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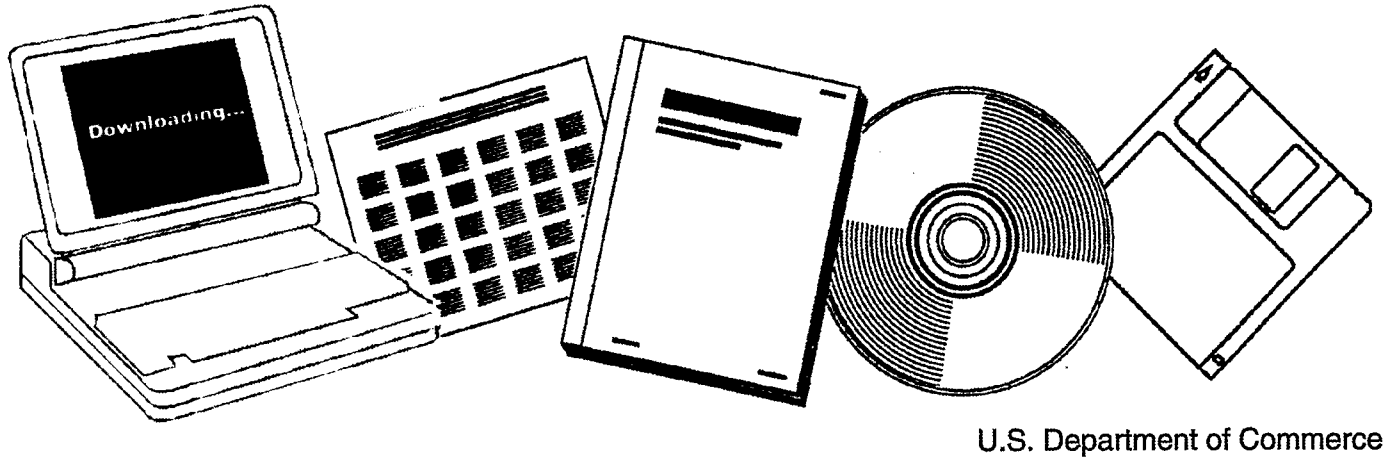
CATALYST AND REACTOR DEVELOPMENT FOR A LIQUID-PHASE FISCHER-TROPSCH PROCESS. QUARTERLY TECHNICAL PROGRESS REPORT, 1 OCTOBER 1982-31 DECEMBER 1982

AIR PRODUCTS AND CHEMICALS, INC.
ALLENTOWN, PA

MAR 1983



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CATALYST AND REACTOR DEVELOPMENT FOR A LIQUID PHASE FISCHER-TROPSCH PROCESS

QUARTERLY TECHNICAL PROGRESS REPORT
FOR PERIOD 1 OCTOBER 1982 - 31 DECEMBER 1982

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MARCH 1983

PREPARED FOR UNITED STATES DEPARTMENT OF ENERGY
UNDER CONTRACT NO. DE-AC22-80PC30021

REPRODUCED BY: NTIS
U.S. Department of Commerce
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Springfield, Virginia 22161

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ABSTRACT

Two major tasks continued in the ninth quarter of the Air Products and Chemicals, Inc./U. S. Department of Energy contract, "Catalyst and Reactor Development for a Liquid Phase Fischer-Tropsch Process": (1) Slurry Catalyst Development, and (2) Slurry Reactor Design Studies.

The first extended slurry test was begun, using a proprietary catalyst. High selectivities and large deviations from the Schulz-Flory distribution were observed. A bulk activity 2.5 times greater than the baseline Fe_2O_3 was determined, with little deactivation over 450 h. Consistently low CH_4 yields of 3-5 wt%, and high C_9-C_{25} fractions of 45-50 wt%, were produced at 240°C, 300 psig and 1:1 CO/H_2. This test is being continued with higher CO/H_2 ratios and operating temperatures.

Parametric gas phase screening studies were concluded for two further "modified conventional" catalysts and the optimum preparations in terms of activity and diesel range selectivity were chosen for subsequent slurry phase testing.

Gas holdup and solid concentration profiles were measured for water/silica slurries in the 5" column, and for paraffin/silica slurries in the 12" column, both with and without heat transfer internals. In both columns, gas holdup was found to be close to the Akita and Yoshida correlation. Very non-uniform solid concentration profiles were observed in the 12" column for the 90-115 μm size silica, with settling of the slurry on the distributor plate. Smaller size particles were more uniformly distributed.

Heat transfer coefficients were determined in the 12" column for paraffin/silica slurries, using seven vertical, tubular heat transfer elements. The values were in good agreement with Deckwer's correlation for the large and medium size particles, but were lower for the 0.5-5 μm size range.

The bubble size diameter probe was successfully calibrated using a strobe technique, and data acquisition will begin next quarter.

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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 a 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 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 advantage 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 in 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 36months and is divided into four major tasks. This report describes the work accomplished during the ninth quarter.

2.0 OBJECTIVE

The overall objective of this program is to evaluate 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. This task was completed in the first quarter.

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 effect 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 bench scale slurry phase Fischer-Tropsch reactor.

3.0 SUMMARY AND CONCLUSIONS

3.1 Task 2 - Slurry Catalyst Development

3.1.1 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.

3.1.2 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 Task 3 - Slurry Reactor Design Studies

In the 5" cold flow simulator (CFS), gas holdup and solid concentration measurements were obtained for the water/air/silicon oxide system. In the 12" CFS, gas holdup and solid concentration profile measurements were obtained for the isoparaffin/N_2/silicon oxide system, with and without heat transfer internals. In both the 5" and 12" CFS, values of gas holdup were close to those predicted by the two-phase Akita and Yoshida correlation, except for the low gas holdup observed with slurries of the smallest 0.5-5 μm particles. In both the 5" and 12" CFS, gas holdup was found to be a function mainly of gas velocity and, to a lesser extent, particle size.

Solid concentration profiles in both CFS's were uniform for the 0.5-5 μm silicon oxide. In the 12" CFS, very non-uniform profiles, with settling on the distributor plate, were observed for the large size silica in isoparaffin. The profiles were much less pronounced in water with no settling, and this difference is greater than can be predicted by density effects.

Heat transfer coefficients were determined in the 12" CFS for isoparaffin/N_2/silica slurries, with vertical tubular heat transfer elements. For the large and medium size silica, the values were in good agreement with those predicted by Deckwer, but were lower for the small size particles. Additional slurry viscosity data is expected to better correlate the data.

The bubble diameter probe is presently being calibrated using a repeating strobe technique. The probe is expected to begin data acquisition in January.

4.0 ACKNOWLEDGEMENTS

The contributions to this program by C. B. A. Freed, P. A. Greene, J. M. LaBar, M. Louie, S. E. Madison, M. L. Morris, S. Motika and L. E. Schaffer are gratefully acknowledged.

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5.0 RESULTS AND DISCUSSION

5.1 Task 2 - Slurry Catalyst Development

5.1.2 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."

5.2 Task 3 - Slurry Reactor Design Studies

5.2.1 5" Cold Flow Simulator

(i) Gas Holdup

Gas holdup measurements were obtained for the water/air/silicon oxide system, and are shown in Tables 1, 2 and 3 and Figures 1, 2 and 3 for the 0.5-5, 45-53 and 90-115 μm particle sizes, respectively. As in previous experiments in isoparaffin, the 0.5-5 μm silica does show the lowest gas holdups of all the particles studied. However, unlike the isoparaffin runs, a decrease in gas holdup with weight loading was not observed for these runs. The gas holdup values are consistent with the other 0.5-5 μm water measurements obtained at $V_L = 0$, reported in the April-June 1982 Quarterly Report. Comparing the figures, the effect of particle size appears negligible. The effect of solid loading on gas holdup, while observable for the 45-53 μm particles, is not observed in the larger or smaller size silica. It is also clear from the tables that ε_G has little or no dependence on slurry velocity.

(ii) Solid Concentration Profiles

Solid concentration profiles are shown in Figures 4 and 5 for the 45-53 μm silicon oxide system, and in Figure 6 for the 90-115 μm silicon oxide system. Solid concentration profiles for

the 0.5-5 μm silicon oxide system were shown to be uniform over the range of gas and liquid velocities studied. This is consistent both with the previous runs at $V_L = 0$ and with predictions based on settling velocity.

The larger sizes' concentration profiles are much less pronounced than was observed in the isoparaffin/silicon oxide systems. This difference is greater than can be accounted by density differences between the water and isoparaffin systems.

Although the solid concentration profiles are slight, the 45-53 μm silicon oxide system clearly shows that the slurry velocity plays an important role in evenly distributing the solid particles throughout the bed, with the most pronounced profiles at zero liquid velocity. For a slurry column with internal cooling, therefore, smaller particles, i.e. in the range of 50 μm, need to be used than for a slurry column with external cooling.

The effects of slurry velocity on concentration profile uniformity are observed to a greater extent with the 90-115 μm silica/water system. However, the situation is quantitatively different when using isoparaffin. As previously reported for the 5" system, and described later in this report for the 12" cold flow simulator, much of the larger particle size silica remained unsuspended in the isoparaffin slurry, staying on the bottom of the bubble column, an unacceptable mode of operation.

5.2.2 12" Cold Flow Simulator

(i) Gas Holdup

Gas holdup measurements were obtained for the isoparaffin/nitrogen/silicon oxide system, both without heat transfer internals (shown in Table 4 and Figure 7) and with heat transfer internals (shown in Table 5 and Figure 8). The average gas holdup value was obtained using the expanded and settled bed height. The intermediate values between adjacent sample ports were obtained using manometer tubes. Differences between these were probably due to experimental error in determining the expanded bed height.

Missing values of intermediate gas holdups were due to limitations in using the manometer tube method (described in the April-July 1982 Quarterly Report). To overcome this difficulty, a different scheme is being installed using a mercury manometer to measure differential pressure.

For a given superficial velocity, the gas volumetric flow rate is less with heat transfer internals because of the lower cross sectional area. Table 5 includes five identical runs of the midpoint condition, as required by the box Behnken experimental design, and it can be seen that reproducibility is good.

With no heat transfer internals, of the five independent variables studied in these eight runs: gas and slurry velocity, solid particle size, weight fraction and distributor hole size, only gas velocity and particle size had any significant effect on gas holdup when statistically analyzed. Rerunning the analysis with just gas velocity and particle size yielded the following preliminary, dimensional correlation:

$$\varepsilon_G/(1-\varepsilon_G)^4 = 0.34\ V_G^{0.165} D_p^{0.152} \qquad R^2 = 0.88 \qquad (1)$$

where V_G = superficial gas velocity, ft/sec
D_p = solid particle diameter, μm

With insertion of the heat transfer internals, the column hydraulic diameter,

$$d_n = 4\ A_x/\rho$$

where d_n = hydraulic diameter, cm
A_x = cross sectional area, cm^2
ρ = wetted perimeter, cm

is decreased from 12" to just under 4". This diameter is considered the minimum diameter, at least for an empty bubble column, that wall effects can be ignored. Thus, it is not surprising that the

Akita and Yoshida correlation is still followed. This would not be the case, though, for the finned transfer internals that will be used in later runs, where the column hydraulic diameter will be only 1.33". In that case, a higher gas holdup would be expected.

With heat transfer internals, the results did not change drastically. The gas holdups are greater for the large size particles at a given gas velocity in Figure 8. This is partly because of the small percentage of large size particles that are actually suspended, and partly due to the higher viscosity of the small size slurry. The small size silica is below the Akita and Yoshida correlation[1], as was seen in the 5" cold flow simulator for both isoparaffin and water. The large size silica agrees with the Akita and Yoshida correlation, as it did in the 5" cold flow simulator. In these nineteen runs, only gas velocity and particle size had any significant effect on gas holdup when statistically analyzed.

(ii) Solid Concentration Profiles

Solid concentration profiles were obtained for each of the experimental runs in Tables 4 and 5, and are shown in Figures 9, 10 and 11 for no heat transfer internals, and Figures 12, 13 and 14 with plain heat transfer internals, for the 0.5-5 μm, 45-53 μm and 90-115 μm silicon oxide cases, respectively. The average weight percent in the table is an arithmetic average. Behavior with and without heat transfer internals was identical. As previously reported for the 5" column, the 0.5-5 μm silicon oxide displayed a uniform profile, regardless of liquid or gas velocities. The large size silica showed very non-uniform concentration profiles in isoparaffin, as it did in the 5" cold flow simulator. However, because 1) in the 5" CFS, the lowest sample port was 3.5" above the distributor, while in the 12" CFS it was only 0.75" above the distributor, and 2) the 12" CFS has a greater solid inventory, it was possible to actually sample the settled solid in the 12" CFS. Therefore, the solid weight fraction at the lowest port is much greater in the 12" CFS than in the 5" CFS.

Figures 11 and 14 indicate that, apart from the amount of settled solid, very similar steady state concentration profiles were obtained, regardless of the initial average weight fraction of the slurry. Also, when the total amount of solids in the column was low enough, the liquid and gas were able to suspend virtually all of the solid. Therefore, the maximum amount of ~90 μm solid that could be suspended under these conditions was about 20 wt%.

(iii) Heat Transfer

Heat transfer measurements, using two 19" heaters, were made for the isoparaffin/nitrogen/silicon oxide system, with plain heat transfer internals, and are shown in Table 6 and Figure 15. For each run, three heat transfer coefficients are reported, along with the heat transfer coefficient predicted by Deckwer[4]. Heater A is located inside the center heat transfer internal about 10' above the distributor plate, or about 2/3 of the distance between the distributor and the top of the bubble column. Two temperatures are measured at the heater midpoint, 150° circumferentially from each other. Heater C, 5' above the distributor, is located on one of the six outer tubes with the surface thermocouple oriented toward the column center. The coefficient determined by heater C in runs 16 to 29, however, was about 6% higher than that of heater A. This was due to the fact that one wattage value was used for both heaters, while heater C had a slightly lower electrical resistance. This caused heater C to have, in reality, a larger wattage than "A".

In Figure 15, the measured heat transfer coefficients are plotted against the values predicted by Deckwer's[4] correlation:

$$St = B(Re^{1/2}FrPr)^{1/2} \qquad (2)$$

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Slurry properties were determined using the following relationships:

$$Cp_{SL} = w_S Cp_S + w_L Cp_L \quad (3)$$

$$k_{SL} = \frac{2k_L + k_S - 2v_S(k_L - k_S)}{2k_L + k_S + v_S(k_L - k_S)} k_L \quad (4)$$

$$\rho_{SL} = v_S \rho_S + v_L \rho_L \quad (5)$$

$$\mu_{SL} = 1.4 \text{ cp} \quad (6)$$

Deviations from Deckwer's correlation for the small size silica, shown in Figure 15, may be due in part to variations in the slurry viscosity. Kolbel[5] quoted a value of 200 BTU/hr ft^2 °F as being fairly constant within the churn turbulent regime.

Nomenclature

B = 0.12 for sand
= 0.1 for other solids
Cp = specific heat
d = diameter
Fr = $V_G/(g\ d_S)^{1/2}$ = Froude number
g = gravitational acceleration
h = heat transfer coefficient
k = thermal conductivity
Pr = $\mu_{SL} Cp_{SL}/k_{SL}$ = Prandtl number
Re = $d_S V_G \rho_{SL}/\mu_{SL}$ = Reynolds number
St = $h/\rho_{SL} Cp_{SL} V_G$ = Stanton number
V = superficial velocity
v = volume fraction
w = weight fraction

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Greek Letters

ρ = density

μ = viscosity

Subscripts

G = gas

L = liquid

S = solid

SL = liquid-solid (slurry)

6.0 EXPERIMENTAL

6.1 Task 2 - Slurry Catalyst Development

6.1.2 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 Task 3 - Slurry Reactor Design Studies

6.2.1 12" Cold Flow Simulator

(i) Bubble Diameter

A bubble diameter calibration chamber was constructed to:

(1) compare the actual bubble diameter to the trace produced by the probe for a two phase system,

(2) compare the actual bubble rise velocity with that observed by the probe, and

(3) determine the smallest bubble that the probe can see before surface tension effects cause the bubble to go around the probe instead of through it.

The calibration chamber is a 5" plexiglass cube with openings for different distributors and for the bubble diameter probe. Calibration of the probe has been hampered by unsatisfactory probe resilience. Several alternative solutions to overcome this problem are being pursued.

(ii) Slurry Viscosity

Analysis of the slurry viscosity data is awaiting more data at intermediate particle sizes and weight loadings.

(iii) Heat Transfer

The 19" cartridge heater was calibrated twice in water and once in air. The water tests, Curves A and B in Figure 16, showed that a large heat leak occurring 8 inches from the heater midpoint had negligible effect upon the temperature reading at midpoint. Differences in curves A and B were due to differing amount of water shear across the heater. The air test, Curve C in Figure 16, showed that the heater was heating uniformly. From the three calibration tests, it can be concluded that heat transfer coefficients can be accurately determined (to within 10%) with the 19" cartridge heater.

7.0 REFERENCES

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

4. Deckwer, W. D., et al, Ind. Eng. Chem. Proc. Des. Dev., 1980, 19, 699.

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8.0 FIGURES

FIGURE 1

5 INCH COLD FLOW SIMULATOR

WATER, 0.5–5 μM SILICON OXIDE, AIR

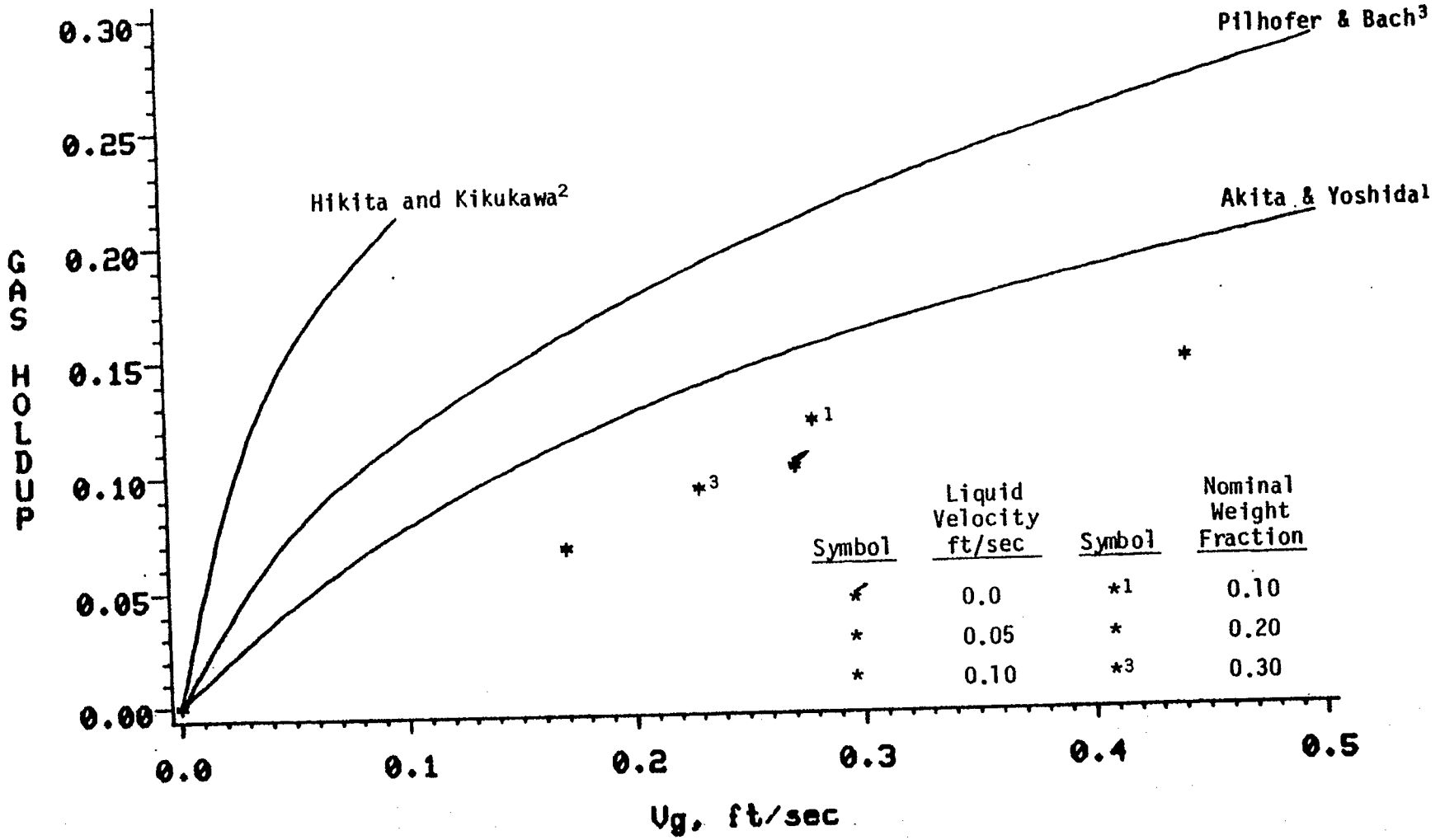


FIGURE 2

5 INCH COLD FLOW SIMULATOR

WATER, 45-53 μM SILICON OXIDE, AIR

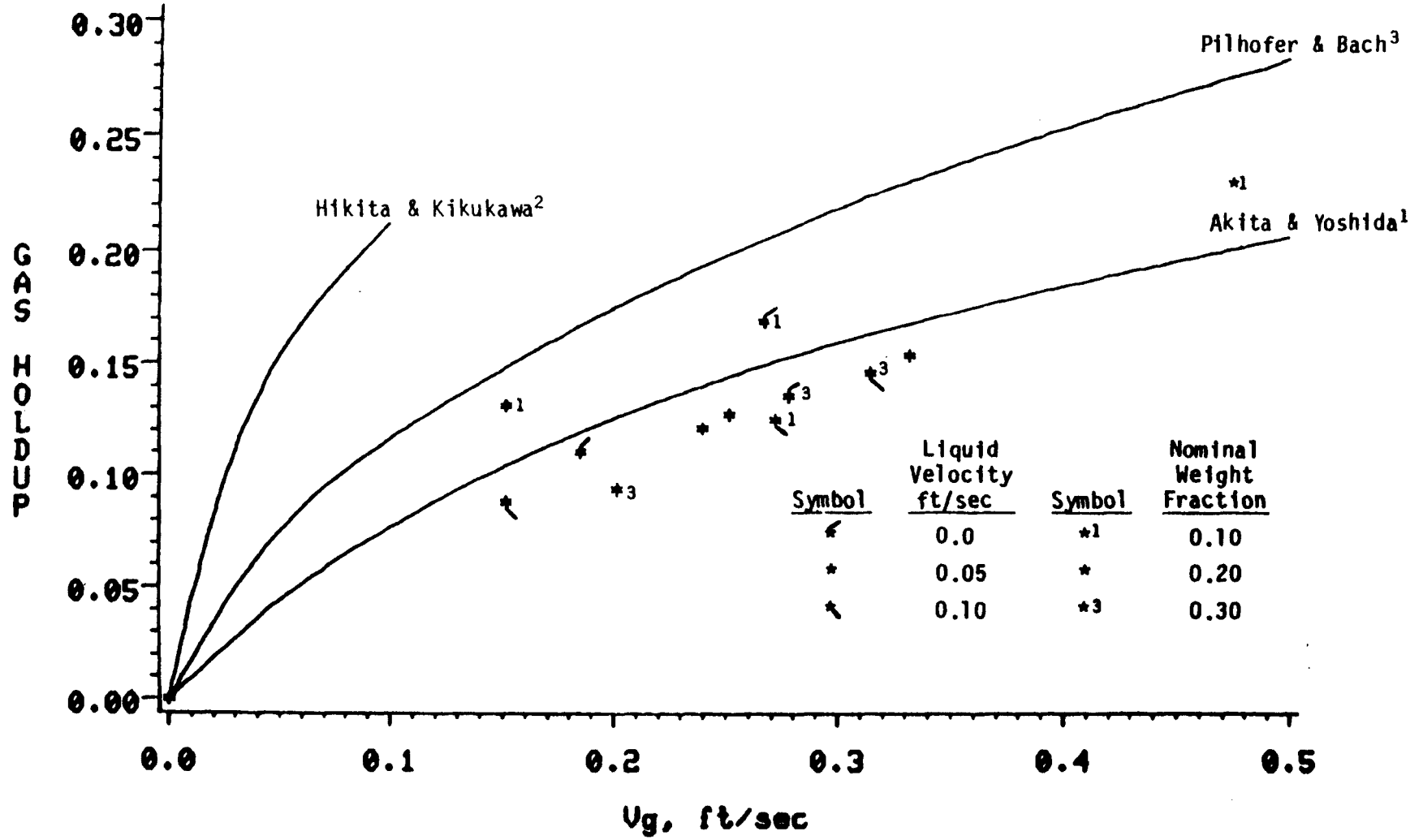


FIGURE 3

5 INCH COLD FLOW SIMULATOR

WATER, 90–115 μM SILICON OXIDE, AIR

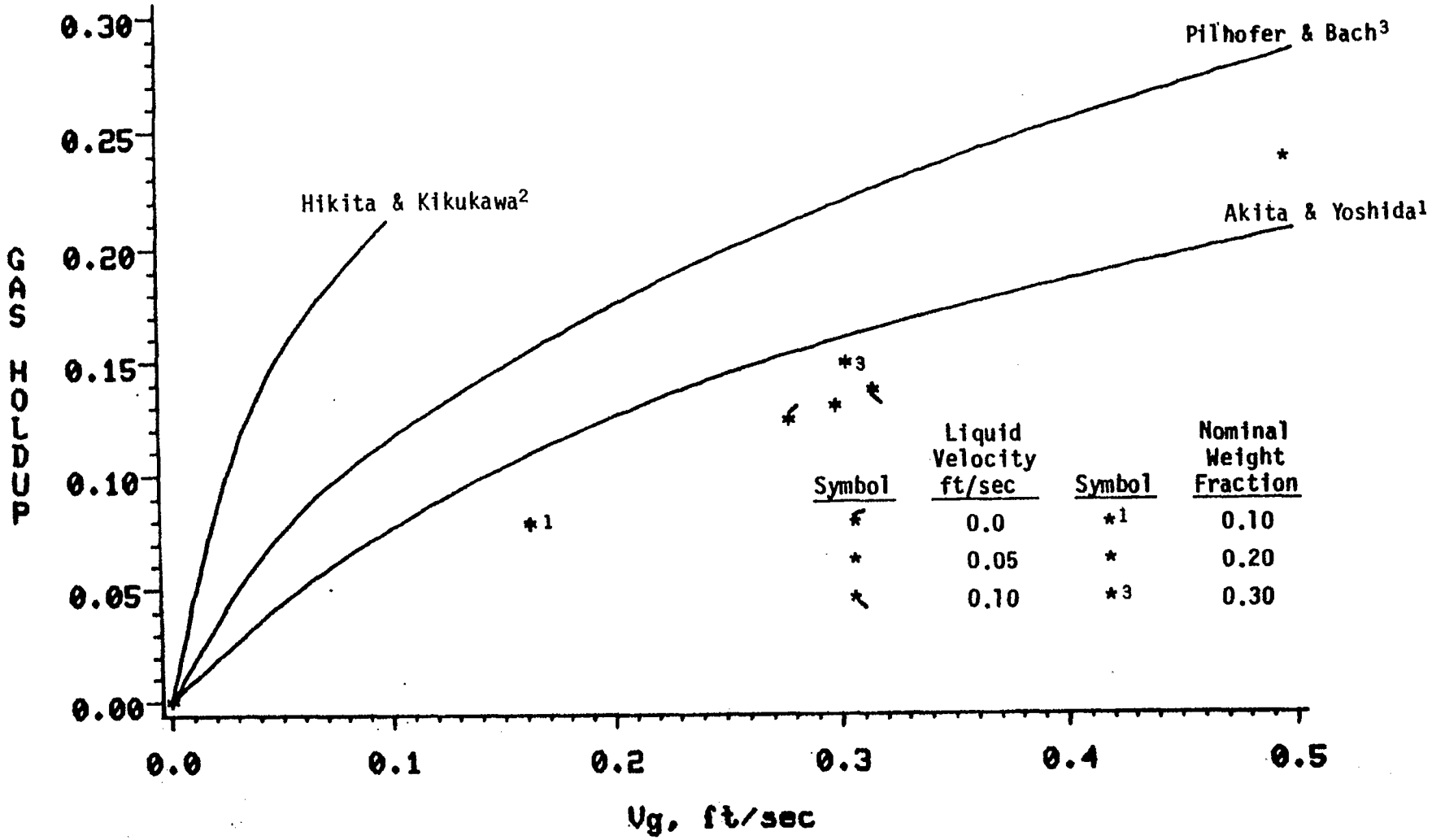


FIGURE 4

5 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES
WATER, 45-53 μM SILICON OXIDE, AIR

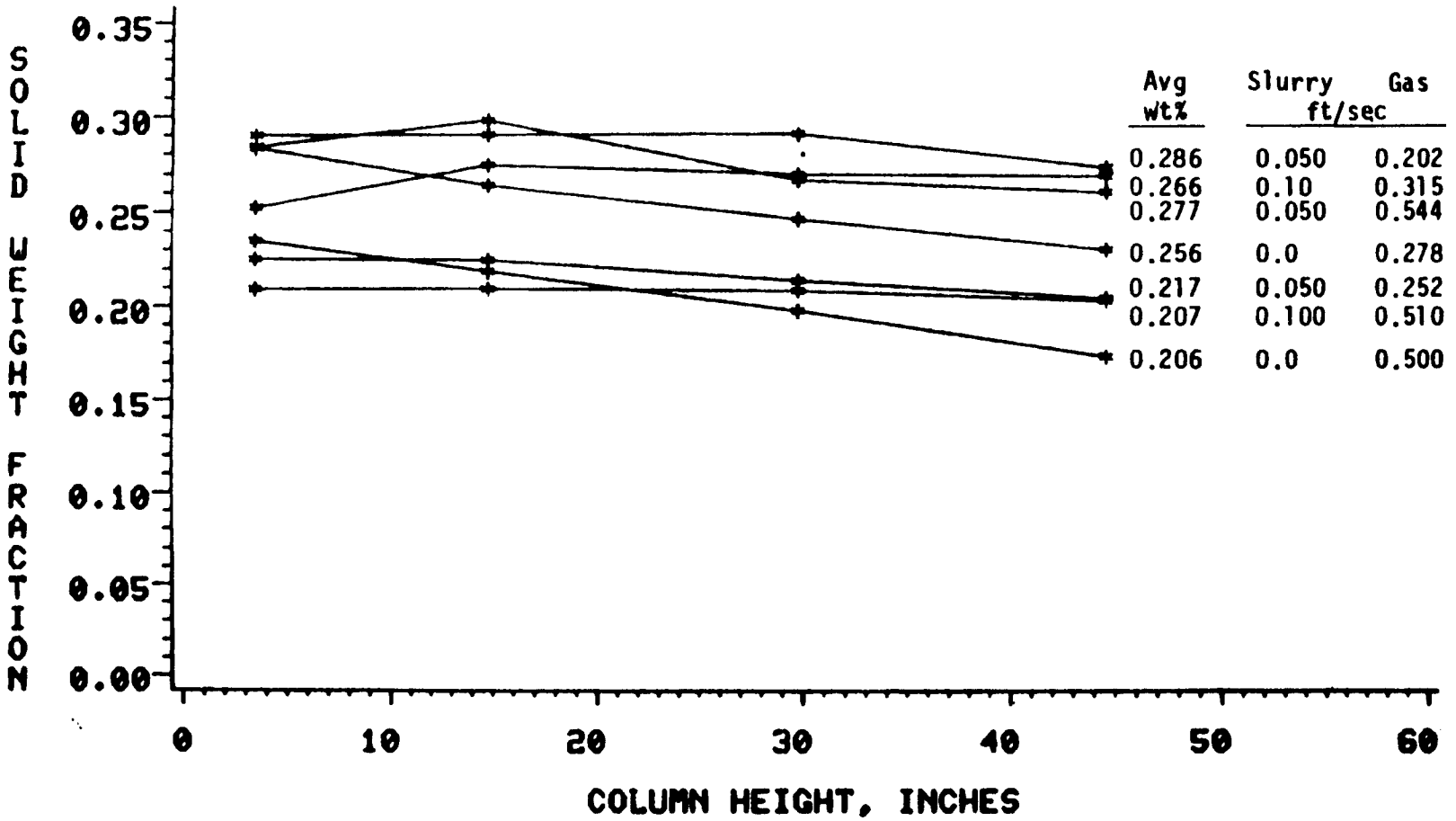


FIGURE 5

5 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES
WATER, 45-53 μM SILICON OXIDE, AIR

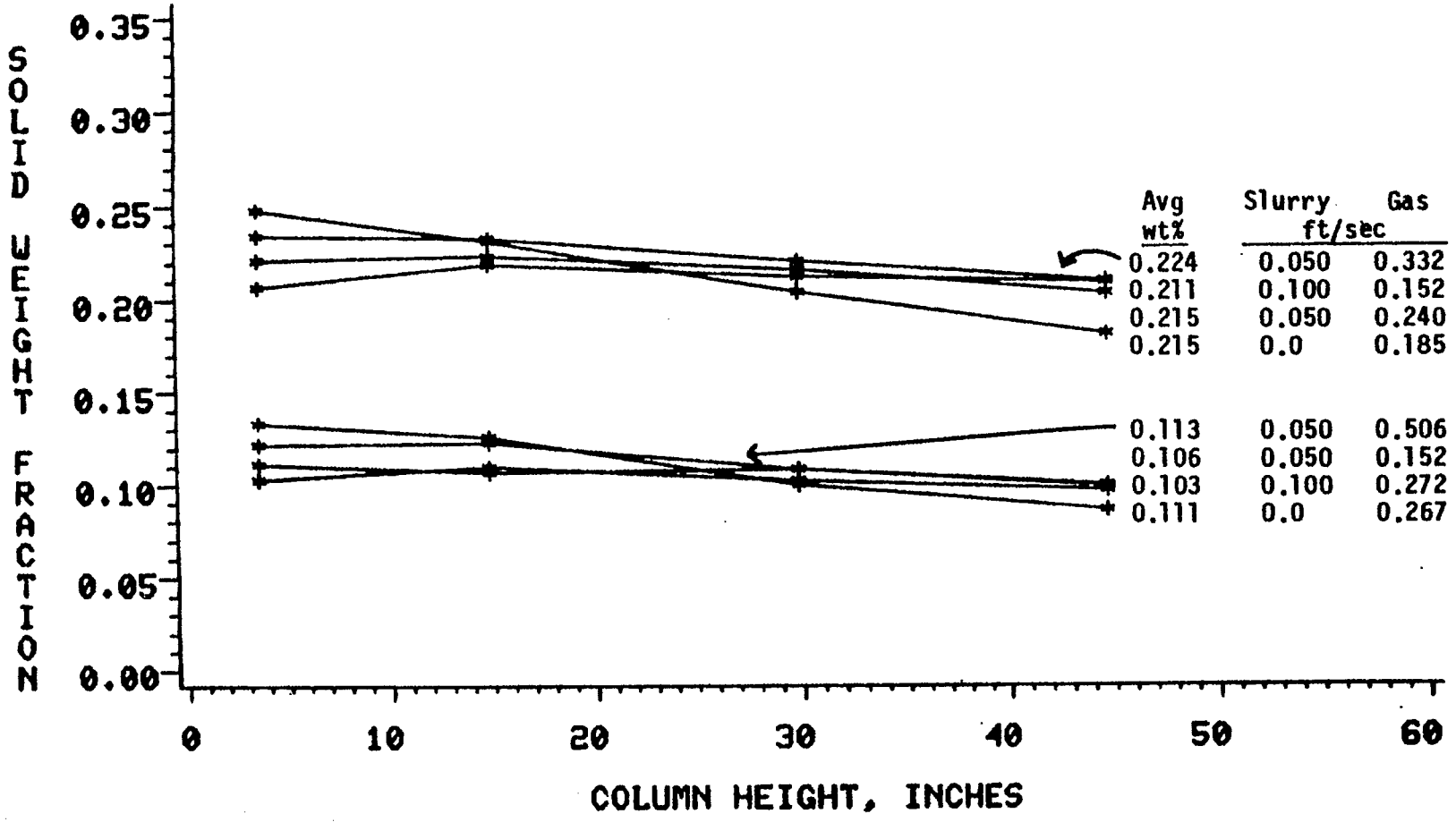


FIGURE 6

5 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES
WATER, 90-115 μM SILICON OXIDE, AIR

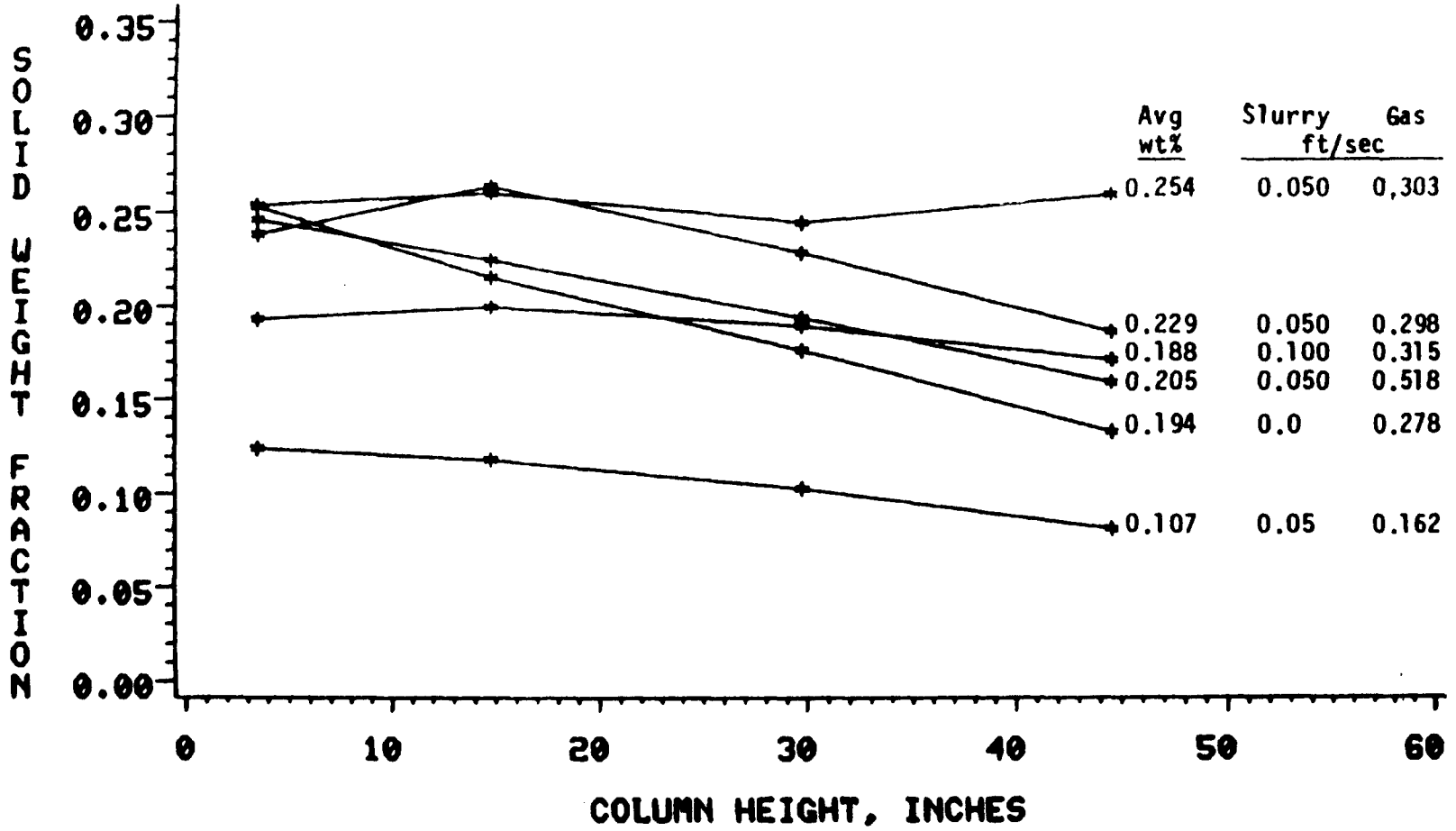


FIGURE 7

12 INCH COLD FLOW SIMULATOR

NO HEAT TRANSFER INTERNALS
ISOPARAFFIN, SILICON OXIDE, N2

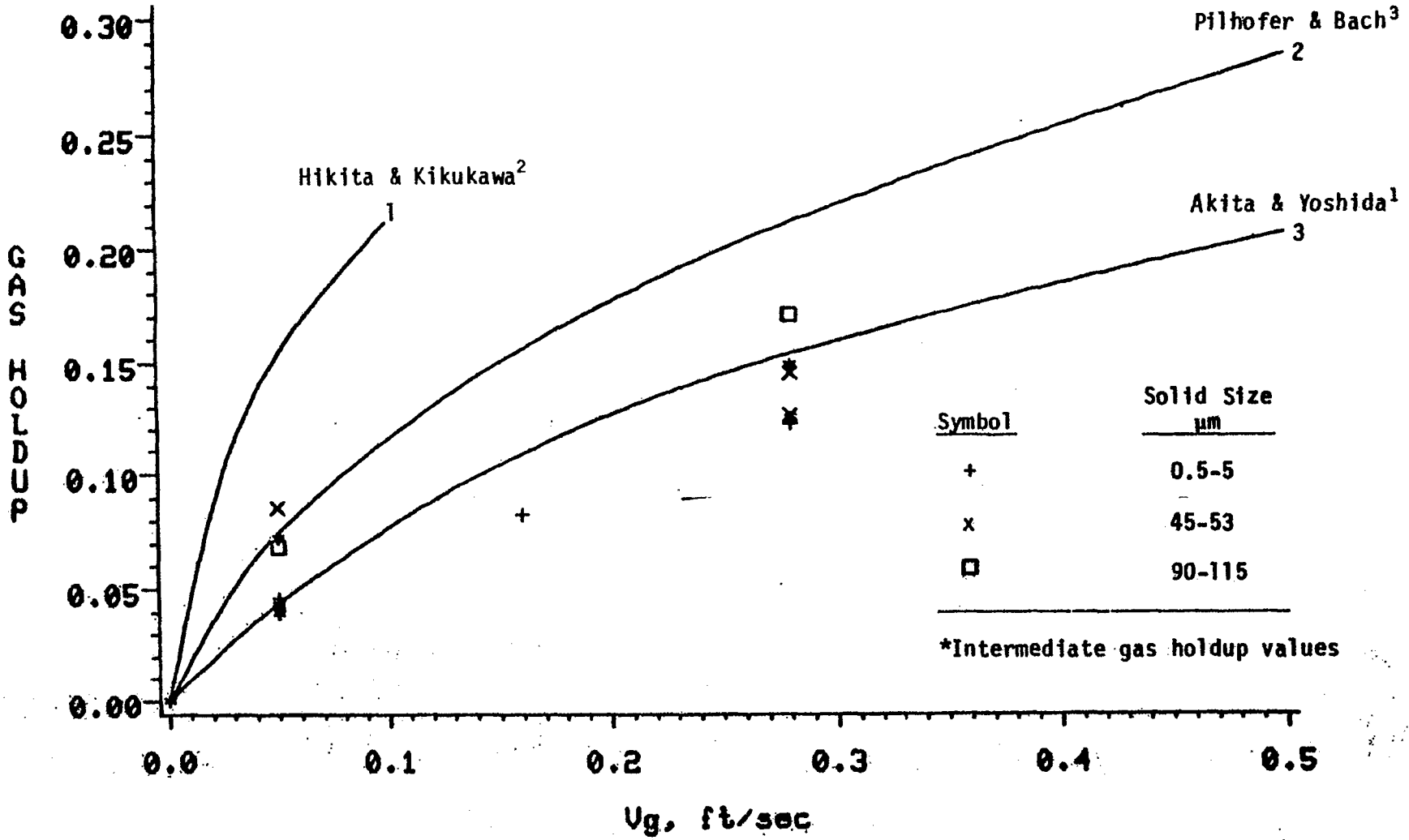


FIGURE 8

12 INCH COLD FLOW SIMULATOR

PLAIN HEAT TRANSFER INTERNALS
ISOPARAFFIN, SILICON OXIDE, N2

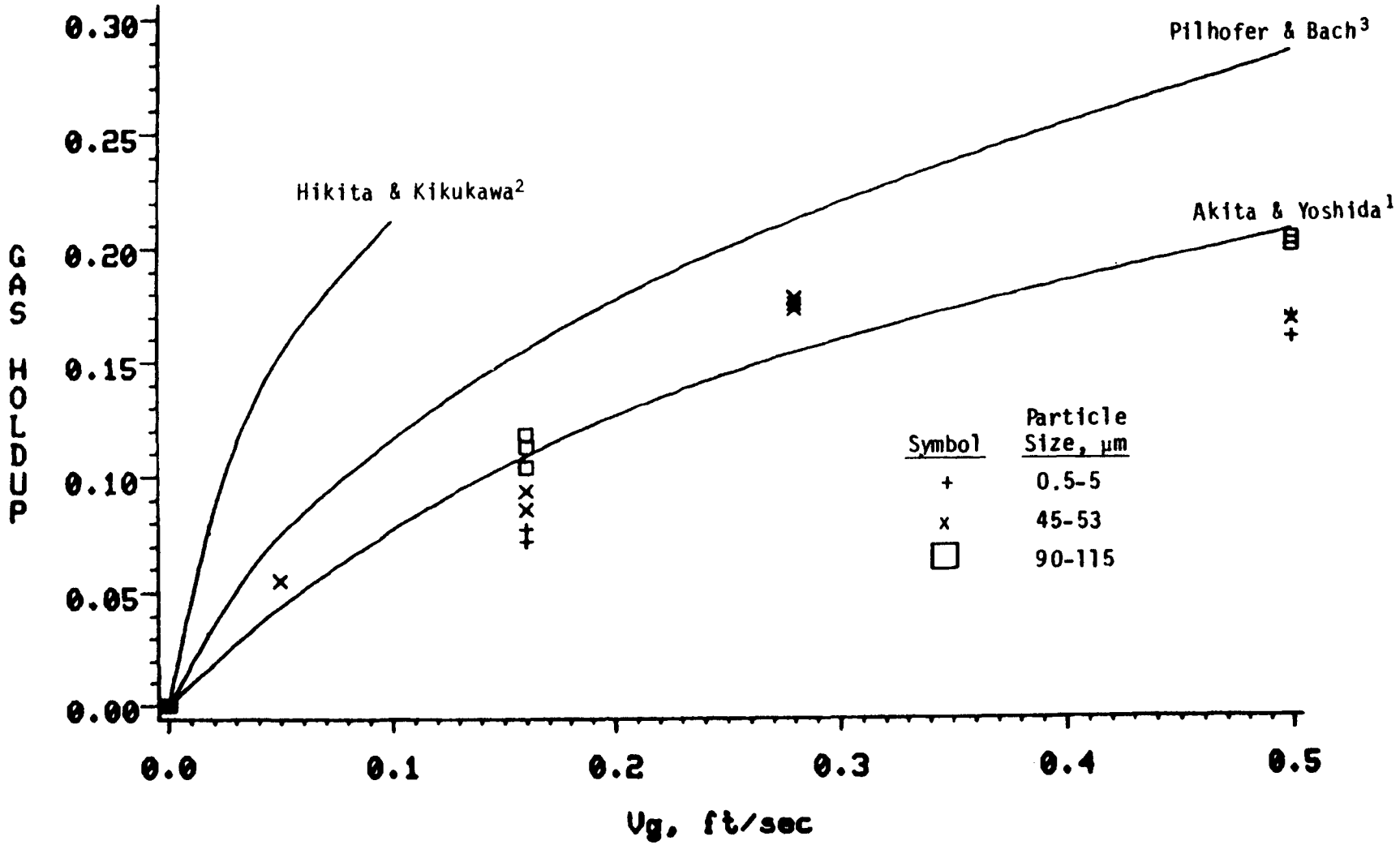


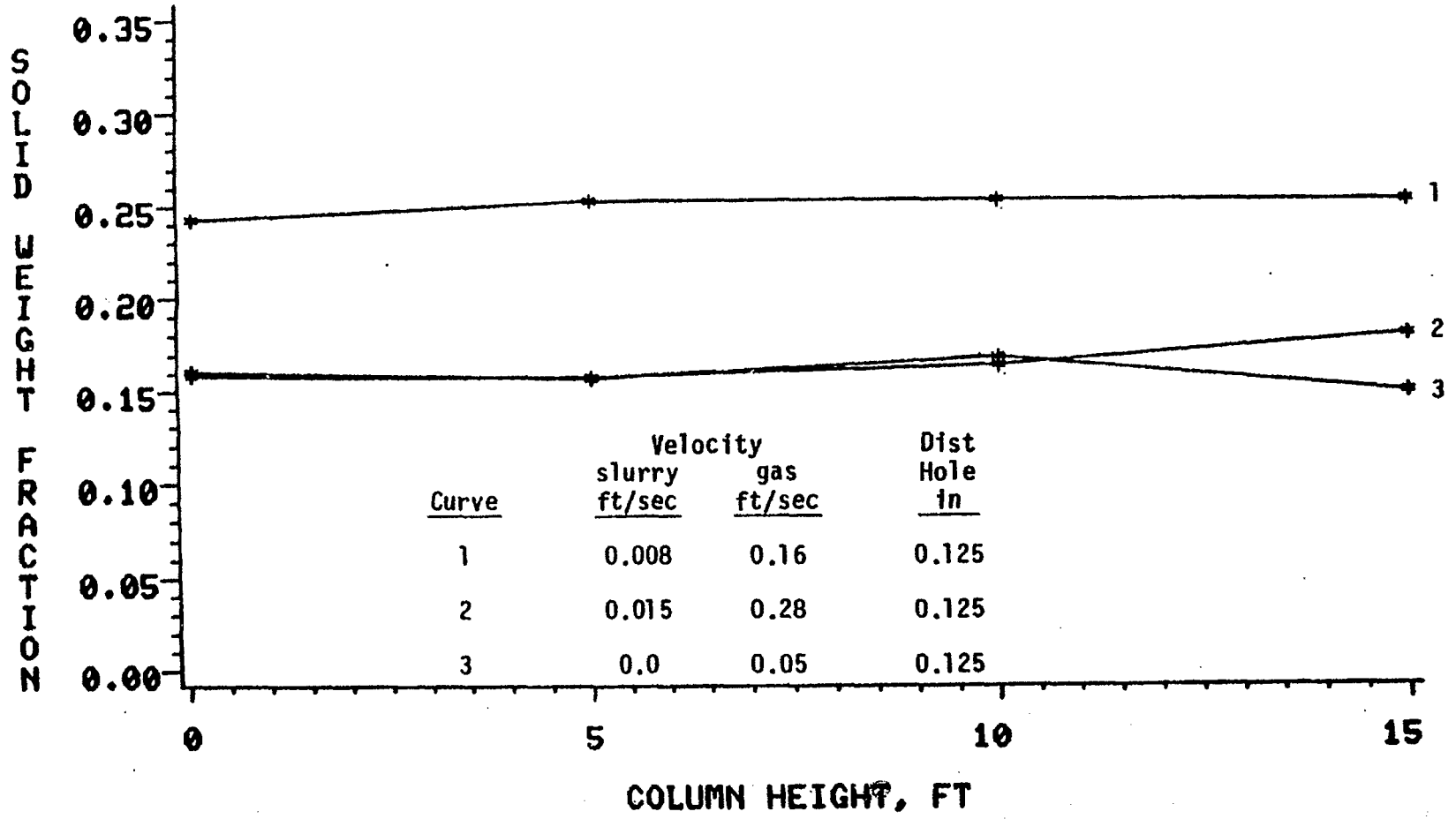
FIGURE 9

12 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES

ISOPARAFFIN, 0.5-5 μM SILICON OXIDE, N2

NO HEAT TRANSFER INTERNALS



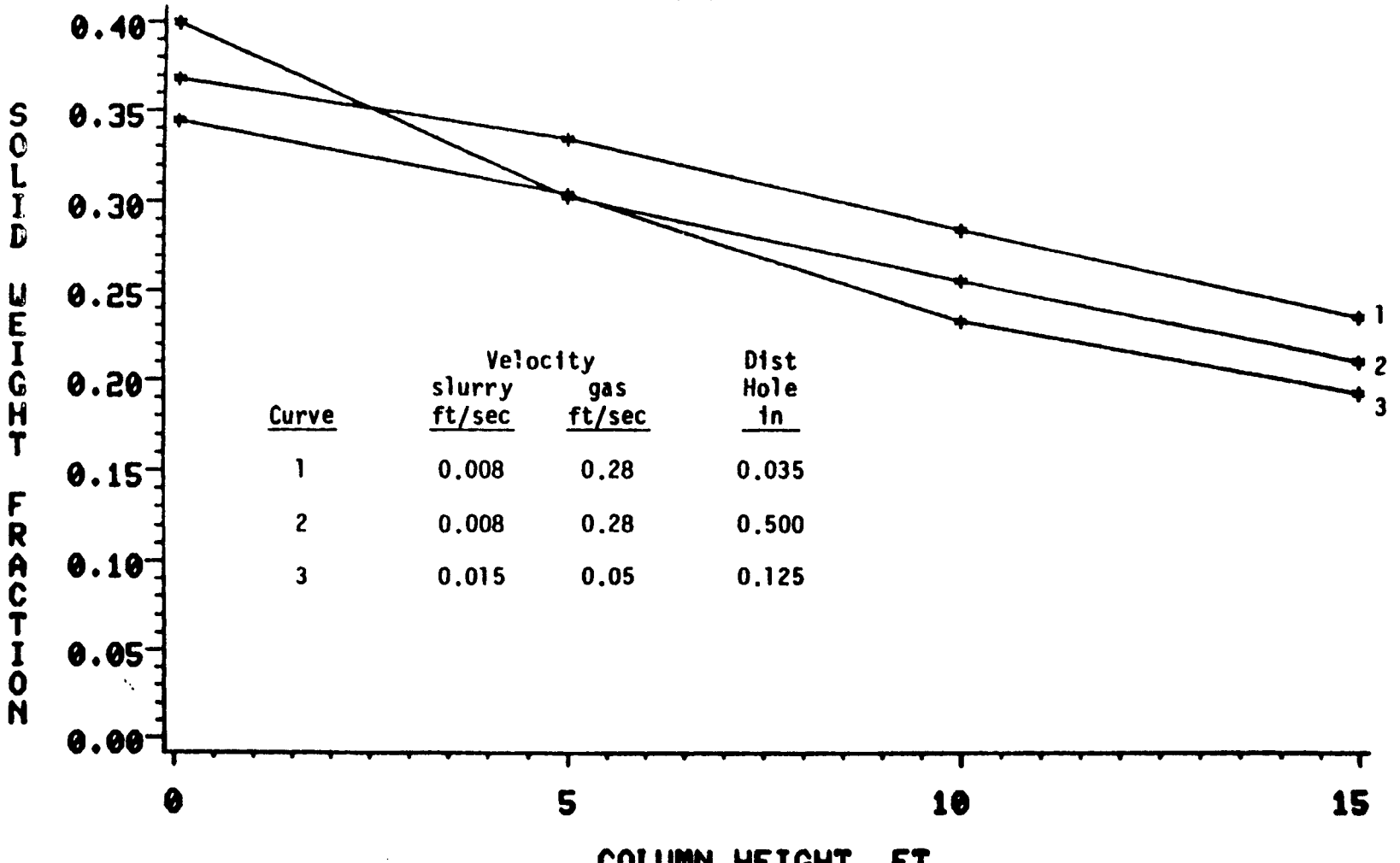
Curve	Velocity slurry ft/sec	Velocity gas ft/sec	Dist Hole in
1	0.008	0.16	0.125
2	0.015	0.28	0.125
3	0.0	0.05	0.125

FIGURE 10

12 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES
ISOPARAFFIN, 45-53 μM SILICON OXIDE, N2

NO HEAT TRANSFER INTERNALS



Curve	Velocity slurry ft/sec	Velocity gas ft/sec	Dist Hole in
1	0.008	0.28	0.035
2	0.008	0.28	0.500
3	0.015	0.05	0.125

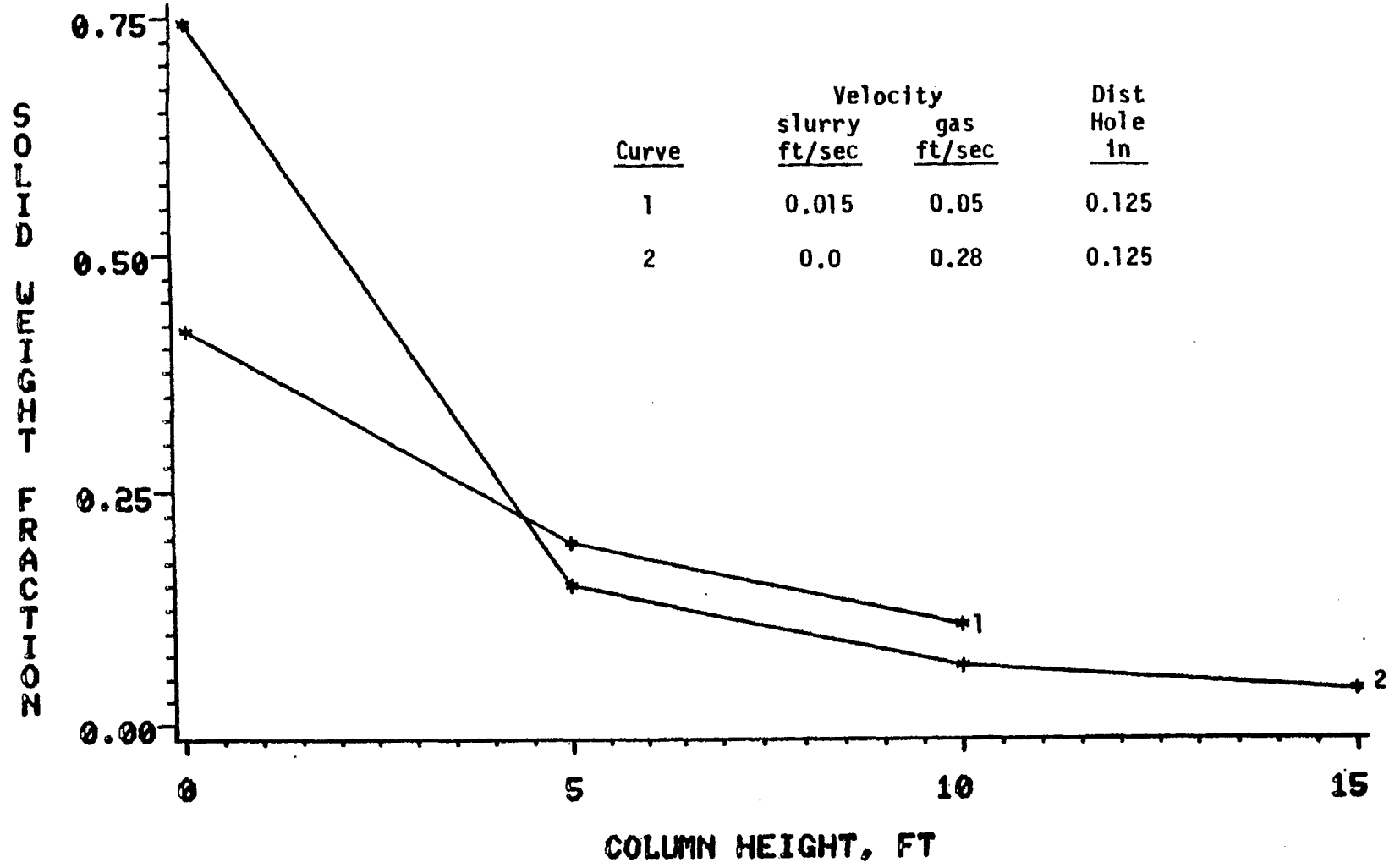
FIGURE 11

12 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES

ISOPARAFFIN, 90-115 μM SILICON OXIDE, N2

NO HEAT TRANSFER INTERNALS



Curve	Velocity slurry ft/sec	Velocity gas ft/sec	Dist Hole in
1	0.015	0.05	0.125
2	0.0	0.28	0.125

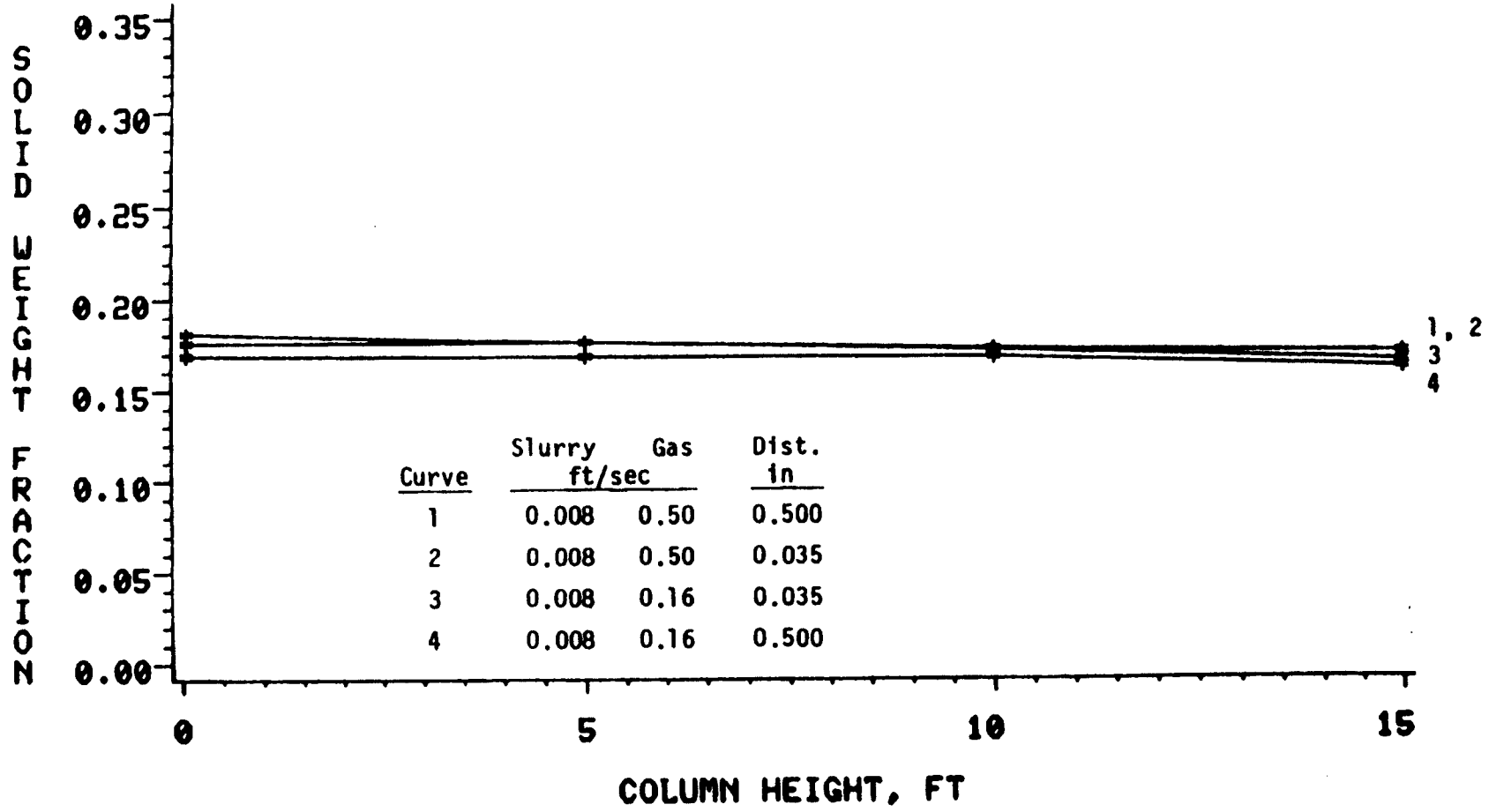
FIGURE 12

12 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES

ISOPARAFFIN, 0.5–5 μM SILICON OXIDE, N2

PLAIN HEAT TRANSFER INTERNALS



Curve	Slurry ft/sec	Gas ft/sec	Dist. in
1	0.008	0.50	0.500
2	0.008	0.50	0.035
3	0.008	0.16	0.035
4	0.008	0.16	0.500

FIGURE 13

12 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES

ISOPARAFFIN, 45–53 μM SILICON OXIDE, N2

PLAIN HEAT TRANSFER INTERNALS

DISTRIBUTOR HOLE SIZE = 0.125"

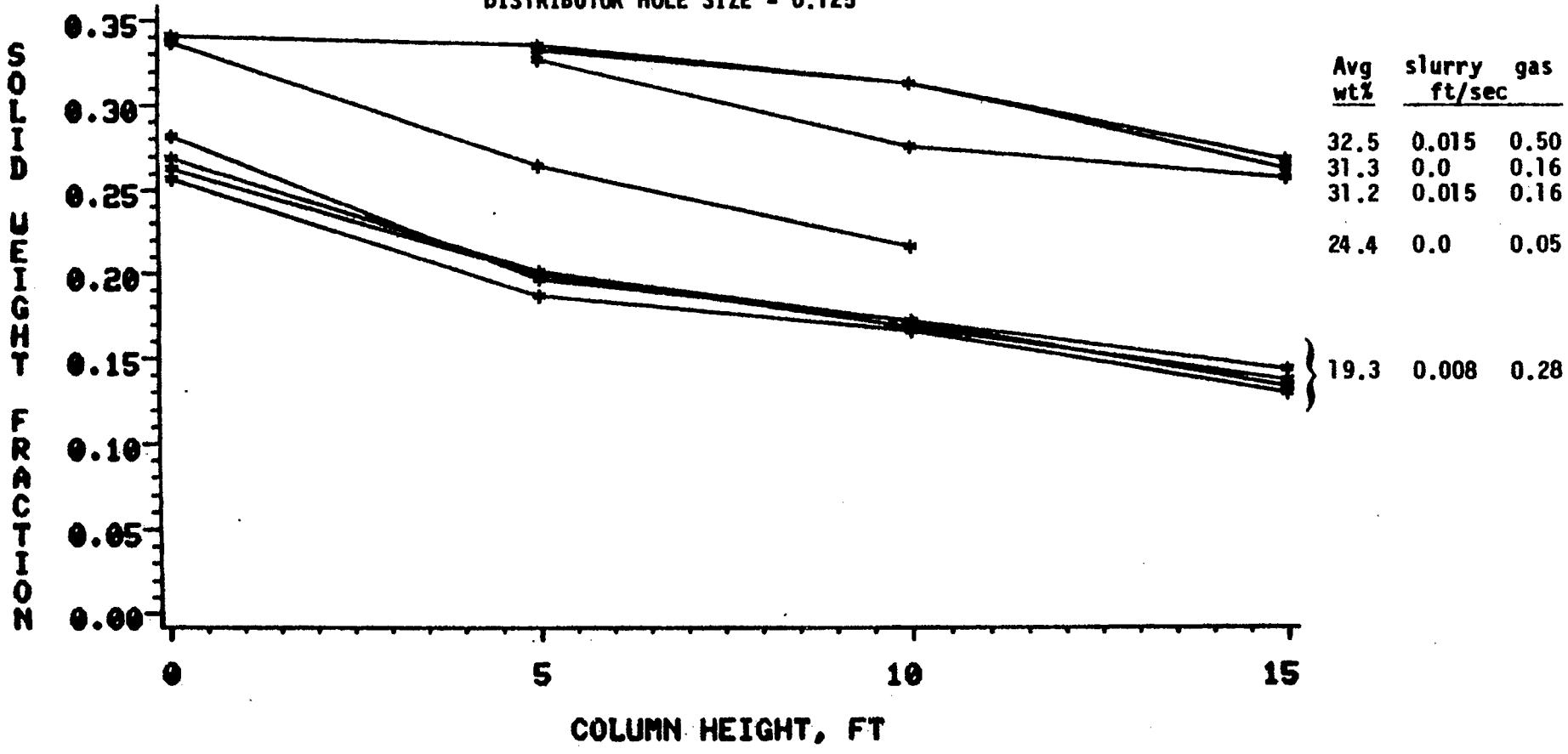


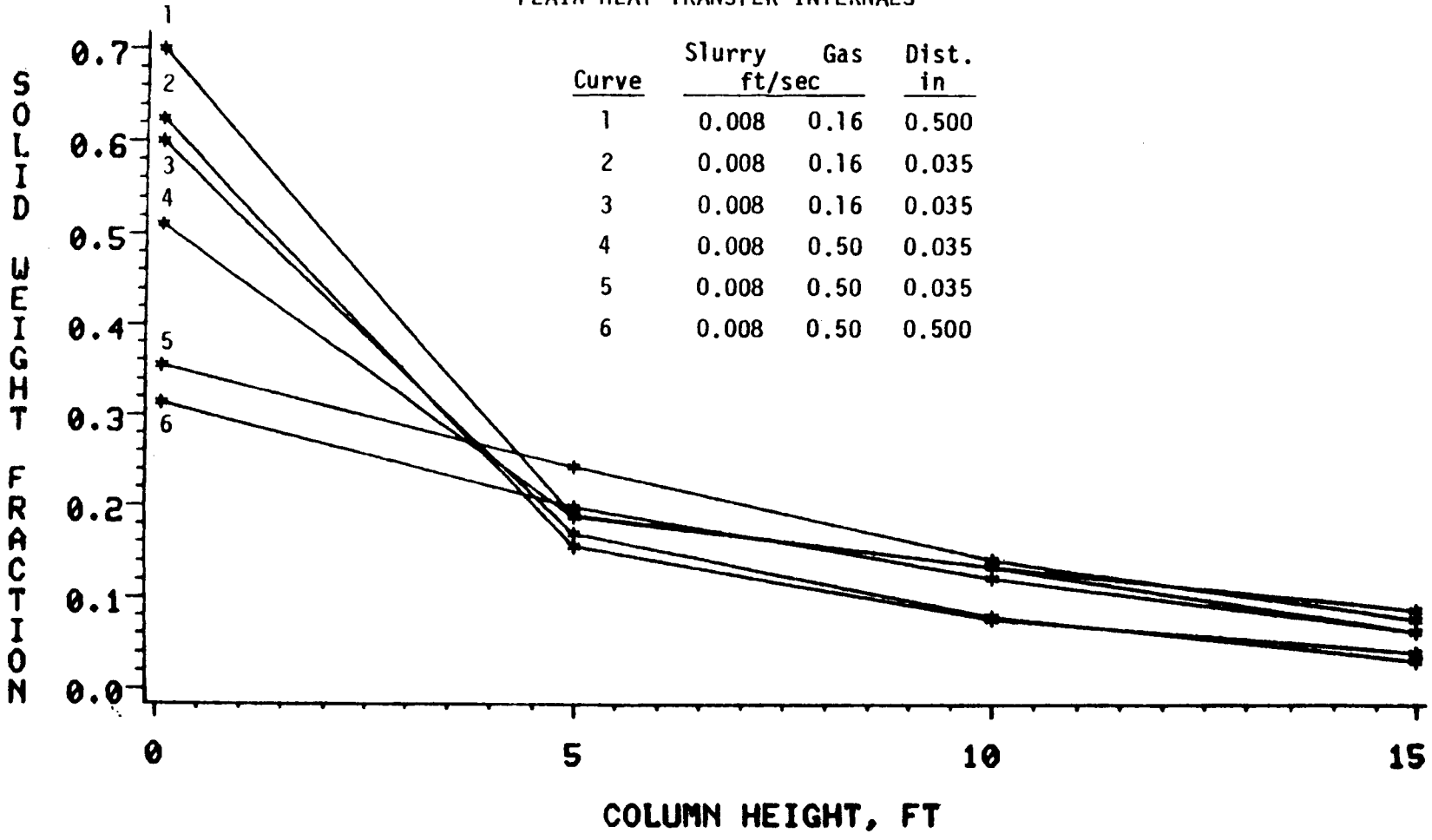
FIGURE 14

12 INCH COLD FLOW SIMULATOR

SOLID CONCENTRATION PROFILES

ISOPARAFFIN, 90–115 μM SILICON OXIDE, N2

PLAIN HEAT TRANSFER INTERNALS



Curve	Slurry ft/sec	Gas ft/sec	Dist. in
1	0.008	0.16	0.500
2	0.008	0.16	0.035
3	0.008	0.16	0.035
4	0.008	0.50	0.035
5	0.008	0.50	0.035
6	0.008	0.50	0.500

FIGURE 15

12 INCH COLD FLOW SIMULATOR

Heat Transfer Coefficients
Plain Heat Transfer Internals
Isoparaffin, Silicon Oxide, N2

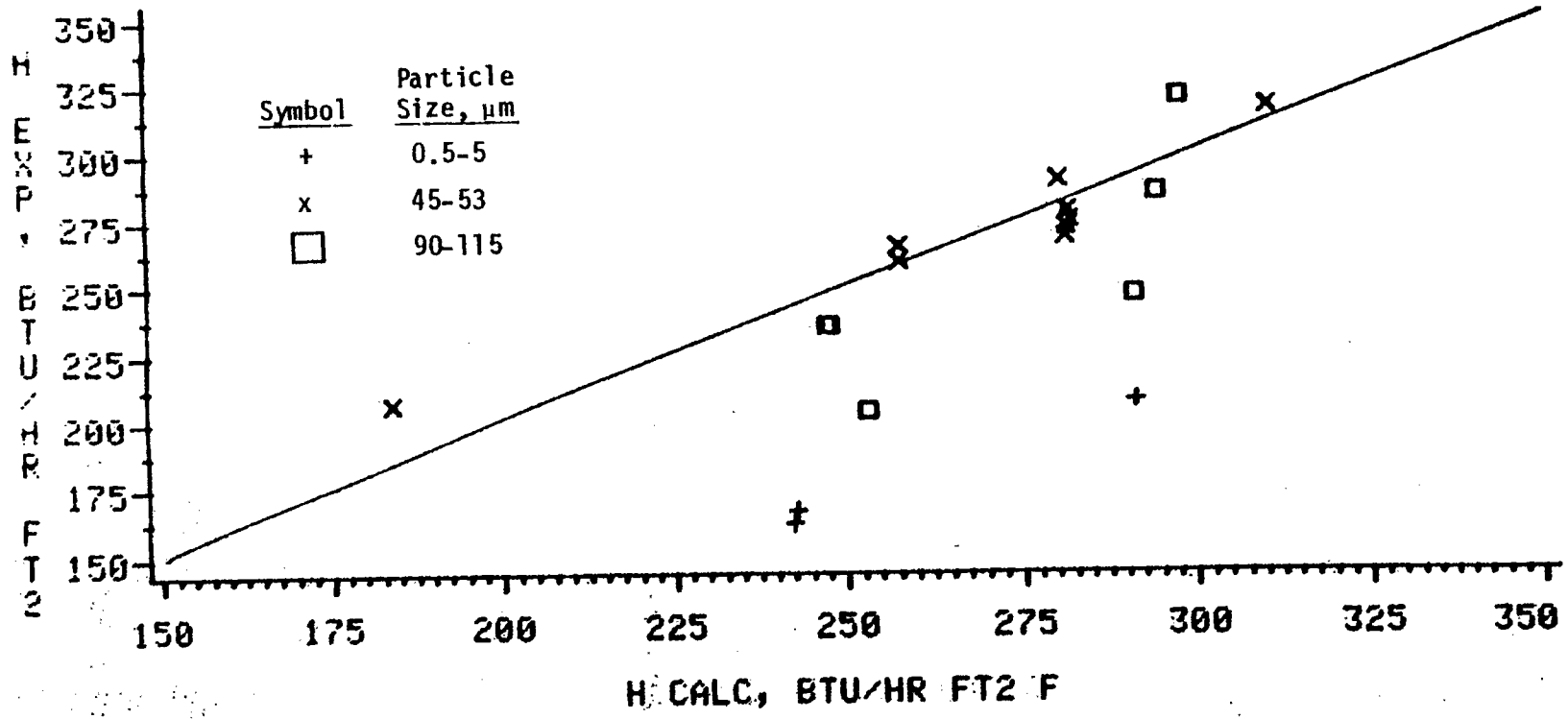
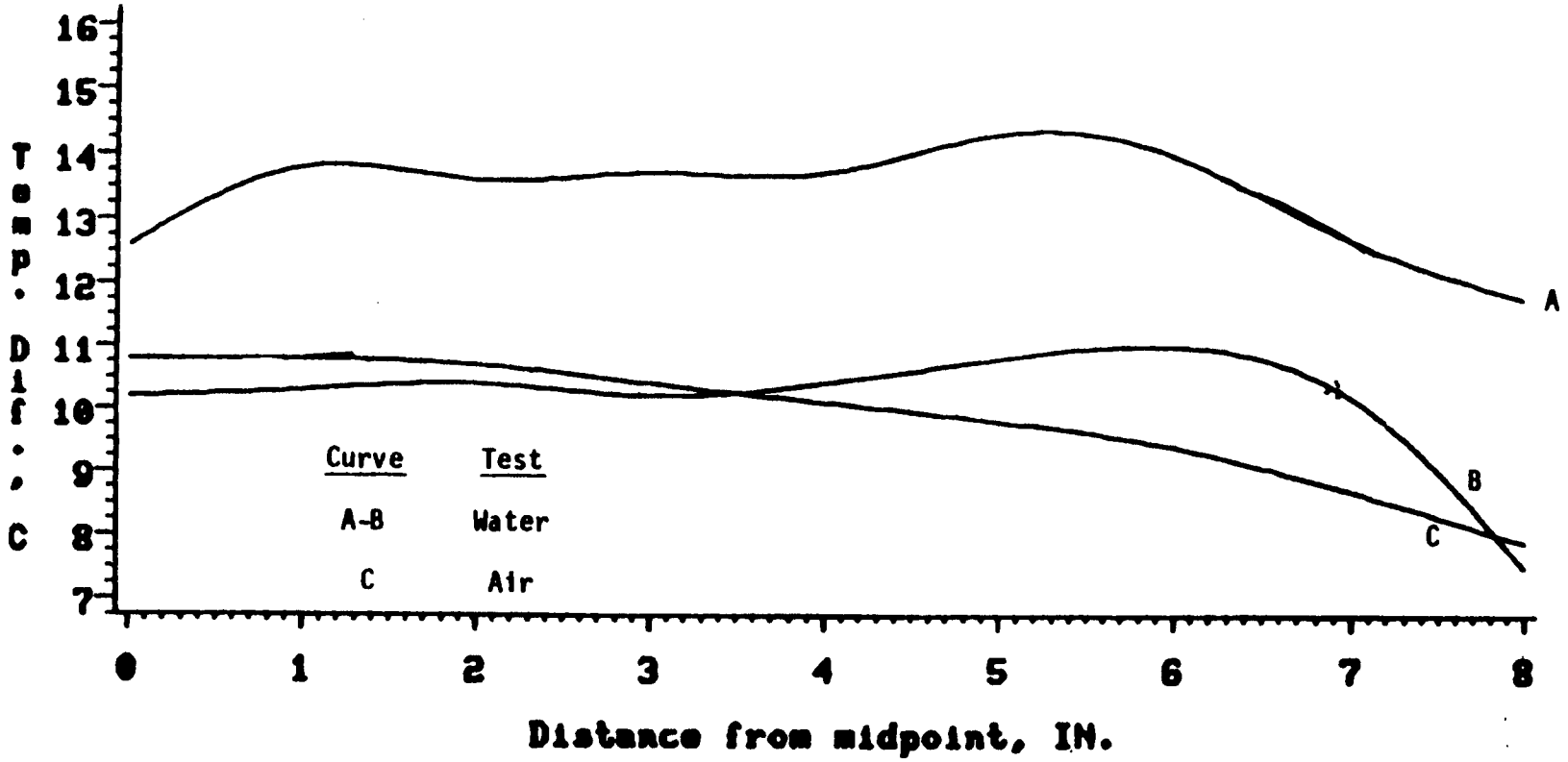


FIGURE 16

19" Heater Tests



Air Products

9.0 TABLES

TABLE 1

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM: THREE PHASE

LIQUID- WATER

SOLID- 0.5-5 μM SILICON OXIDE

GAS- AIR

RUN	VG FT/SEC	VL FT/SEC	EG EXP	EG23 EXP	EG34 EXP	EG45 EXP	WEIGHT FRACTION
7258-14-1	0.280	0.050	0.121	0.109	0.109	0.121	0.100
7258-15-1	0.230	0.050	0.092	0.080	0.080	0.088	0.300
7258-16-1	0.170	0.050	0.067	0.075	0.061	0.066	0.200
7258-17-1	0.272	0.0	0.101	0.114	0.088	0.091	0.200
7258-18-1	0.294	0.100	0.063	0.060	0.045	0.066	0.200
7258-19-1	0.442	0.050	0.146	0.133	0.130	0.134	0.200

TABLE 2

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM: THREE PHASE

GAS- AIR

LIQUID- WATER

SOLID- 45-53 μM SILICON OXIDE

RUN	VG FT/SEC	VL FT/SEC	EG EXP	EG23 EXP	EG34 EXP	EG45 EXP	WEIGHT FRACTION
7258-20-1	0.152	0.050	0.131	0.130	0.146	0.140	0.106
7258-21-1	0.267	0.0	0.169	0.145	0.162	0.169	0.111
7258-22-1	0.272	0.100	0.125	0.155	0.139	0.129	0.103
7258-23-1	0.506	0.050	0.221	0.260	0.260	0.222	0.113
7258-24-1	0.185	0.0	0.110	0.083	0.096	0.087	0.215
7258-25-1	0.152	0.100	0.088	0.094	0.094	0.097	0.211
7258-26-1	0.332	0.050	0.154	0.145	0.134	0.143	0.224
7258-27-1	0.240	0.050	0.121	0.116	0.105	0.118	0.215
7258-28-1	0.252	0.050	0.127	0.130	0.114	0.123	0.217
7258-29-1	0.500	0.0	0.186	0.164	0.180	0.172	0.206
7258-30-1	0.510	0.100	0.196	0.191	0.202	0.195	0.207
7258-31-1	0.202	0.050	0.094	0.133	0.066	0.093	0.286
7258-32-1	0.278	0.0	0.136	0.109	0.094	0.121	0.256
7258-33-1	0.315	0.100	0.146	0.152	0.126	0.141	0.266
7258-34-1	0.544	0.050	0.200	0.193	0.199	0.192	0.277

TABLE 3

GAS HOLDUP: 5" COLD FLOW SIMULATOR

SYSTEM: THREE PHASE

GAS- AIR

LIQUID- WATER

SOLID- 90-115 μM SILICON OXIDE

RUN	VG FT/SEC	VL FT/SEC	EG EXP	EG23 EXP	EG34 EXP	EG45 EXP	WEIGHT FRACTION
7258-35-1	0.162	0.050	0.079	0.075	0.075	0.075	0.107
7258-36-1	0.298	0.050	0.130	0.106	0.087	0.118	0.229
7258-37-1	0.278	0.0	0.124	0.089	0.097	0.106	0.194
7258-38-1	0.315	0.100	0.137	0.106	0.125	0.139	0.188
7258-39-1	0.518	0.050	0.208	0.060	0.187	0.213	0.205
7258-40-1	0.303	0.050	0.149	0.098	0.106	0.139	0.254

TABLE 4

GAS HOLDUP AND SOLID FRACTION : 12" COLD FLOW SIMULATOR

NO HEAT TRANSFER INTERNALS

SYSTEM:THREE PHASE

GAS- NITROGEN

LIQUID- ISOPARAFFIN

SOLID- SILICON OXIDE

RUN NO.	DIST HOLE IN	SOLID SIZE µM	SOLID AVG WT%	VELOCITY SLURRY FT/SEC	VELOCITY GAS FT/SEC	GAS HOLDUP 1-2 VOL %	GAS HOLDUP 2-3 VOL %	GAS HOLDUP 3-4 VOL %	GAS HOLDUP AVG VOL %	SOLID FRACTION 1 WT %	SOLID FRACTION 2 WT %	SOLID FRACTION 3 WT %	SOLID FRACTION 4 WT %
8	0.125	2.5	25.0	0.008	0.16	.	.	.	8.3	24.3	25.2	25.3	25.4
9	0.125	2.5	15.8	0.0	0.05	4.0	4.3	.	4.6	16.1	15.5	16.8	14.9
10	0.125	2.5	16.5	0.015	0.28	12.5	12.5	.	12.2	15.9	15.6	16.4	18.1
11	0.500	50.0	29.4	0.008	0.28	.	.	.	12.6	40.7	30.2	25.6	21.2
12	0.035	50.0	30.6	0.008	0.28	.	.	.	14.5	36.8	33.4	28.5	23.7
13	0.125	50.0	26.9	0.015	0.05	.	.	.	8.6	34.5	30.4	23.3	19.5
14	0.125	98.0	25.0	0.015	0.05	.	7.3	.	6.9	74.5	15.2	6.5	3.9
15	0.125	98.0	18.1	0.0	0.28	.	14.9	.	17.1	41.9	19.8	10.9	.

TABLE 5

GAS HOLDUP AND SOLID FRACTION: 12" COLD FLOW SIMULATOR

PLAIN HEAT TRANSFER INTERNALS

SYSTEM: THREE PHASE

GAS- NITROGEN

LIQUID- ISOPARAFFIN

SOLID- SILICON OXIDE

RUN NO.	DIST HOLE IN	SOLID SIZE μM	SOLID AVG WT%	VELOCITY SLURRY FT/SEC	VELOCITY GAS FT/SEC	GAS HOLDUP 1-2 VOL %	GAS HOLDUP 2-3 VOL %	GAS HOLDUP 3-4 VOL %	GAS HOLDUP AVG VOL %	SOLID FRACTION 1 WT %	SOLID FRACTION 2 WT %	SOLID FRACTION 3 WT %	SOLID FRACTION 4 WT %
16	0.125	50.0	24.4	0.0	0.05	.	.	.	5.5	33.7	26.4	21.6	.
17	0.125	50.0	32.5	0.015	0.50	.	.	.	16.9	38.9	33.1	31.2	26.9
18	0.125	50.0	31.2	0.015	0.16	.	.	.	8.6	39.0	32.6	27.5	25.8
19	0.125	50.0	31.3	0.0	0.16	.	.	.	9.4	34.1	33.4	31.2	26.4
20	0.125	50.0	19.2	0.008	0.28	.	.	18.0	17.7	26.3	19.9	16.8	13.8
21	0.125	50.0	18.5	0.008	0.28	.	.	18.0	17.5	25.7	18.6	16.6	13.0
22	0.125	50.0	19.8	0.008	0.28	.	.	18.1	17.5	28.2	19.6	17.2	14.4
23	0.125	50.0	19.4	0.008	0.28	.	.	17.8	17.3	26.9	20.1	17.1	13.4
24	0.125	50.0	19.6	0.008	0.28	.	.	18.2	17.7	28.0	19.6	17.2	13.8

TABLE 5 (cont'd)

GAS HOLDUP AND SOLID FRACTION: 12" COLD FLOW SIMULATOR

PLAIN HEAT TRANSFER INTERNALS

SYSTEM: THREE PHASE

GAS- NITROGEN

LIQUID- ISOPARAFFIN

SOLID- SILICON OXIDE

RUN NO.	DIST HOLE IN	SOLID SIZE μM	SOLID AVG WT%	VELOCITY SLURRY FT/SEC	VELOCITY GAS FT/SEC	GAS HOLDUP 1-2 VOL %	GAS HOLDUP 2-3 VOL %	GAS HOLDUP 3-4 VOL %	GAS HOLDUP AVG VOL %	SOLID FRACTION 1 WT %	SOLID FRACTION 2 WT %	SOLID FRACTION 3 WT %	SOLID FRACTION 4 WT %
25	0.125	50.0	19.4	0.008	0.28	.	.	18.4	17.8	25.5	20.7	16.5	14.8
26	0.035	98.0	23.0	0.008	0.50	.	.	.	20.4	51.0	18.8	13.5	8.8
27	0.035	98.0	20.4	0.008	0.50	.	.	.	20.4	35.4	24.2	14.2	7.8
28	0.035	98.0	22.0	0.008	0.16	.	.	12.0	11.8	60.0	16.9	8.1	3.2
29	0.035	98.0	22.5	0.008	0.16	.	.	11.8	11.3	62.4	15.6	7.8	4.1
30	0.500	98.0	27.3	0.008	0.16	.	.	10.6	10.4	70.3	18.9	13.5	6.4
31	0.500	98.0	17.4	0.008	0.50	.	.	21.3	20.1	31.3	19.8	12.3	6.3
32	0.500	2.5	16.7	0.008	0.16	.	.	.	7.2	16.9	16.8	16.8	16.3
33	0.500	2.5	17.5	0.008	0.50	.	.	17.7	16.1	18.1	17.6	17.3	17.1
34	0.035	2.5	17.5	0.008	0.50	.	.	18.4	17.0	18.1	17.6	17.2	17.1
35	0.035	2.5	17.3	0.008	0.16	.	.	7.8	7.7	17.6	17.6	17.2	16.7

TABLE 6

SHELL-SIDE HEAT TRANSFER COEFFICIENTS: 12" COLD FLOW SIMULATOR

PLAIN HEAT TRANSFER INTERNALS

SYSTEM: THREE PHASE

GAS- NITROGEN

LIQUID- ISOPARAFFIN

SOLID- SILICON OXIDE

RUN NO.	DIST HOLE IN	SOLID SIZE M	SOLID AVG WT%	VELOCITY SLURRY FT/SEC	VELOCITY GAS	HEAT COEFF. A	HEAT COEFF. A BTU/HR	HEAT COEFF. C FT2	HEAT COEFF. DECKWER F
16	0.125	50.0	20.4	0.0	0.05	206.3	204.5	218.0	183.7
17	0.125	50.0	32.5	0.015	0.50	316.6	321.0	330.1	310.4
18	0.125	50.0	31.2	0.015	0.16	265.6	259.7	259.7	257.5
19	0.125	50.0	31.3	0.0	0.16	259.7	256.8	275.1	257.5
20	0.125	50.0	19.2	0.008	0.28	268.7	271.9	278.4	281.3
21	0.125	50.0	18.5	0.008	0.28	289.5	289.5	284.0	280.4
22	0.125	50.0	19.8	0.008	0.28	275.1	275.1	278.4	282.0
23	0.125	50.0	19.4	0.008	0.28	271.9	271.9	281.8	281.5
24	0.125	50.0	19.6	0.008	0.28	278.4	275.1	292.5	281.8
25	0.125	50.0	19.4	0.008	0.28	278.4	275.1	288.9	281.5
26	0.035	98.0	23.0	0.008	0.50	320.2	320.2	350.0	297.7
27	0.035	98.0	20.4	0.008	0.50	285.3	281.8	321.0	294.5
28	0.035	98.0	22.0	0.008	0.16	235.8	226.6	245.8	247.4
29	0.035	98.0	22.5	0.008	0.16	235.8	228.8	243.3	247.8
30	0.500	98.0	27.3	0.008	0.16	202.7	202.7	198.7	253.0
31	0.500	98.0	17.4	0.008	0.50	247.2	244.2	266.7	291.0
32	0.500	2.5	16.7	0.008	0.16	162.2	159.6	143.8	242.1
33	0.500	2.5	17.5	0.008	0.50	206.9	206.9	198.7	291.1
34	0.035	2.5	17.5	0.008	0.50	206.9	204.8	200.7	291.1
35	0.035	2.5	17.3	0.008	0.16	166.6	163.9	153.4	242.7

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