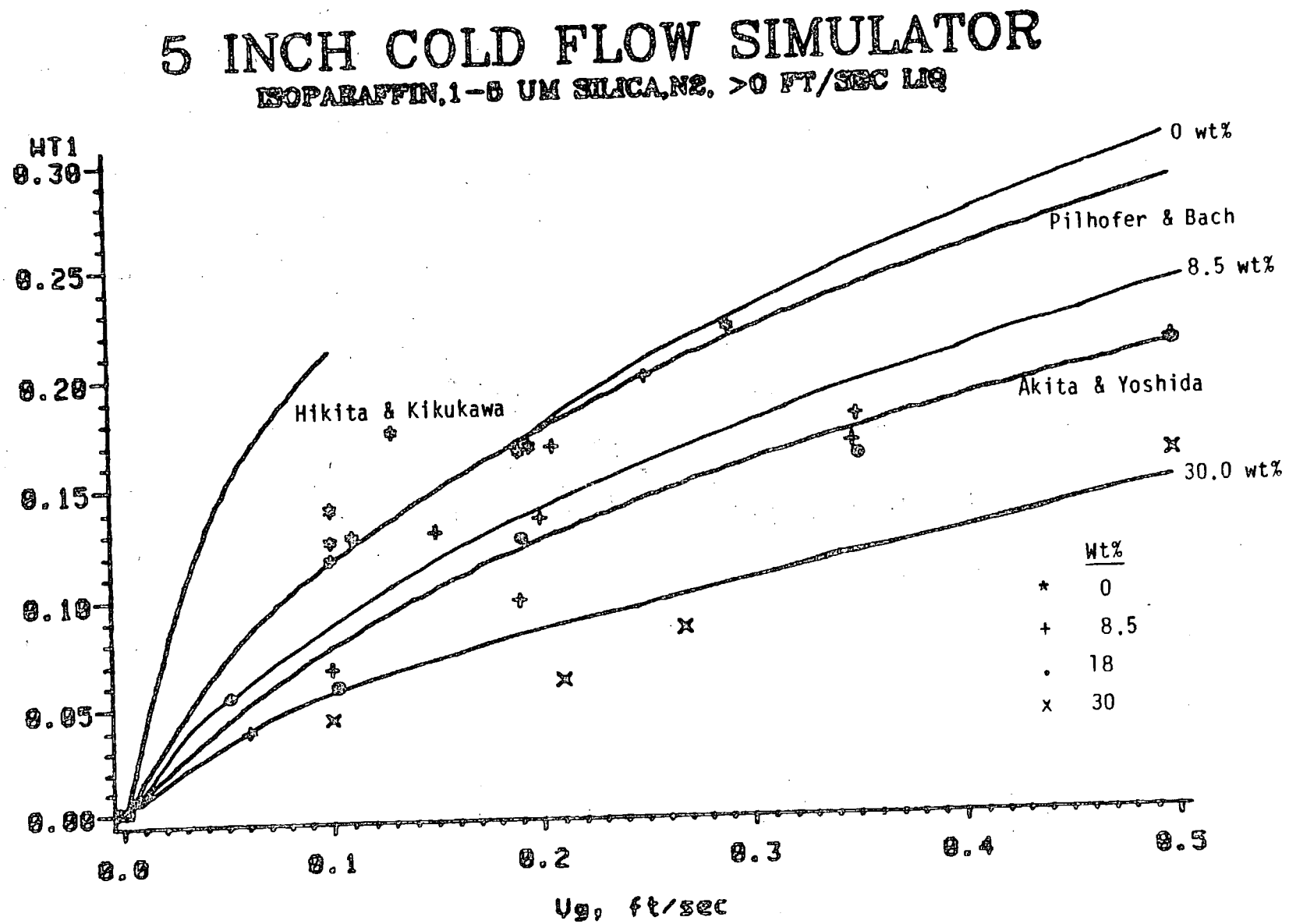

5 INCH COLD FLOW SIMULATOR
ISOPARAFFIN,1-6 UM SILICA,N2, >0 FT/SEC LIQ
HT1
0.30
0.25
0.20
0.15
0.10
0.05
0.00
0.0
0.1
0.2
0.3
0.4
0.5
Ug, ft/sec
0 wt%
Pilhofer & Bach
8.5 wt%
Akita & Yoshida
30.0 wt%
Hikita & Kikukawa
Wt%
* 0
+ 8.5
. 18
x 30

FIGURE 3

5 INCH COLD FLOW SIMULATOR

2-PHASE: ISOPARAFFIN-NITROGEN

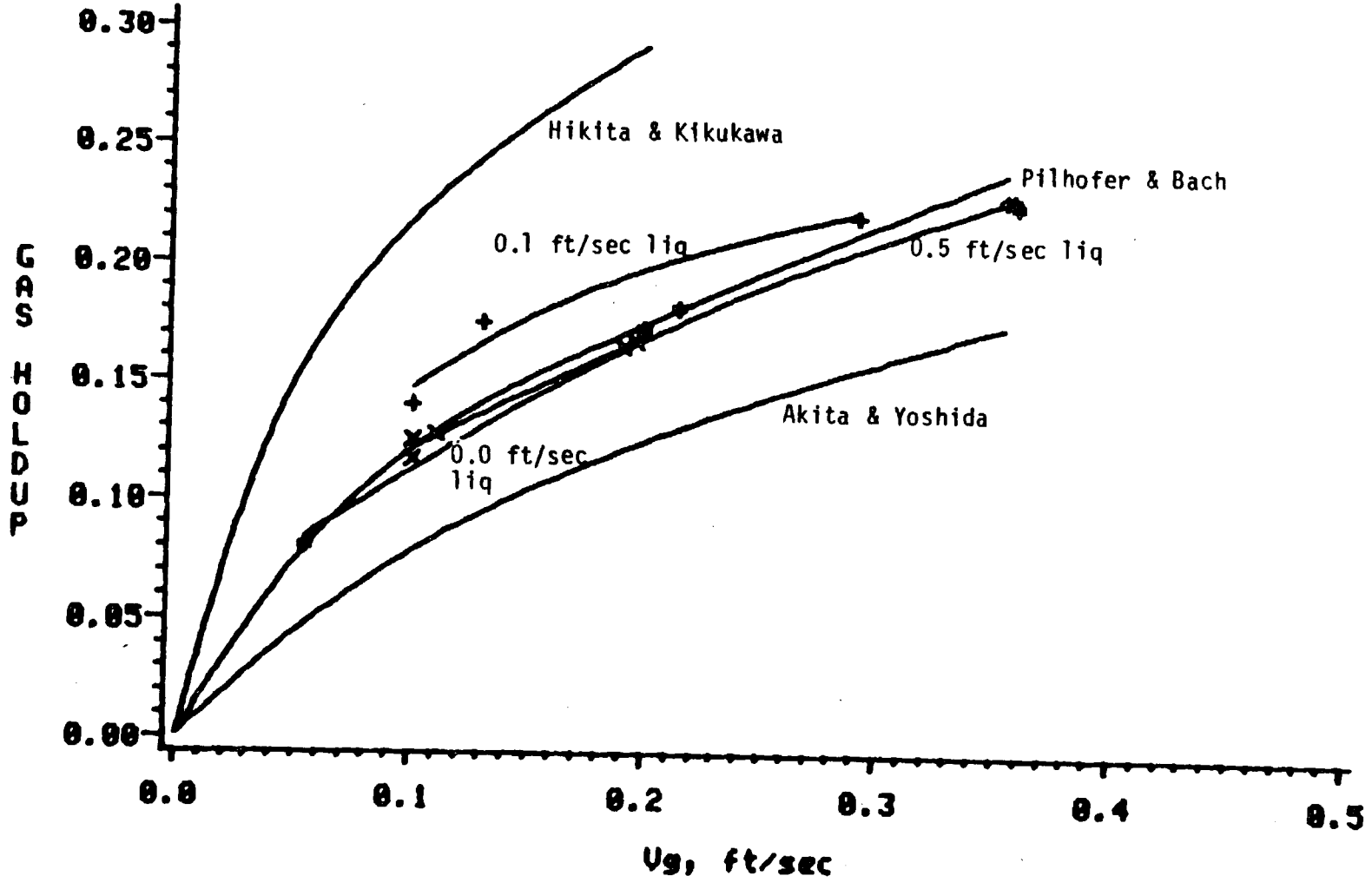


FIGURE 4

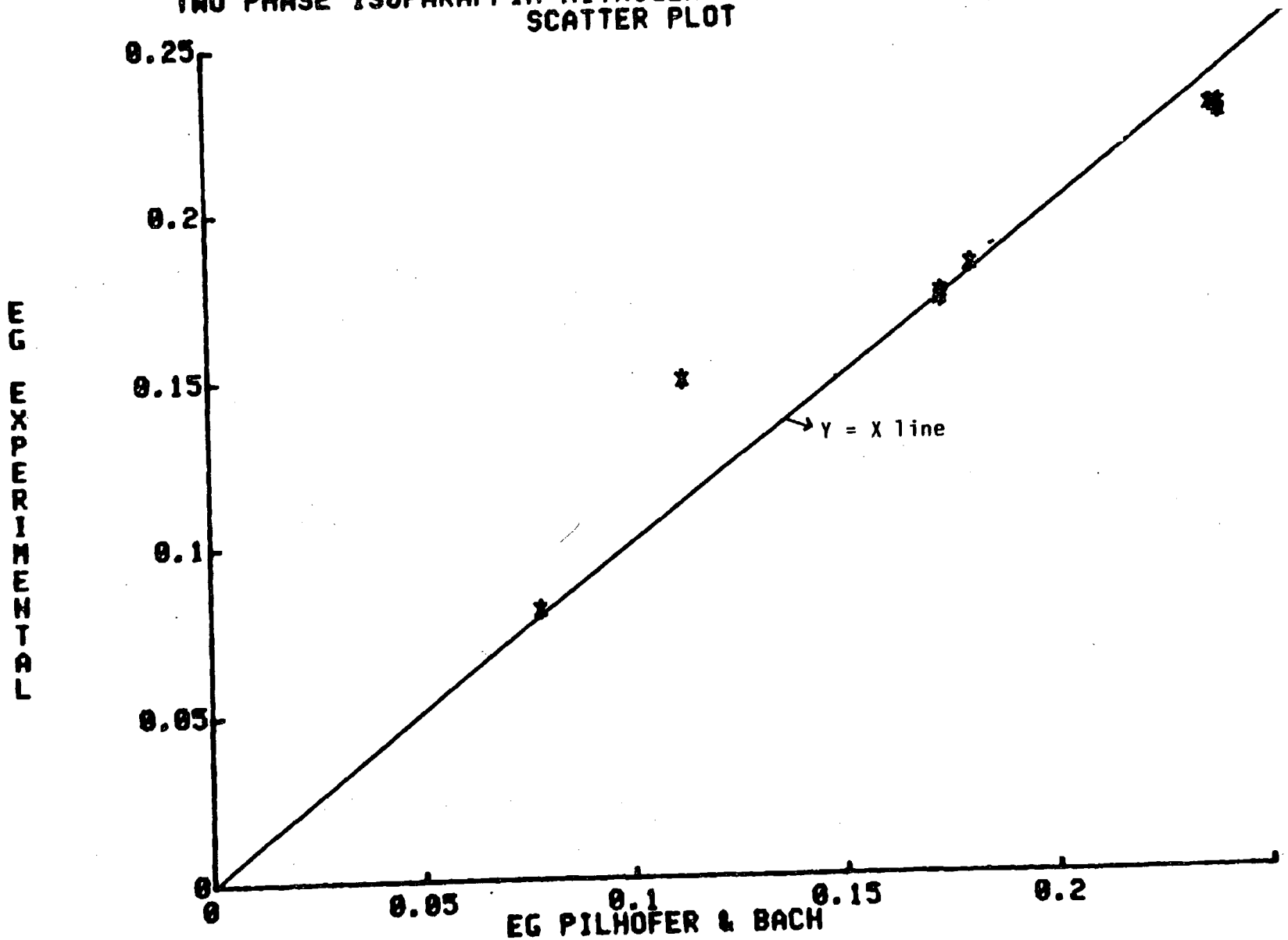

TWO PHASE ISOPARAFFIN-NITROGEN SYSTEM, 0.0 FT/SEC LIQ
SCATTER PLOT
EG EXPERIMENTAL
EG PILHOFER & BACH
Y = X line
0.25
0.2
0.15
0.1
0.05
0
0
0.05
0.1
0.15
0.2

FIGURE 5

5 INCH COLD FLOW SIMULATOR

3-PHASE ISOPARAFFIN - 8.5 WT % 1-5 UM SILICA - N2

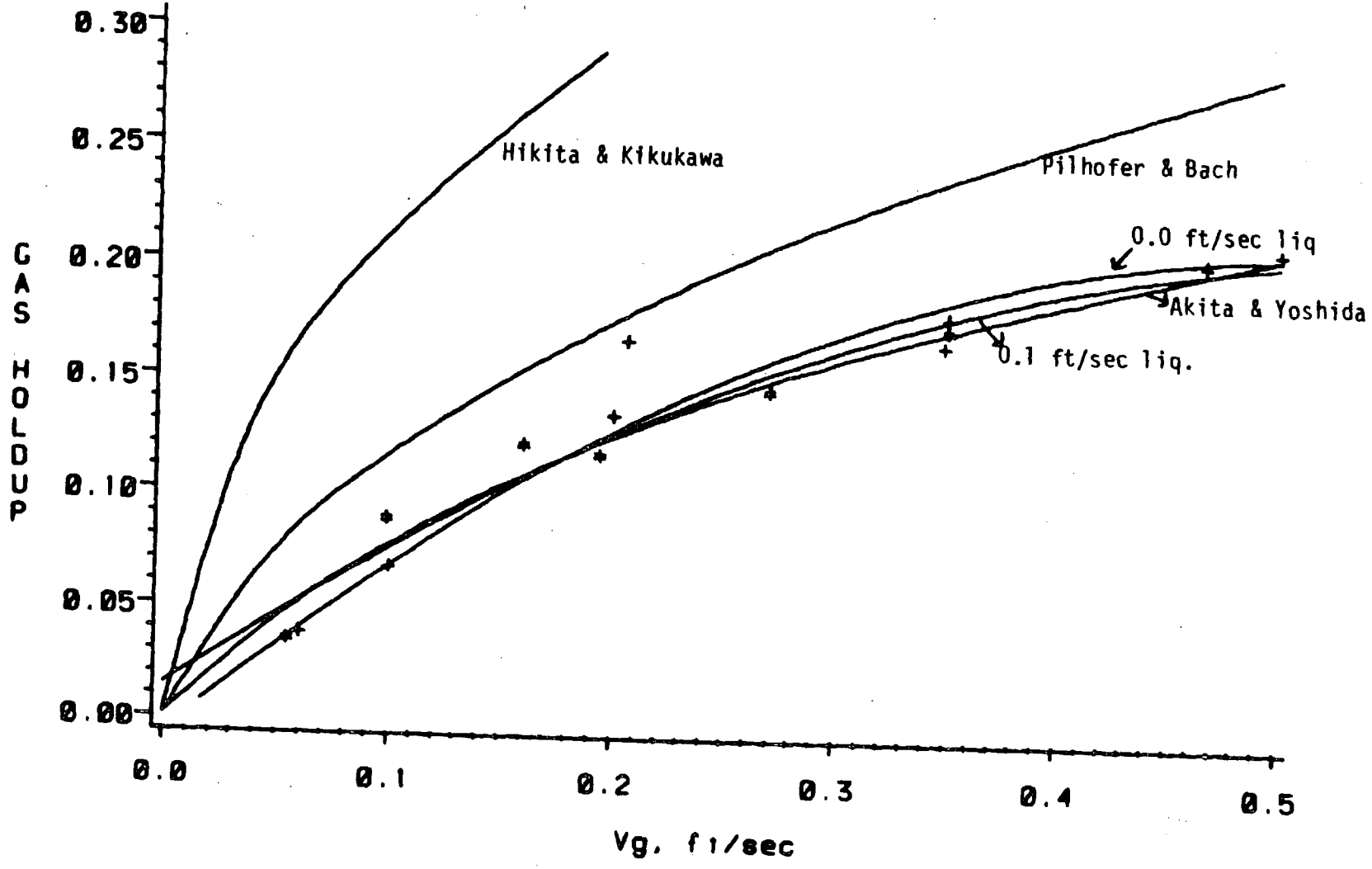


FIGURE 6

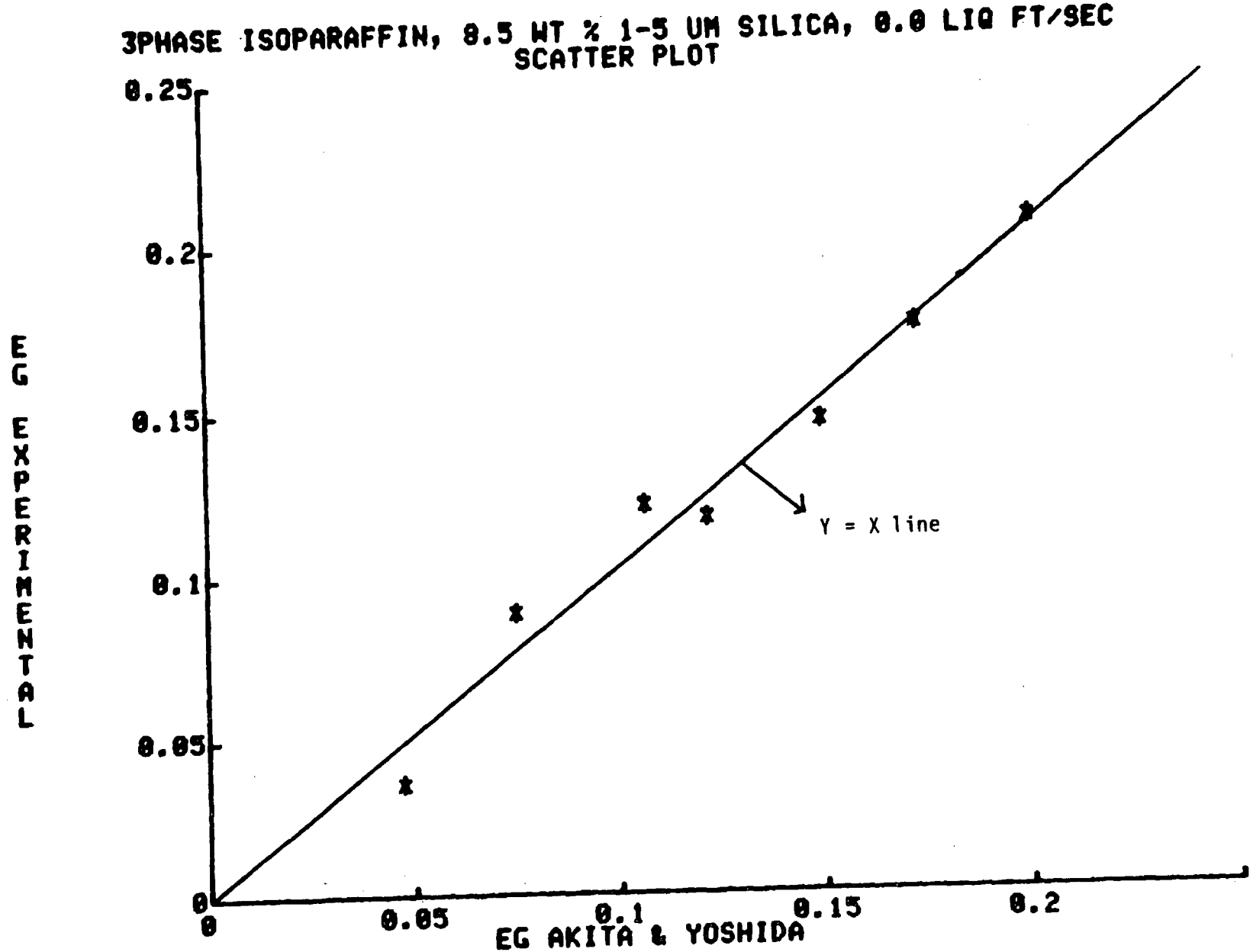

3PHASE ISOPARAFFIN, 8.5 WT % 1-5 UM SILICA, 0.0 LIQ FT/SEC
SCATTER PLOT
EG EXPERIMENTAL
EG AKITA & YOSHIDA
0.25
0.2
0.15
0.1
0.05
0
0.05
0.1
0.15
0.2
Y = X line

FIGURE 7

PLOT OF DECODED ACTUAL AND PREDICTED VALUES

PLOT OF _ACT#_PRED LEGEND: A = 1 OBS, B = 2 OBS, ETC.

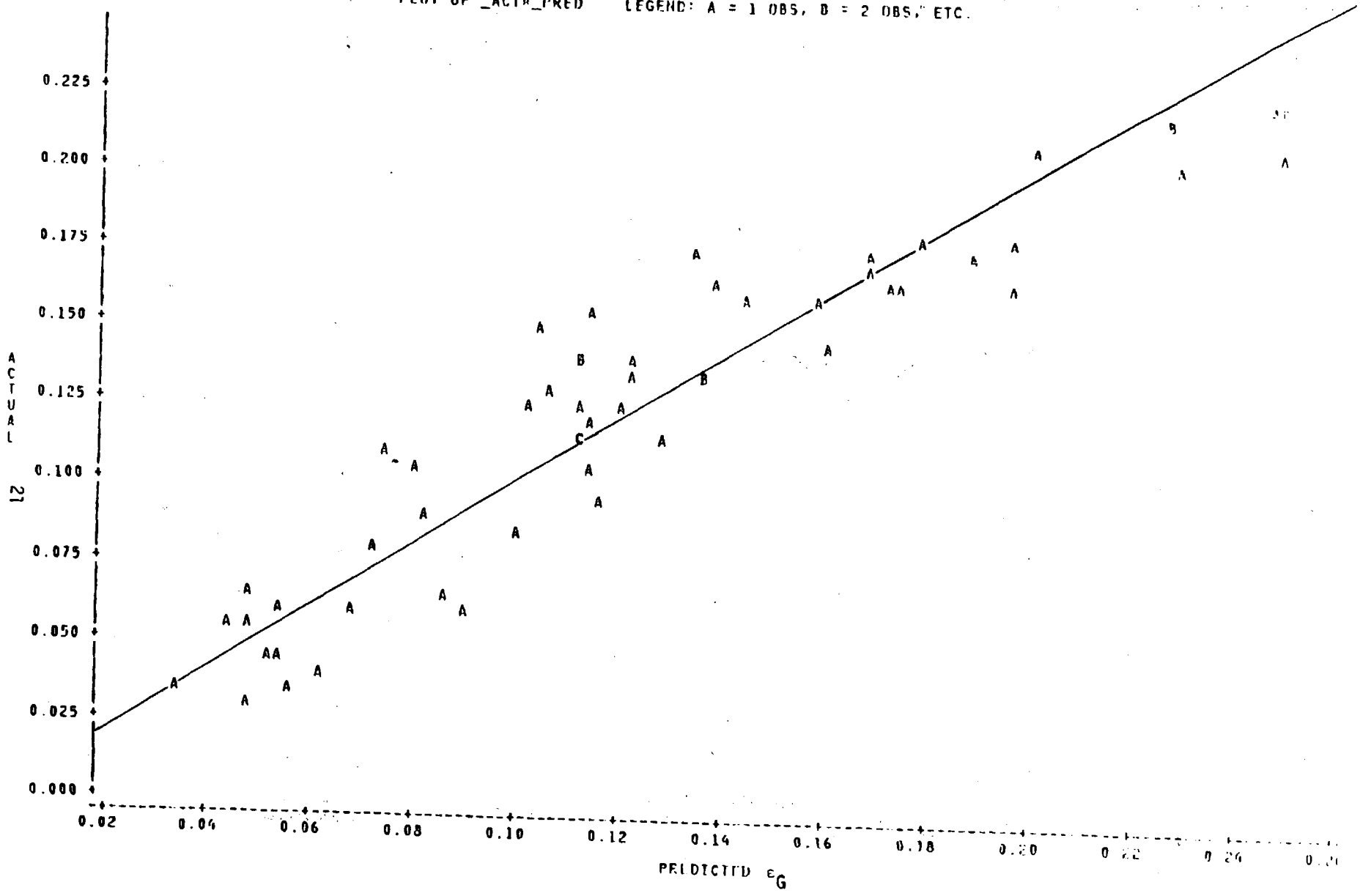


FIGURE 8

5 INCH COLD FLOW SIMULATOR

BUBBLE DIAMETER VS GAS VELOCITY

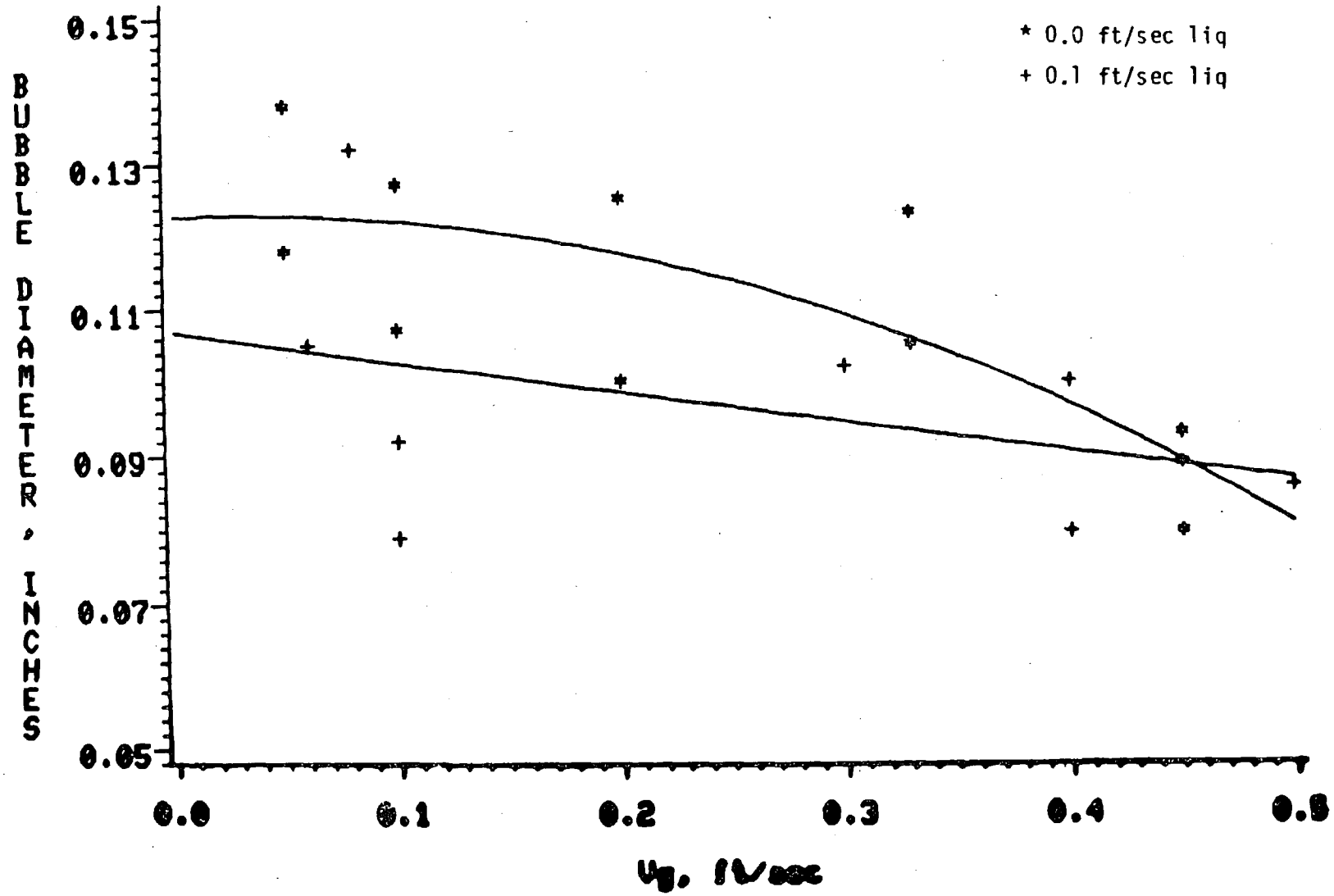
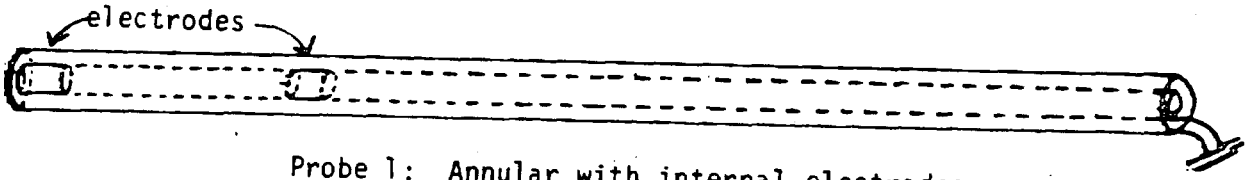
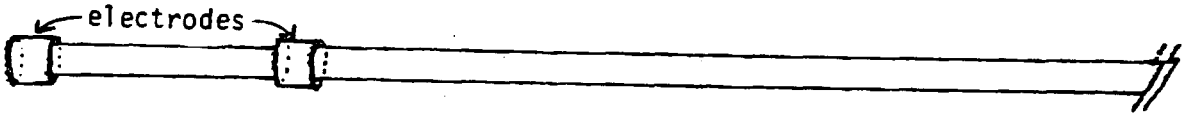


FIGURE 9

CONDUCTIVITY PROBES



Probe 1: Annular with internal electrodes



Probe 2: Cylindrical with external electrodes

FIGURE 10

PROBE 1 RESPONSE

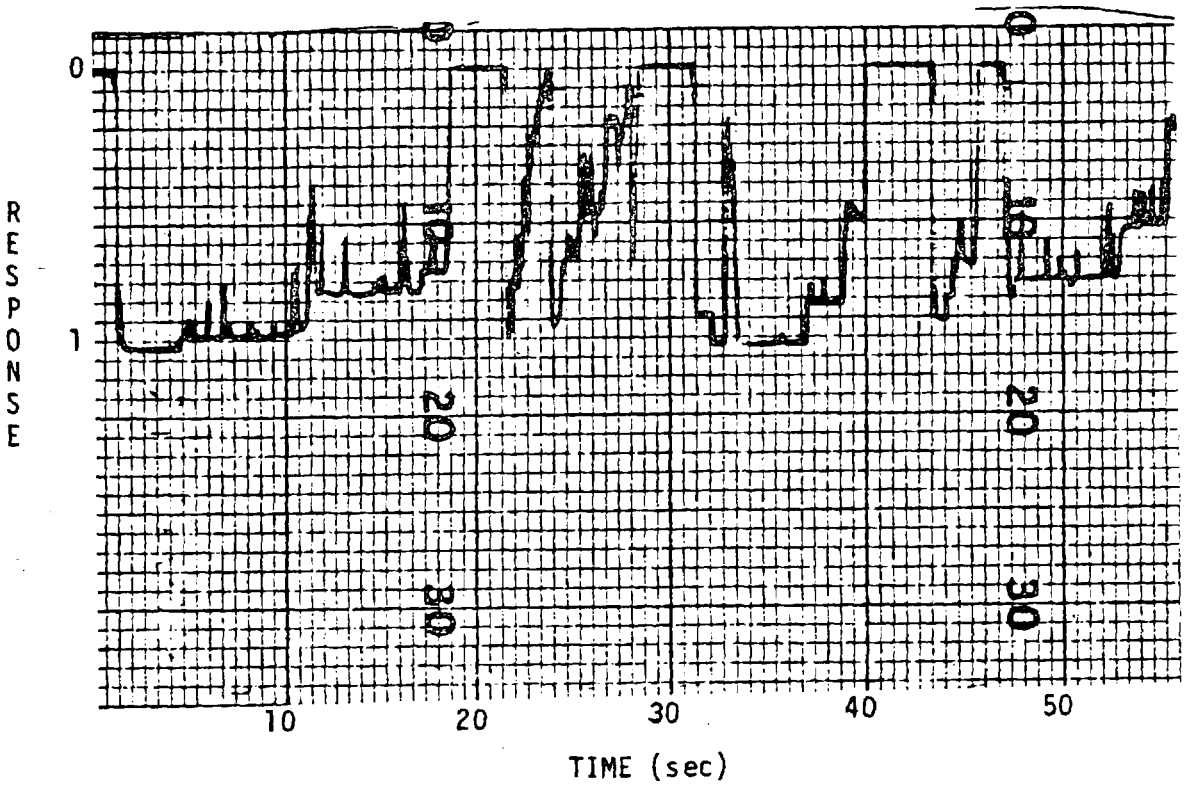
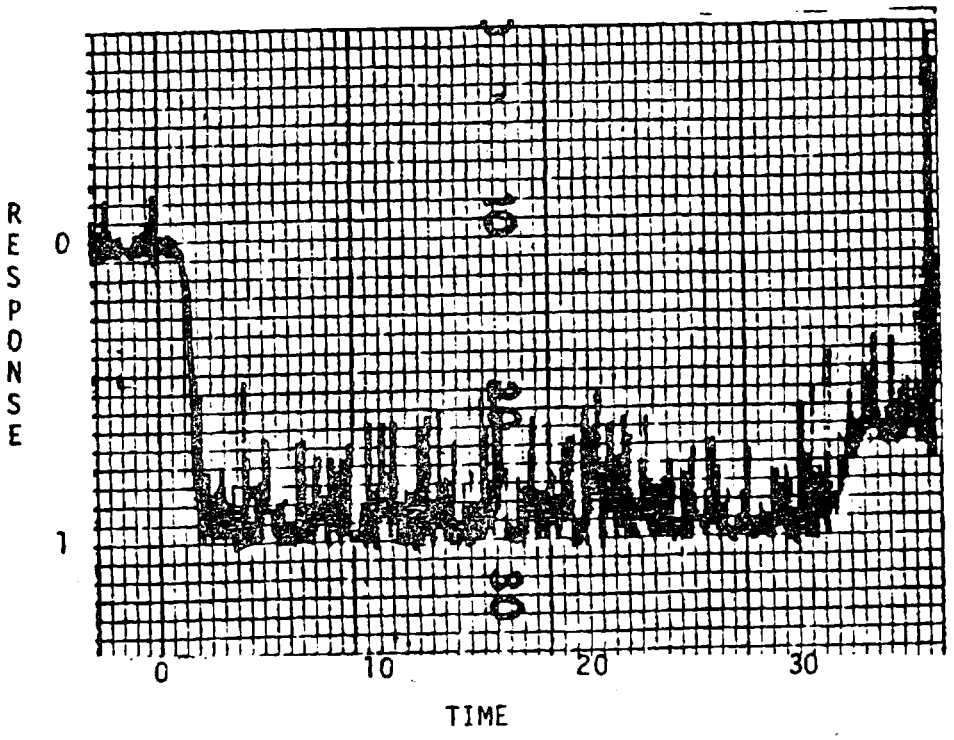


FIGURE 11

PROBE 2 RESPONSE



9.0 TABLES

TABLE 1

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM:TWO PHASE

LIQUID-ISOPARAFFIN

Gas - Nitrogen

RUN	VG FT/SEC	VL FT/SEC	EG EXP.	EG AKITA & YOSHIDA[2]	EG HIKITA & KIKUKAWA[5]	EG FILHOFER & Bachl
6000-35-1	0.130	0.094	0.175	0.093	0.240	0.136
6000-37-1	0.100	0.090	0.140	0.077	0.212	0.116
6000-38-1	0.290	0.094	0.220	0.156	0.350	0.215
6000-39-1	0.100	0.052	0.117	0.077	0.212	0.116
6000-37-1	0.100	0.090	0.140	0.077	0.212	0.116
6000-38-1	0.290	0.094	0.220	0.156	0.350	0.215
6000-39-1	0.100	0.052	0.117	0.077	0.212	0.116
6000-40-1	0.100	0.052	0.125	0.077	0.212	0.116
6000-41-1	0.110	0.048	0.127	0.083	0.222	0.123
6000-42-1	0.195	0.046	0.167	0.122	0.290	0.172
6000-43-1	0.190	0.049	0.165	0.120	0.287	0.170
6000-44-1	0.100	0.050	0.117	0.077	0.212	0.116

TABLE 2

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM:THREE PHASE

LIQUID-ISOPARAFFIN

SOLID-10 WT% 1-5 UM SILICA

GAS-NITROGEN

RUN	VG FT/SEC	VL FT/SEC	EG EXP.	EG AKITA & YOSHIDA[2]	EG HIKITA & KIKUKAWA[5]	EG PILHOFER & Bach[1]
6000-47-1	0.160	0.0	0.121	0.107	0.265	0.154
6000-48-1	0.270	0.0	0.146	0.150	0.336	0.207
6000-49-1	0.194	0.0	0.117	0.122	0.290	0.172
6000-50-1	0.099	0.0	0.088	0.076	0.211	0.115
6000-51-1	0.466	0.0	0.205	0.201	0.437	0.275
6000-52-1	0.350	0.0	0.174	0.173	0.382	0.237
6000-53-1	0.055	0.0	0.035	0.048	0.160	0.080
6000-54-1	0.348	0.050	0.167	0.173	0.381	0.237
5992-69-1	0.100	0.100	0.067	0.077	0.212	0.116
5992-70-1	0.500	0.100	0.210	0.208	0.452	0.285
5992-71-1	0.060	0.100	0.038	0.051	0.167	0.084
5992-72-1	0.300	0.100	0.134	0.124	0.294	0.175

TABLE 2 (cont'd)

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM:THREE PHASE

LIQUID-ISOPARAFFIN

SOLID-10 WT% 1-5 UM SILICA

GAS-NITROGEN

RUN	VG FT/SEC	VL FT/SEC	EG EXP.	EG AKITA & YOSHIDA[2]	EG HIKITA & KIKUKAWA[5]	EG FILHOFER & Bach[1]
6000-55-1	0.206	0.050	0.167	0.127	0.298	0.178
6000-61-1	0.350	0.089	0.179	0.173	0.382	0.237
5992-72-1	0.200	0.100	0.134	0.124	0.294	0.175
6000-83-1	0.190	0.088	0.097	0.120	0.287	0.170
6000-84-1	0.200	0.0	0.107	0.124	0.294	0.175
6000-85-1	0.050	0.918	0.005	0.044	0.153	0.075
6000-86-1	0.250	0.100	0.198	0.143	0.326	0.198
6000-87-1	0.150	0.100	0.129	0.103	0.257	0.148

TABLE 3

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM:THREE PHASE

LIQUID-ISOPARAFFIN

SOLID-20 WT% 1-5 UM SILICA

GAS-NITROGEN

RUN	VG FT/SEC	VL FT/SEC	EG EXP.	EG AKITA & YOSHIDA[2]	EG HIKITA & KIKUKAWA[5]	EG FILHOFER & Bach[1]
6000-90-1	0.102	0.085	0.058	0.078	0.214	0.117
6000-93-1	0.190	0.067	0.125	0.120	0.287	0.170
5992-74-1	0.500	0.050	0.208	0.206	0.452	0.285
5992-75-1	0.350	0.074	0.160	0.173	0.382	0.237
5992-76-1	0.050	0.082	0.054	0.044	0.153	0.075

TABLE 4

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM:THREE PHASE

LIQUID-ISOPARAFFIN

SOLID-30 WT% 1-5 UM SILICA

GAS-NITROGEN

RUN	VG FT/SEC	VL FT/SEC	EG EXP.	EG AKITA & YOSHIDA[2]	EG HIKITA & KIKUKAWA[5]	EG FILHOFER & Bach[1]
6000-66-1	0.350	0.0	0.154	0.173	0.382	0.237
6000-68-1	0.112	0.0	0.062	0.084	0.224	0.124
6000-69-1	0.210	0.0	0.109	0.126	0.301	0.180
6000-70-1	0.064	0.0	0.033	0.054	0.172	0.088
6000-72-1	0.100	0.100	0.043	0.077	0.212	0.116
6000-73-1	0.210	0.096	0.060	0.128	0.301	0.180
6000-74-1	0.268	0.096	0.083	0.149	0.337	0.206
6000-76-1	0.500	0.100	0.159	0.206	0.453	0.285

TABLE 5

SUMMARY OF STATISTICAL ANALYSIS ON EQUATION

$$\ln \epsilon_G = 4.37 + 0.654 \ln V_G + 0.006 \ln V_L - 1.31 \ln \rho_{SL}$$

STATISTIC	VALUE	EXPLANATION
R Squared	0.897	Goodness of fit
F Value	119.77	Significance of model
Probability greater than F value	0.0001	Probability model is not significant
Sum of Squared Residuals	2.19	Residual = (Actual - predicted value
Sum of Squares of Model	14.57	

VARIABLE	F VALUE	PROBABILITY > F Value	
$\ln V_G$	248.55	0.0001	Probability that effect
$\ln V_L$	0.62	0.4330	on dependent variable
$\ln \rho_{SL}$	123.16	0.0001	is due to random chance

TABLE 6

BUBBLE DIAMETER: 5" Cold Flow Simulator
System: Two Phase
Liquid - Isoparaffin
Gas - Nitrogen

Slide	V_G ft/sec	V_L ft/sec	$\bar{d}$ inches
1	0.20	0	0.110
2	0.20	0	0.125
3	0.33	0	0.123
4	0.33	0	0.105
5	0.45	0	0.093
6	0.45	0	0.089
7	0.45	0	0.080
8	0.10	0	0.127
9	0.10	0	0.107
11	0.05	0	0.118
12	0.05	0	0.138
18	0.30	0.10	0.102
19	0.40	0.10	0.100
20	0.40	0.10	0.080
21	0.10	0.10	0.079
22	0.10	0.10	0.092
23	0.06	0.10	0.105
24	0.08	0.10	0.132
25	0.50	0.10	0.086
26	0.50	0.10	

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