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Hydrodynamics of Fischer-Tropsch Synthesis in Slurry Bubble Column Reactors

Quarterly Technical Progress Report

for the period 1 June 1985 - 31 August 1985

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TABLE OF CONTENTS

I. Abstract

The installation of two stainless steel bubble columns (2" ID and 9.5" ID, 10 ft tall), and their shakedown using a mineral spirit and FT-300 wax as liquid mediums was completed. The shakedown of a large glass column (9" ID, 10 ft tall) also has been completed early in June.

The effect of temperature (T = 160-280°C), superficial gas velocity (u_g = 1 - 13 cm/s), and gas distributor type (40 μm sintered metal plate 1.85 mm and 4 mm single hole orifice plate) on the average gas holdup was studied in a small glass column (2" ID, 10 ft tall) using molten paraffin wax as a liquid medium. The hysterisis behavior on the ε_g vs. u_g diagram was observed for the first time with this type of liquid medium.

Some preliminary work on the measurement of axial gas holdup distribution was done in the small stainless steel column.

The work on photographic techniques for determination of bubble size distribution, and mapping of flow regimes was initiated.

II. Objective and Scope of Work

The overall objective of this contract is to determine effects of reactor geometry, distributor design, operating conditions (i.e., temperature and gas flow rate), and oxygenated compounds on hydrodynamics of slurry bubble column reactors for Fischer-Tropsch synthesis, using a hard paraffin wax as the liquid medium. To accomplish these objectives, the following specific tasks will be undertaken.

Task 1 - Project Work Plan

The objective of this task is to establish a detailed project work plan covering the entire period of performance of the contract, including estimated costs and manhours expended by month for each task.

Task 2 - Bubble Column Reactor Design/Construction

Two bubble columns made of borosilicate glass of approximately 2" ID and 9" ID, and 10 ft tall will be designed, and assembled for measurement of the gas hold-up, and the bubble size distribution. After the design, procurement of equipment and instrumentation, and construction of the unit is completed, a shakedown of test facilities will be made to verify achievement of planned operating conditions. During this period instruments will be calibrated.

Task 3 - Process Variable Studies

The objective of this task is to determine the effect of various system variables (e.g. gas flow rate, temperature, and addition of minor amounts of oxygenated compounds) on hydrodynamic properties using the two-bubble columns (2" and 9" ID) and different types of distributors. All experiments will be conducted using nitrogen at atmospheric pressure. It

is planned to determine the following hydrodynamic parameters: gas hold-up, flow regime characterization, bubble size distribution, and the gas-liquid interfacial area.

Task 4 - Correlation Development and Data Reduction

Correlations based on our experimental data, for prediction of average gas hold-up, and the gas-liquid interfacial area will be developed.

III. Summary of Progress

Shakedown of a large glass column (9" ID, 10 ft. tall) was completed using mineral spirit, and FT-300 wax. The installation of the two stainless steel columns, and their shakedown were accomplished in the early part of June.

The effect of operating conditions (temperature and gas flow rate), and gas distributor types (SMP and orifice plate distributors) on the average gas holdup was studied in a small glass bubble column (2" ID, 10 ft. tall) using a Fischer-Tropsch derived paraffinic wax designated FT-300 as the liquid medium. It was found that over a range of values of superficial gas velocity one can have two values for the average gas holdup, ε_g. The high values of ε_g are caused in part by existence of a stable foam layer on top of the liquid level, and this regime is called a "foamy" regime. This type of regime has been observed earlier by several investigators who used a paraffinic wax as the liquid medium in their studies. In the flow regime where lower values of ε_g are obtained ther is very little foam, and this flow regime is called a "nonfoamy" regime or a liquid circulation regime.

The start-up procedure determines which of the two regimes will be attained. Transition from the "foamy" to the "nonfoamy" regime occurs when u_g exceeds a certain critical value, and similarly transition from the "nonfoamy" to the "foamy" regime occurs as the superficial gas velocity drops below a certain critical value. These critical values of superficial gas velocity were transitions take place are influenced by temperature, and gas distributor design. The existence of this hysterisis type of behavior has not been observed in any of the previous studies with paraffin wax as the liquid medium. Similar type of behavior was observed with some

other gas-liquid systems (Maruyama et al., 1981).

There is a good agreement between average gas holdups obtained in our study, and the ones obtained by researchers at Mobil under similar conditions (i.e. operating conditions, same type of flow regime, distributor design, reactor geometry and the liquid medium).

It was found that Bach and Pilhofer's (1978) correlation provides excellent fit for the average gas holdups obtained in our study with the orifice plate distributors in the "nonfoamy" regime.

Preliminary measurements of the axial gas holdup distribution were made in the small stainless steel column using differential pressure method.

Work on photographic techniques for determining bubble size distribution, and mapping of flow regimes has been initiated.

IV. Detailed Description of Technical Progress

A. Task 1 - Project Work Plan

The work on this Task was completed during the first quarter of the project.

B. Task 2 - Bubble Column Reactor Design/Construction

Fabrication of all metal parts for both stainless steel columns (2" ID and 9.5" ID, 10 ft. tall) has been completed early in June, and the columns were installed. They have been subsequently tested for leaks using air and water, as well as nitrogen and FT-300 paraffin wax. All leaks were repaired and both columns are fully operational.

A leak that developed, during our tests with kerosene as the liquid medium (Quarterly report March -May, 1985), at the distributor level of the large glass column (9" ID, 10 ft. tall), was fixed by replacing a gasket. The column was then tested using a mineral spirit as the liquid medium at the temperature of 127°C, and superficial gas (air) velocity of 8 cm/s with the static liquid height of 190 cm. No leaks were detected during 6 hours of operation under these conditions. After this the column was filled up with the wax (static height of 1.5 m), and tested at temperatures between 230°C and 275°C by passing nitrogen at superficial gas velocity of about 5 cm/s, for approximately 3 hours. The test was terminated due to clogging of the vent line above the gas-liquid separator. No leaking of the liquid was observed during this test. The experimental set-up (Fig. 1) was modified by adding a scrubber (3) above the gas-liquid separator (2). The 2 inch pipe which connects the separator and the scrubber is electrically heated, as well as the scrubber which is filled up with a mineral spirit solvent. This was done to prevent clogging of the vent line, and to collect components of

the wax that evaporate from bubble columns at higher operating temperatures.

Further modifications and/or additions to the experimental apparatus will be done as required.

C. Task 3 - Process Variable Studies

1. Average Gas Holdup Measurements in a Small Bubble Column

The effects of superficial gas velocity (u_g = 0 - 13 cm/s), temperature (T = 160 - 280°C), and distributor design (sintered metal plate -SMP with the average pore size of approximately 40μm, 1.85mm and 4mm single orifice plates) on the average gas holdup were studied in a small glass bubble column (5.1 cm ID, and 305 cm tall). All experiments were done at atmospheric pressure using nitrogen as a gas, and the Fischer-Tropsch derived paraffinic wax FT-300 (also known as SH-105 Vestowax), with an average molecular weight of 730, as a liquid medium.

1a. Experimental Apparatus and Procedures

A schematic representation of the experimental set up is shown in Figure 1. The flow rate of nitrogen from the gas cylinders is measured and controlled by a mass flow meter FC2 (Brooks; Model 5816). The metered gas passes through a preheater PH (electrically heated U shaped tube), and its temperature is monitored by two thermocouples (one located after the preheater and one just below the distributor). The inlet temperature of nitrogen is manually controlled by a variable transformer. The mass flow meter calibration is checked daily using a wet test meter (Precision Scientific). The wax in storage tank (1) is heated to a temperature of about 150°C before it is transported to the column (BC2) through 1/2" inch tube using a slight nitrogen overpressure. The column is preheated to a temperature of 150°C, before the wax is transferred into the column, using

the heating tapes that are wound around the column circumference. The column is brought to a desired operating temperature by increasing gradually a set point on a temperature controller. The flow of nitrogen through the column is maintained during the entire preheating period. Temperature inside the column is measured by two thermocouples that are located 33 cm and 200 cm above the distributor. These thermocouples are placed into two thermowells made out of 3/16 inch tubing. The disengagement zone on the top of the column, tubes connecting the column and the gas liquid separator, and the separator are maintained at temperatures above the melting point of the wax (~ 110°C), to prevent solidification of any entrained liquid. The hot gas leaves separator through a 2 inch pipe and passes through a scrubber (3) filled with mineral spirit before it is vented to the atmosphere. The scrubber is used to recover components of wax that evaporate from the column, and it is kept at about 100°C.

The average gas holdup is calculated from visual observations of the expanded and static liquid heights, i.e.

$$\varepsilon_g = \frac{(H-H_s)}{H} \times 100 \text{ (Volumetric \%)} \qquad (1)$$

The first reading of the expanded liquid height is made 10-15 min after the desired column temperature and the gas flow rate are reached. These readings are repeated several times (minimum of 3 readings for a given set of operating conditions) with 15-20 min time intervals between the two successive measurements to ensure that the steady state is achieved. The was particularly necessary in cases where substantial amount of foam was formed at the top. This foam tends to rise with time and erroneous results would be obtained if the measurements were completed over

the short period of time. After the last measurement for a given set of conditions is made the valve below the column is closed, and the main flow of nitrogen is stopped. Then the expanded liquid level starts to drop, and the static height is checked after the foam layer disappears. During this period the flow of nitrogen through two purge lines (~ 30 cm^3 /min per purge line), located 6 cm below and 6 cm above the distributor, is continued. These purge lines are connected to a differential pressure cell (Validyne Model DP 15 TL) to measure pressure drop across the distributor. The static liquid height for most runs was in the range 150-240 cm, but as the holdup increases the liquid has to be drained into the wax storage tank in order to maintain the expanded liquid level within the glass column. In the extreme case of gas holdups around 70% (SMP distributor in the "foamy" regime) the minimum value of static liquid height was 77 cm.

1b. Effect of Temperature

The results of average gas holdup measurements with a 1.85 mm single hole orifice plate distributor are shown in Fig. 2. It is found that two distinct types of operation are possible that will be referred to as a "foamy" regime (open symbols) and a "nonfoamy" regime (solid symbols). In the "foamy" regime a significant amount of foam is present on the top of the liquid layer, and the gas holdup is a function of temperature. The gas holdup in the "foamy" regime tends to increase with temperature, with the exception of data at 250°C and 265°C. On the other hand the values of gas holdup in the "nonfoamy" regime (or the liquid circulation regime according to Maruyama et al., 1981) are essentially independent of temperature in the range 160-280°C.

The past history, i.e. the startup procedure, determines which of the two flow regimes will be attained. The "foamy" regime is attained if one starts experiments at low superficial gas velocities and then increases the gas flow rate. Formation of the foam is related to the existence of small bubbles that are formed at low gas flow rates. As the superficial gas velocity is increased the foam region increases, which gives very high gas holdups.

Procedure for attaining the "nonfoamy" regime contains patentially patentalbe material and is described in the Appendix marked "Not For Publication". The liquid circulation (nonfoamy) regime is characterized by intensive liquid mixing (turbulence), and by presence of relatively large bubbles (1-2 cm in diameter) that are surrounded by small bubbles (about 1 mm or less). In this flow regime the height of foam layer is only about 1-2 cm, and the upper liquid surface tends to oscillate with an amplitude of 5-10 cm. The latter indicates the presence of slug type bubbles. The liquid circulation flow regime is not stable at low values of the superficial gas velocities, i.e. if one decreases the operating velocity beyond a certain critical value transition from the liquid circulation regime to the "foamy" regime takes place. These transitions are shown in Fig.2 with broken lines connecting the points in these two flow regime. It should be noted that these transitions are tentative at the present time ,since no attempt has been made to reduce values of superficial gas velocity in small increments in order to determine the minimum value of u_g, for a given temperature, at which the liquid circulation regime still exists. Nevertheless, the data show a trend that the critical velocity at which the transition takes place decreases with temperature. In

particular, at T= 160°C we have not noticed any significant foam formation over the entire range of superficial gas velocities investigated (i.e. u_g = 1-7 cm/s). This behavior can be, at least partially, explained as the liquid viscosity effect. It is well known that the bubbles are larger as the liquid viscosity increases (i.e. as the temperature decreases), and at low temperatures the number of small bubbles is insufficient to create a stable foam layer in the upper zone of the column.

Visual observations of the flow field in the "foamy" regime made at the distance of about 1.5 m above the distributor may be summarized as follows. At lower superficial gas velocities (less than about 4 cm/s) the liquid is relatively still, and there is a fairly wide distribution of bubble sizes. There is a large number of small bubbles (~ 1 mm in diameter), a small number of relatively large bubbles (1-2 cm), and there are bubbles with diameters between 1 mm and 1 cm. The height of foam layer increases from 4-6 cm at $u_g \approx 2$ cm/s to about 70 cm at $u_g \approx 7$ cm/s. Large bubbles occupying the entire column diameter (slugs) were observed at superficial gas velocities higher than 4 cm/s. The frequency of slugs increases with increasing superficial gas velocity.

All comments about bubble sizes and the flow field characterization should be considered tentative at this point, as they are based on a limited number of observations at one or two locations along the column. More detailed studies on the flow field characterization will be made during the next quarter.

Fig. 3 shows results obtained using a 4 mm single hole orifice plate distributor. The hysterisis type of behavior for the gas holdup as a function of superficial gas velocity is observed as in the case of

experiments with the 1.85 mm orifice plate. In general, with the 4 mm orifice plate the bubbles are somewhat larger than with the 1.85 mm orifice plate, and this helps prevent formation of a stable foam layer and favors existence of the "nonfoamy" regime over the wider range of conditions. For example, in experiments with the 4 mm orifice at 230°C the "nonfoamy" regime could be maintained over the entire range of superficial gas velocities investigated (u_g = 1.3 - 9.3 cm/s), while with the 1.85 mm orifice the transition from the "nonfoamy" to "foamy" regime took place when the superficial gas velocity was decreased from 4.4 cm/s to 2.2 cm/s (Fig. 2). At higher temperatures (265°C and 280°C) the transition from the "nonfoamy" to "foamy " regime takes place as one decreases the superficial gas velocity, which is denoted by broken lines with arrows in Fig. 3.

With the 4 mm orifice plate distributor the transition from the "foamy" to the "nonfoamy" regime was achieved at 230°C and at 265°C. At 230°C the transition occurred when the superficial gas velocity was increased from 3.3 cm/s to 5.3 cm/s, while at 265°C the foam disappeared at about 13 cm/s. From the data shown in Fig. 3 one may expect that the curves corresponding to the "foamy" and "nonfoamy" regimes will merge at a sufficiently high value of u_g, and that only one flow regime will exist beyond this point. This type of behavior (i.e. the closing of hysterisis loop) has been reported in literature for several gas-liquid systems (eg. Maruyama et al., 1981). The change of gas holdup from a higher to a lower value (i.e. the transition from "foamy" to "nonfoamy" regime in our terminology) with the increase of superficial gas velocity was reported by Farley and Ray (1964), who used a paraffin wax as the liquid medium in a 9 1/4 inch reactor. They reported that the increase of superficial gas

velocity from 0.1 ft/s (~ 3 cm/s) to 0.2 ft/s (~ 6 cm/s) has eliminated foaming within one hour after the change was made.

Experimental data obtained with a sintered metal plate (SMP) distributor with the average pore size of approximately 40 μm are shown in Figure 4. Again, the hysterisis type of behavior was observed. The SMP distributor generates much smaller bubbles than the orifice plate distributors, and thus the values of gas holdups are much higher than in the previous two cases, and the range of superficial gas velocities over which the "nonfoamy" regime could be maintained is smaller. The holdup increases with temperature in both regimes. At 230°C, where somewhat larger bubbles are expected (viscosity effect discussed earlier), the "nonfoamy" regime exists for superficial gas velocities between 5-9 cm/s. Also, we were able to break up the foam by increasing the superficial gas velocity from 5 cm/s to 9 cm/s.

1c. Effect of Gas Distributor Designs

The average gas holdups obtained using the three gas distributors are plotted in Figures 5, 6 and 7 for temperatures of 230°C, 265°C and 280°C respectively. The main features that are common for all three temperatures are as follows. The gas holdups with the SMP distributor are significantly higher than the ones obtained with the orifice sintered plates in both "foamy" and "nonfoamy" regimes. The gas holdups at the same operating conditions (i.e. temperature and superficial gas velocity) for the two orifice plate distributors are similar in both regimes, except for T = 280°C and $u_g > 5$ cm/s in the "foamy" regime (Figure 7), where the values of gas holdup with the 4 mm orifice are significantly lower than the ones obtained with the 1.85 mm orifice plate. This might be due to the fact

that the process of foam break up starts earlier as the orifice size increases.

At 230°C (Figure 5) in experiments with the SMP and the 4 mm distributors we were able to achieve the transition from the "foamy" to "nonfoamy" regime by increasing the gas flow rate, but this did not occur in experiments with the 1.85 mm orifice plate. The reasons for this are not completely understood, and further study is required to clarify this behavior.

1d. Comparison of Results with Experimental Data from Literature

Values of gas holdups obtained in our study have been compared with the existing literature data which were obtained under similar conditions, and results are shown in Figure 8 for SMP distributors, and in Figure 9 for orifice plate distributors.

Our data, in the "foamy" regime, with the 40 μm SMP (curve 2 in Fig. 8) are in good agreement with Mobil's data obtained using a 20 μm SMP distributor (curves labeled 3 in Fig. 8). Mobil's data were obtained in a bubble column with 5.1 cm ID (9.1 m tall), using a Fischer-Tropsch derived paraffinic wax designated FT-200 (average molecular weight 600) at 260°C, and are reproduced from Figure 10 in Kuo et al. (1984 a). A discontinuity in the curve 3 is due to the fact that measurements were taken at different static liquid heights (H_s = 483-640 cm for lower values of u_g, and H_s = 305 cm for $u_g > 2$ cm/s). Curves labeled 1(20 μm SMP) and 4 (100 μm SMP) were constructed from experimental data reported by Mobil's workers (Fig. 7 in Kuo et al., 1983). These experiments were conducted in a small hot flow column (5.3 cm ID, 1.9 m tall) at 200°C using FT-200 as the liquid medium. Curve labeled 5 (75 μm SMP) is constructed from experimental data reported

by Deckwer (Fig. 7 in Deckwer et al., 1980). Deckwer et al. data were obtained in two bubble columns having diameters of 4.1 cm and 10 cm, using FT-300 as the liquid medium at temperatures between 250-285°C.

The gas holdups reported by Deckwer et al. (curve 5) are significantly lower than the values obtained in our study (curve 2), or in Mobil's study (curves 1, 3 and 4), even though the operating conditions, the reactor geometry, and the liquid medium were very similar in all cases. Possible reasons for this discrepancy might be: 1. Deckwer et al. used constant temperature anemometry technique to detect the expanded liquid height, which is not sufficiently accurate when a significant amount of foam is present. 2. The system did not reach the steady state, and thus values of expanded liquid height were underestimated (see section 1a).

In Mobil's experiments with the 100 μm SMP distributor at 200°C (curve 4) lower holdups were obtained than in our study (curve 2), which is due to the use of the larger pore size (100 μm vs 40 μm) and the lower operating temperature (200°C vs 265°C).

Mobil's gas holdups obtained with the 60 μm SMP at 200°C (curve 1) are too large, and do not agree neither with our data, nor with their own data obtained with other SMP distributors (curves 3 and 4). These data are expected to lie between the curves labeled 2 (or 3) and 4.

Comparison of gas holdups obtained with single hole orifice plate distributors is shown in Fig. 9. Experimental points obtained in our laboratory are marked with open symbols for the data in the "foamy" regime, and the data from the "nonfoamy" regime are marked with solid symbols. The results from Mobil's study are represented by continuous solid curves that were constructed from their experimental data. Mobil's data were obtained

in the 5.1 cm ID, 9.1 m tall bubble column with FT-200 wax as the liquid medium at 260°C. In the "foamy" regime the gas holdups obtained with the 1 mm orifice plate, (curve 1 is constructed from Fig. 11 in Kuo et al. 1984a), are generally higher than the corresponding values obtained in our study with the 1.85 and 4 mm orifice plate distributors, which is to be expected. In the "nonfoamy" regime there is no effect of the orifice size in the range 1.85-4 mm. Our data with the 1.85 mm and 4 mm orifices almost coincide with Mobil's data obtained in their experiments with the 2 mm orifice. The solid curve labeled 2 representing Mobil's data was constructed from data reported in Fig. 3 of Kuo et al. (1984 b). It is interesting to note that Mobil workers were able to maintain the "nonfoamy" regime over the wide range of velocities (u_g = 1-12 cm/s). They have not reported any data in the "foamy" regime with the 2 mm orifice, nor data in the "nonfoamy" regime with the 1 mm orifice plate distributor. However, their experiments were not designed with the objective to obtain data in both of these two regimes. Specifically designed experiments would be required to test possibility of existence of hysterisis behavior in a very tall column used in Mobil's study.

1e. Analysis of Data Using Empirical Correlations from Literature

Deckwer et al. (1980) found that experimental gas holdup values obtained in their study are much higher than predicted by any of existing empirical correlations. The gas holdups obtained in our study in the "foamy" regime are even higher than the ones in Deckwer et al. study, as discussed in the previous section, and thus it is clear that the existing correlations can not be used to describe our data in this regime. Deckwer et al. proposed an empirical correlation of the form

$$\epsilon_g = 0.053\ u_g^{1.1} \qquad (2)$$

on the basis of their experimental data, but this correlation predicts lower holdups than found in our study, even with the 1.85 mm orifice plate distributors, as shown in Fig. 10. The average values of observed gas holdups are marked by squares in Fig. 10, and the range of values observed in different runs is indicated by vertical bars.

Several empirical correlations were used to analyze the data in the "nonfoamy" regime, and it was found that the Bach and Pilhofer (1978) correlation agrees very well with the data obtained using the orifice plate distributors (1.85 mm and 4 mm) over the range of temperatures that was investigated (230 - 280°C). Akita and Yoshida's (1973) correlation underpredicts the observed gas holdup values. This is shown in Fig. 10, where the experimental data obtained with the 1.85 mm orifice plate distributor at 265°C are shown together with predictions obtained from the correlations. Similar type of behavior was found at other temperatures (230 and 280°C), and with the 4 mm orifice plate distributor. The gas holdups values with the 40 μm SMP in the "nonfoamy" regime are significantly higher than predicted by Bach and Pilhofer's correlation.

2. Axial Gas Holdup Measurements

Preliminary work was conducted with the 2" ID stainless steel column for the purpose of determining axial gas hold-up. The Validyne pressure transducer was used to measure local pressure drops. Pressure taps are located about the distributor as well as heights of approximately 1.5, 4.5, 7 and 10 ft above the distributor.

Several unsuccessful attempts were made to obtain data for axial gas hold up. The major problem that occurred during these attempts was the plugging of the pressure tap lines with wax. Initially, only nitrogen purge flow (40-50 cm^3/min) was used to try to prevent the wax from entering the pressure tap lines. Later, these lines were heated in order to prevent the wax that might seep through from solidifying. Also, the system was modified so that the pressure tap lines could be cleared by forcing nitrogen through them before each reading.

Figure 11 shows a schematic of the pressure tap system used to collect some initial data. The data were collected for the 40 μm SMP, the 1.85 mm and 4 mm orifice plate distributors, but have not been analyzed yet.

3. Photographic Techniques for Determining Bubble Size Distribution and Mapping of Flow Regimes

During this reporting period work on development of photographic techniques for determination of bubble size distribution and mapping of flow regimes was initiated.

Canon AE1/P 35 mm SLR camera was used for taking photographs for determination of the bubble size distribution in the small glass bubble column. The effect of type of light source, and its location relative to the column on the quality of photographs was studied using 50 mm and 135 mm lenses. The best quality photographs were obtained with a continuous reflective light source located about 2 ft. directly behind the column. In order to monitor the flow regimes and collect data necessary for mapping of various flow regimes a Hitachi (Model GP-5AU) video camera was used. Different types of lighting (continuous non-reflective, continuous reflective, and strobe light), as well as lenses (101.2 mm and zoom) were

attempted in order to find the best possible combination. The use of a continuous reflective light source located behind the column was found to be the most satisfactory.

4. Future Work

- Continue work on hydrodynamic studies in the small glass bubble column in the following areas:
  (a) Complete studies on the observed hysterisis behavior of the average gas holdup.
  (b) Study the foam break up at higher superficial gas velocities, and the long term stability of the "nonfoamy regime.
  (c) Determine the map of flow regimes using the video camera.
  (d) Analyze photographs of the flow field to determine the bubble size distribution using an image analyzer.
- Modify the system for axial gas holdup measurements in the small stainless column by differential pressure method, and obtain experimental data for selected operating conditions.
- Study the effect of operating conditions on the average gas holdup in the large glass column with a multiple hole orifice plate distributor.

D. Task 4 - Correlation Development and Data Reduction

No work on this Task has been planned for this quarter.

V. Nomenclature

H expanded liquid height, (cm)

H_s static liquid height, (cm)

T column temperature, (°C)

u_g superficial gas velocity at operating conditions, (cm/s)

Greek Letters

ϵ_g average gas holdup, (-)

Acronyms

BC bubble column

DOE Department of Energy

FT Fischer-Tropsch

ID inside diameter

SMP sintered metal plate

TAMU Texas A&M University

VI. Literature References

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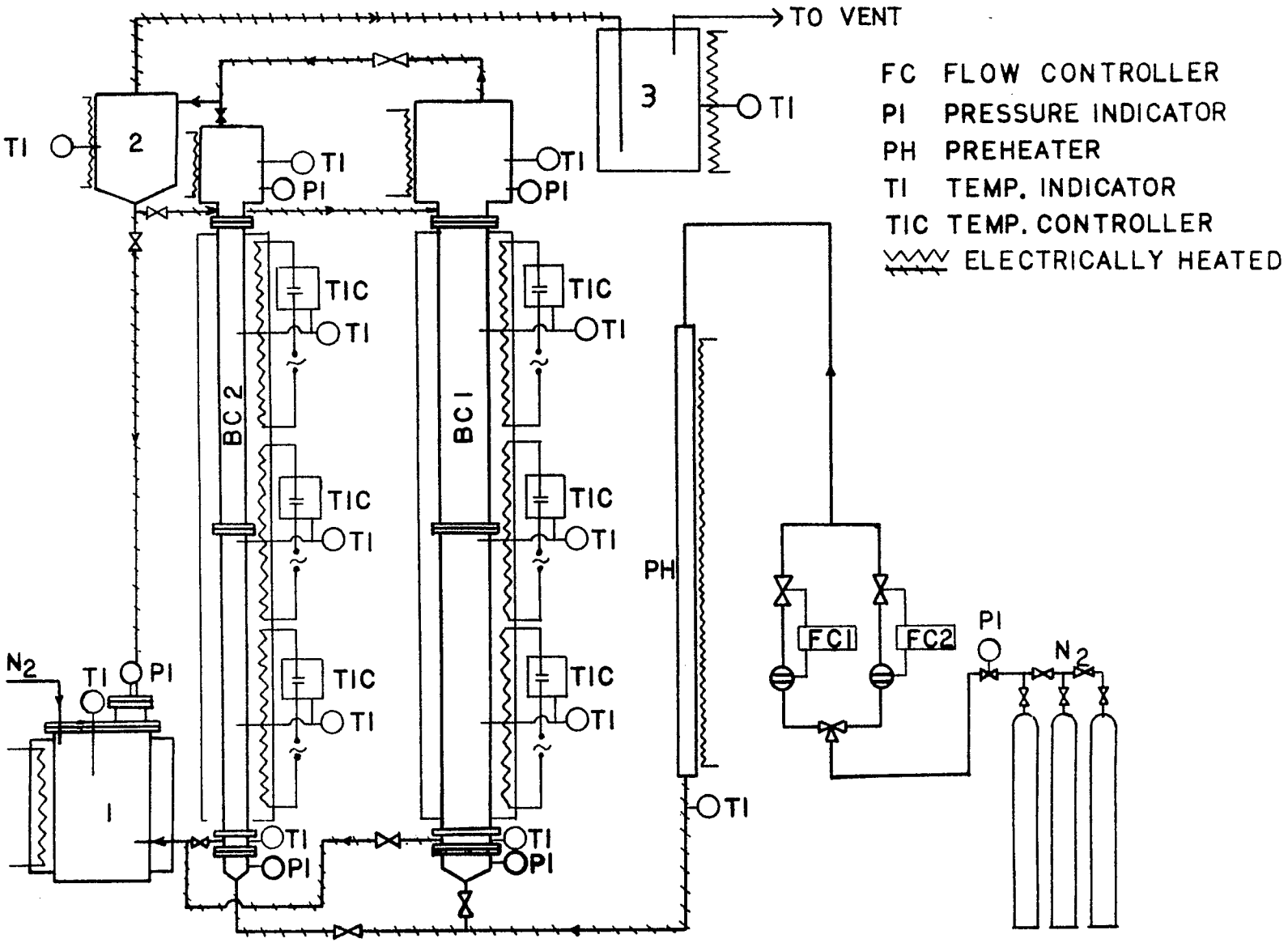

Figure 1. Flow diagram of experimental apparatus

Legend: 1- Storage tank; 2- Gas-liquid separator; 3- scrubber;
BC1- Bubble column (I.D.=22.9 cm); BC2- Bubble column (I.D.=5.1 cm)

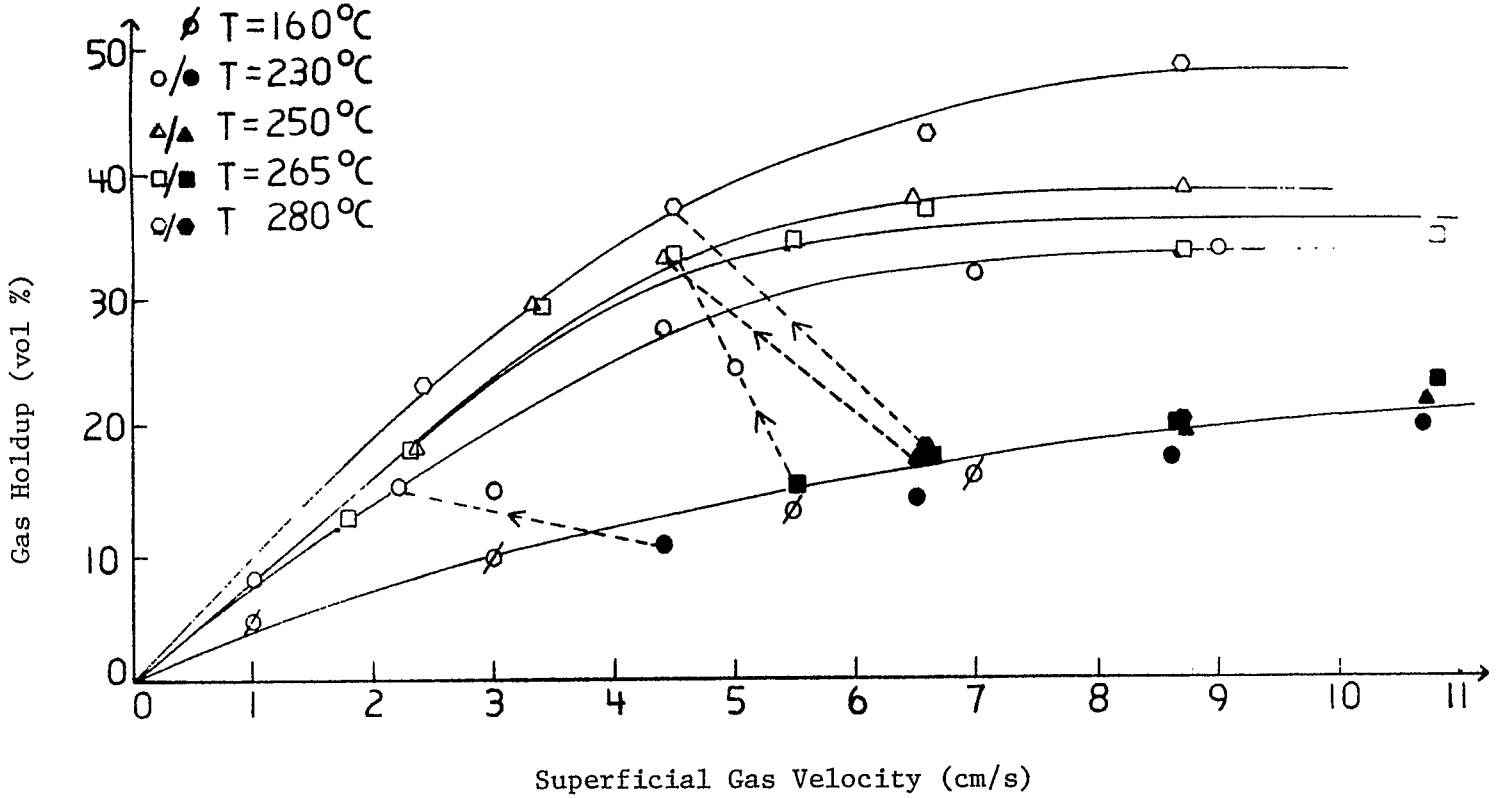

Figure 2. Effect of temperature on gas holdup
Distributor: 1.85 mm single orifice plate
Solid symbols - non-foamy regime
Open symbols - foamy regime

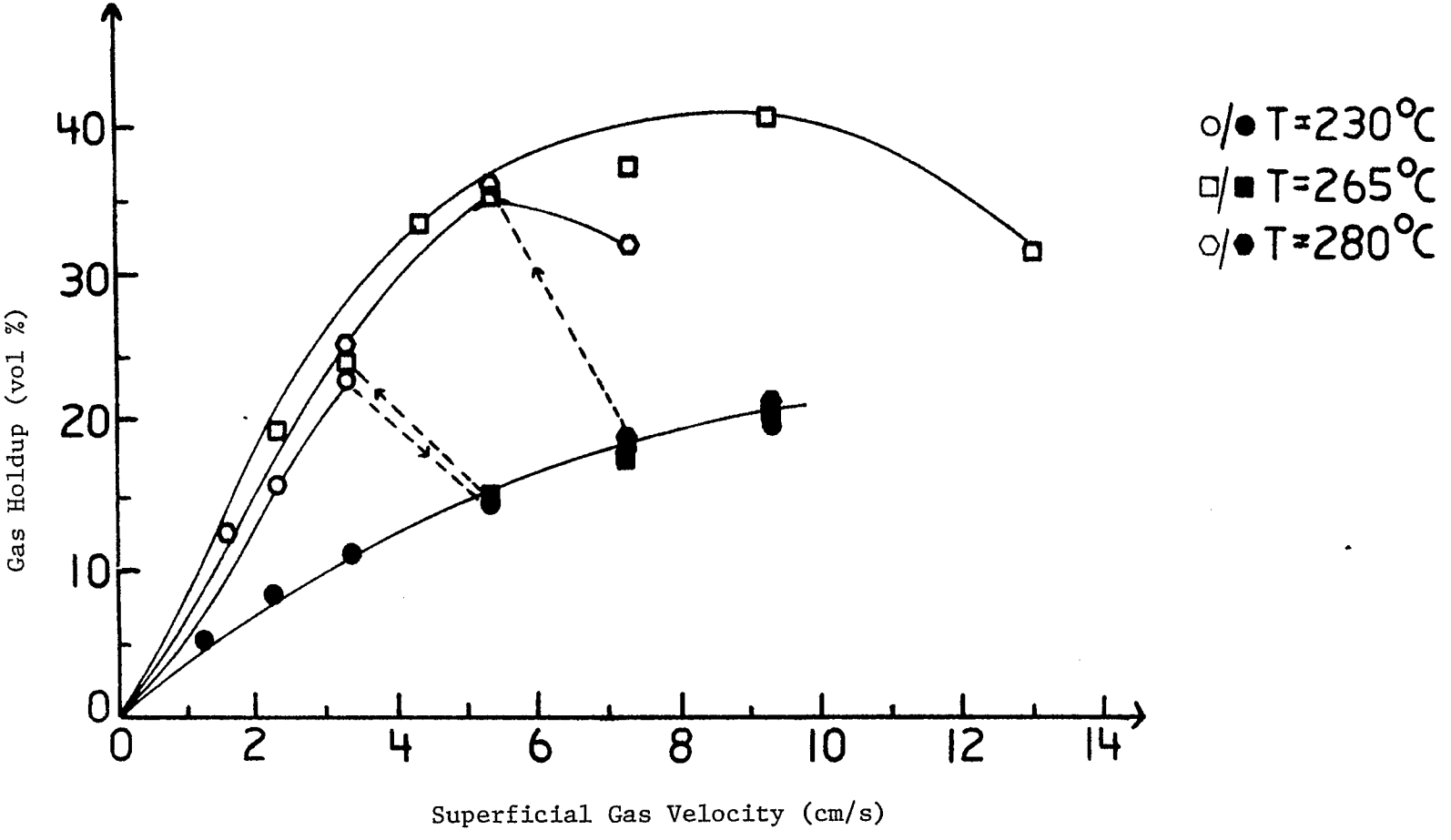

Figure 3. Effect of temperature on gas holdup
Distributor: 4 mm single orifice plate
Solid symbols - non-foamy regime
Open symbols - foamy regime

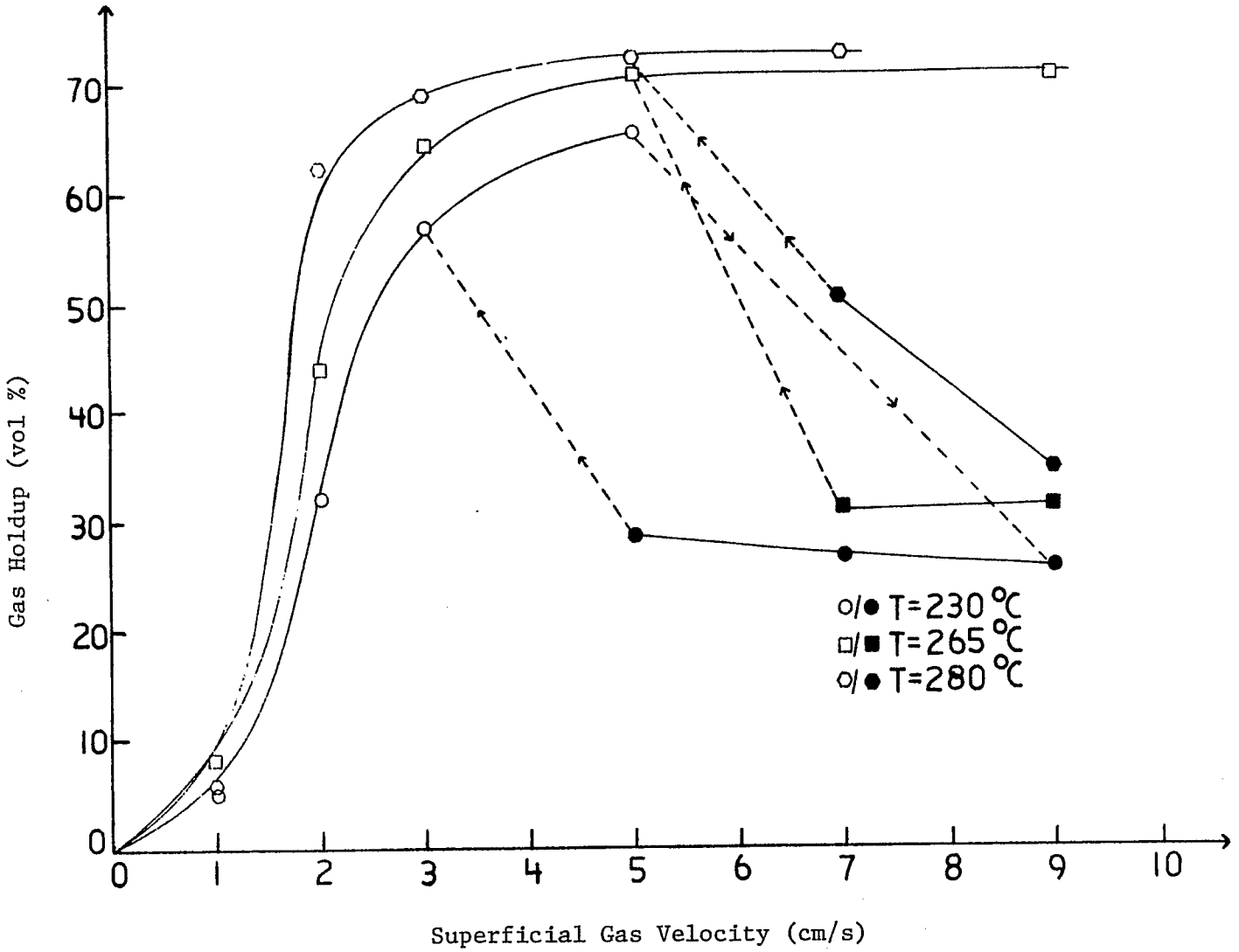

Figure 4. Effect of temperature on gas holdup (40 μm SMP; solid symbols - non-foamy regime; open symbols - foamy regime)

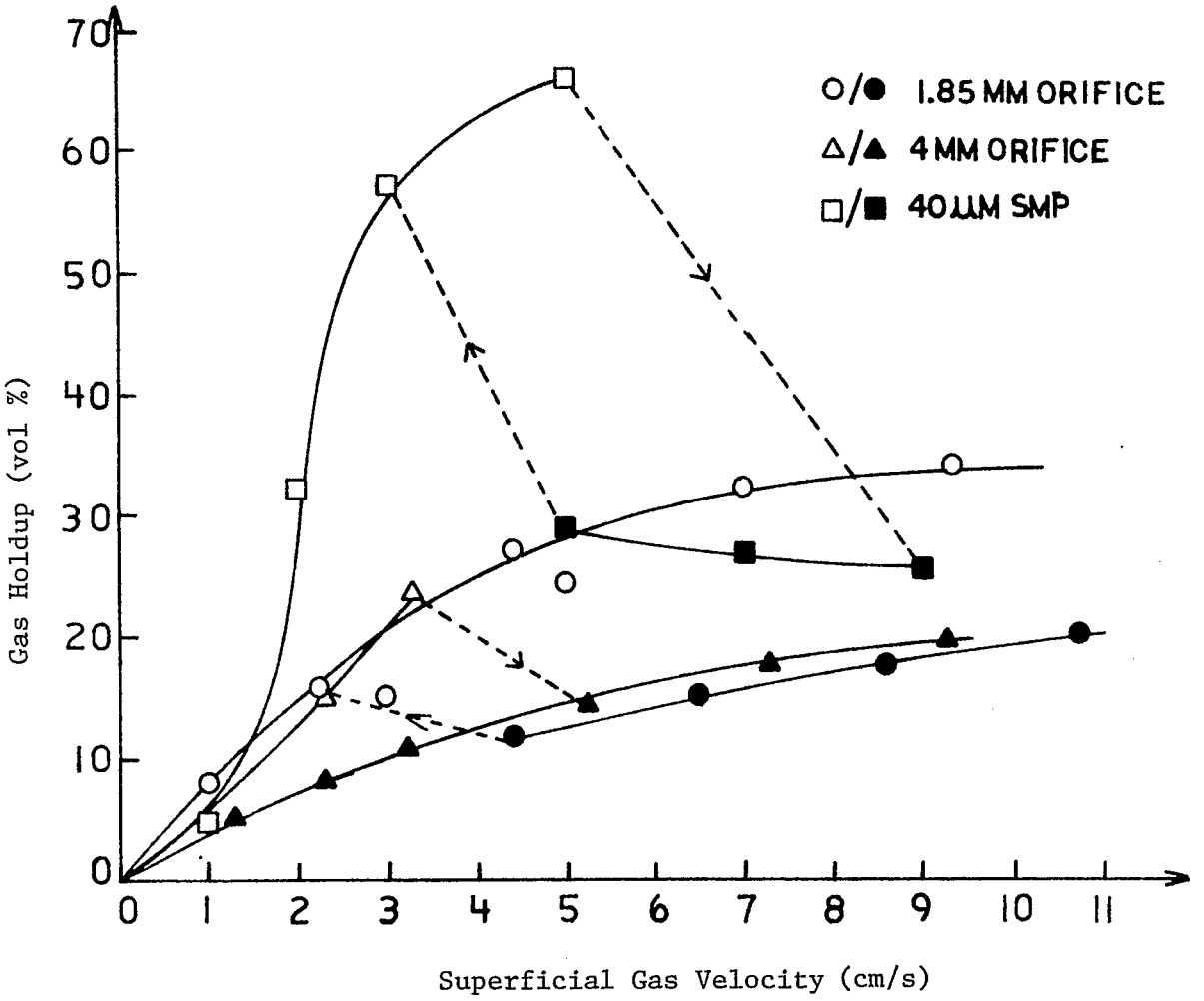

Figure 5. Effect of distributor type on gas holdup (T-230°C; FT-300; solid symbols - non-foamy regime; open symbols - foamy regime)

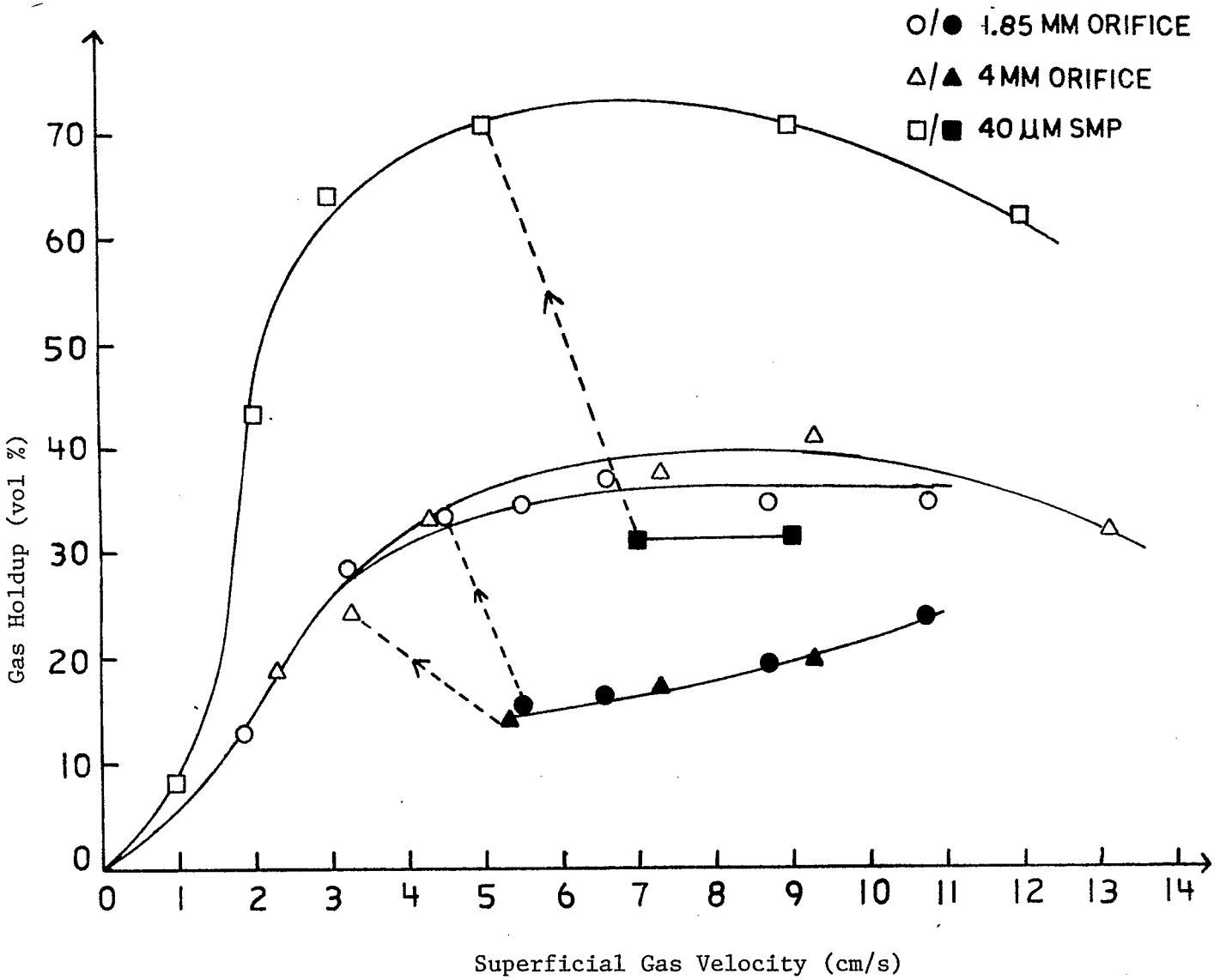

Figure 6. Effect of distributor type on gas holdup (T=265°C; FT-300; solid symbols - non-foamy regime; open symbols - foamy regime)

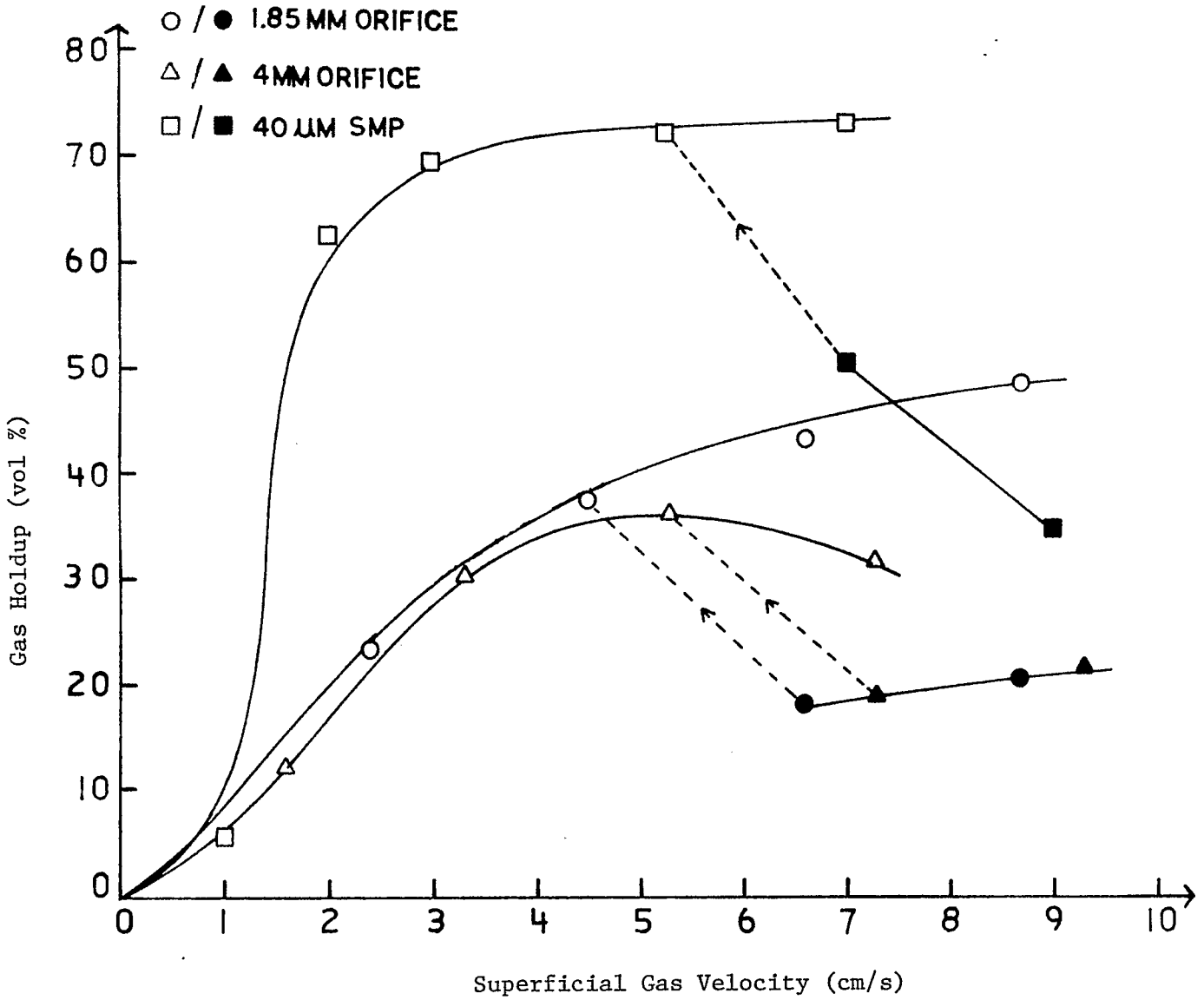

Figure 7. Effect of distributor type on gas holdup (T=280°C; FT-300; solid symbols - non-foamy regime; open symbols - foamy regime)

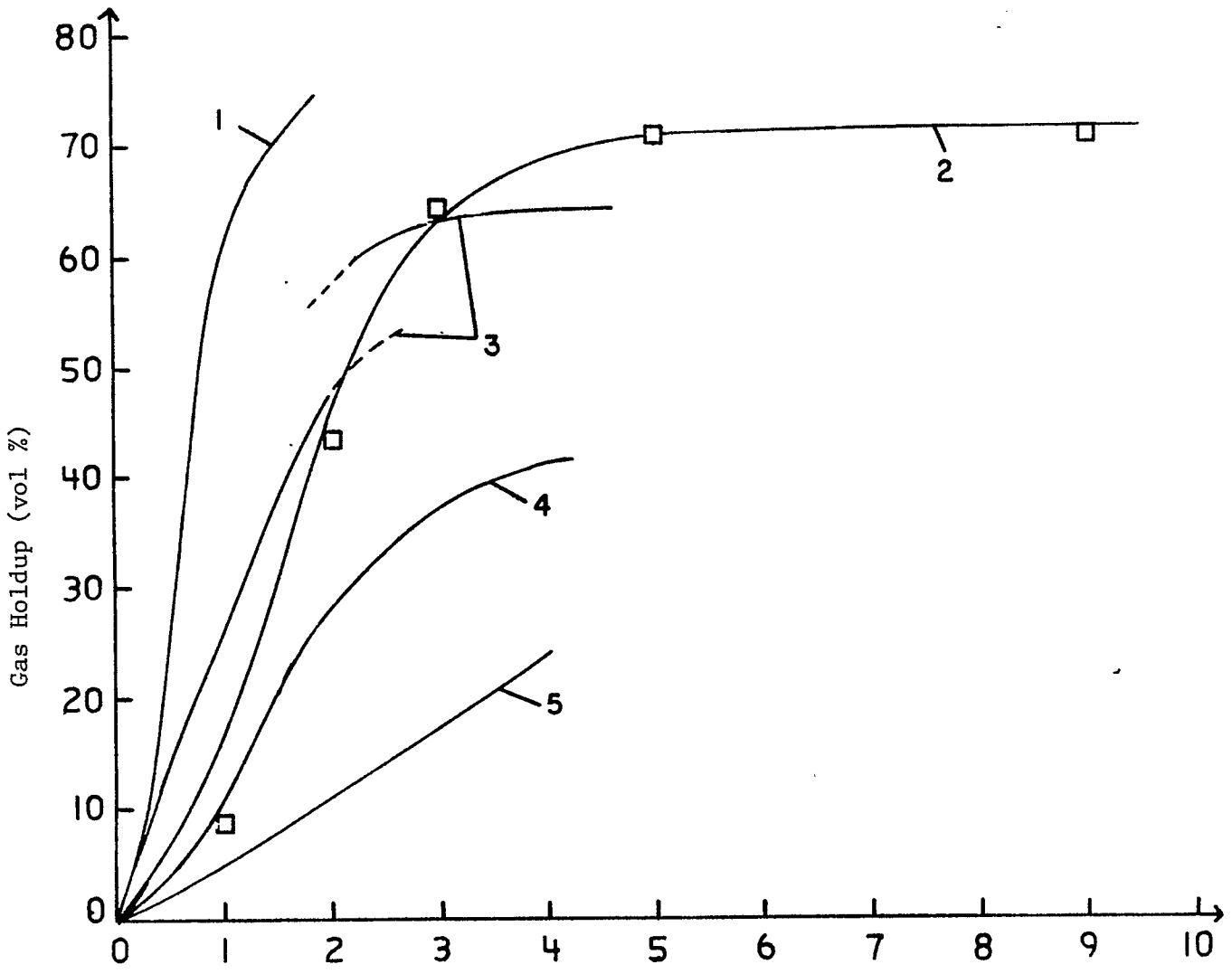

Figure 8. Comparison of sintered plate distributors
(1,4:Mobil; T=200°C; FT-200; 1-20 μm SMP; 4-100 μm SMP;
2:TAMU; T=265°C; FT-300; 40 μm SMP; 3:Mobil; T=260°C;
FT-200; 60 μm SMP; 5:Deckwer; T>250°C; FT-300; 75 μm SMP)

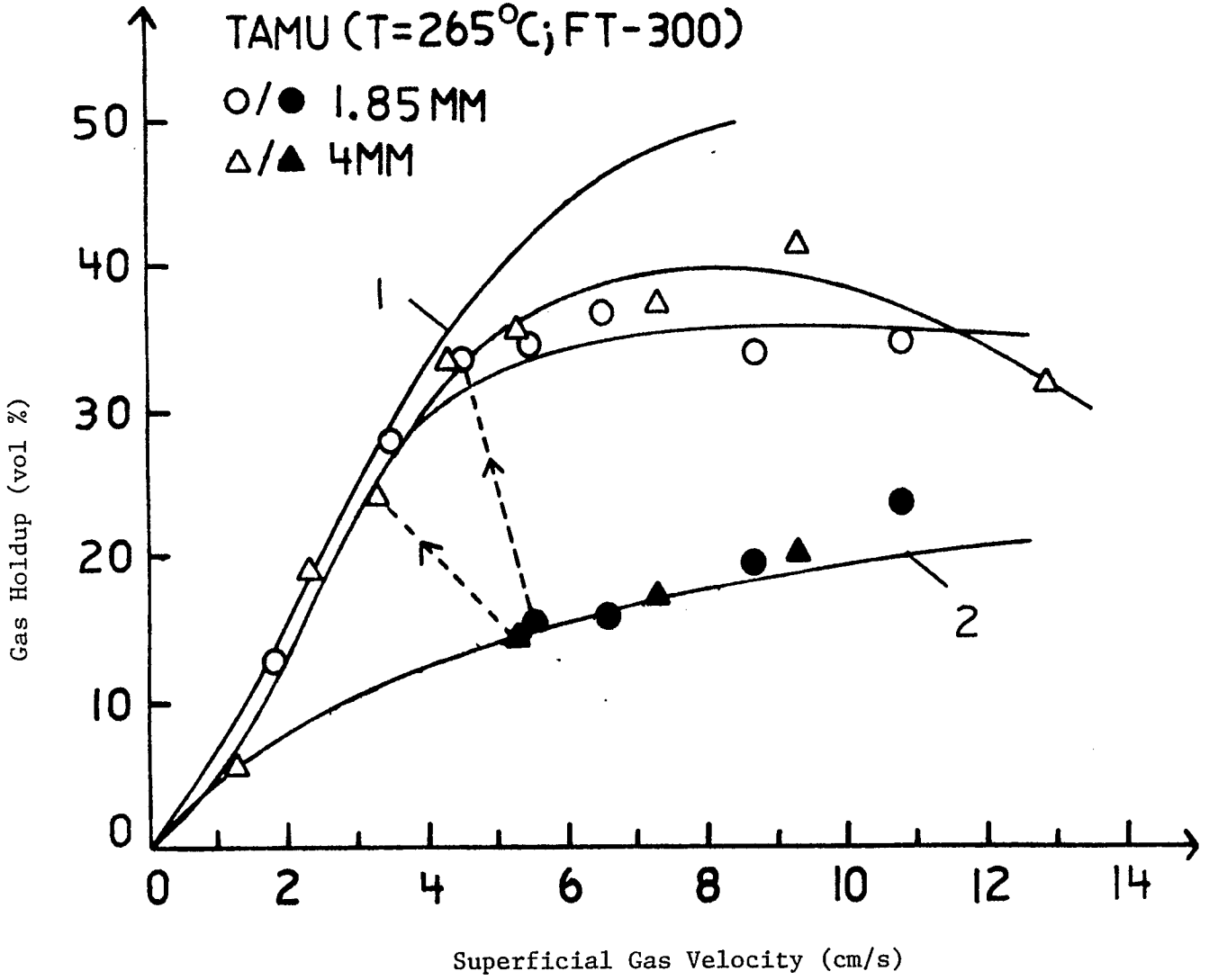

Figure 9. Comparison of single orifice plate distributors.
(1,2-Mobil; T=260°C; FT-200; 1-1 mm orifice; 2-2 mm orifice;
TAMU; solid symbols - non-foamy regime; open symbols - foamy regime)

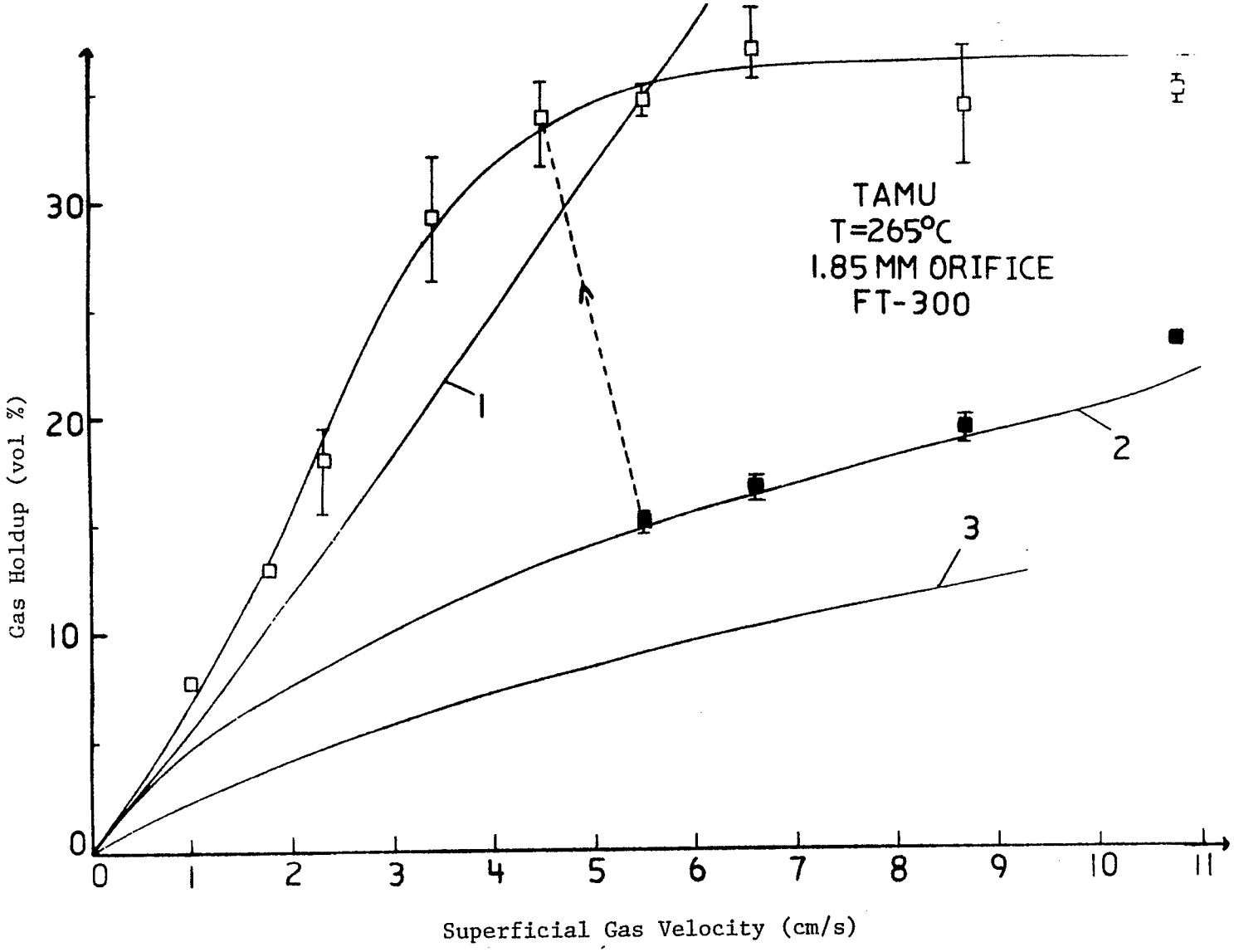

Figure 10. Comparison of gas holdup with various correlations.

(Legend: ☐ foamy regime; ■ non-foamy regime; 1- Deckwer, et al.; 2- Bach and Pilhofer; 3- Akita and Yoshida)

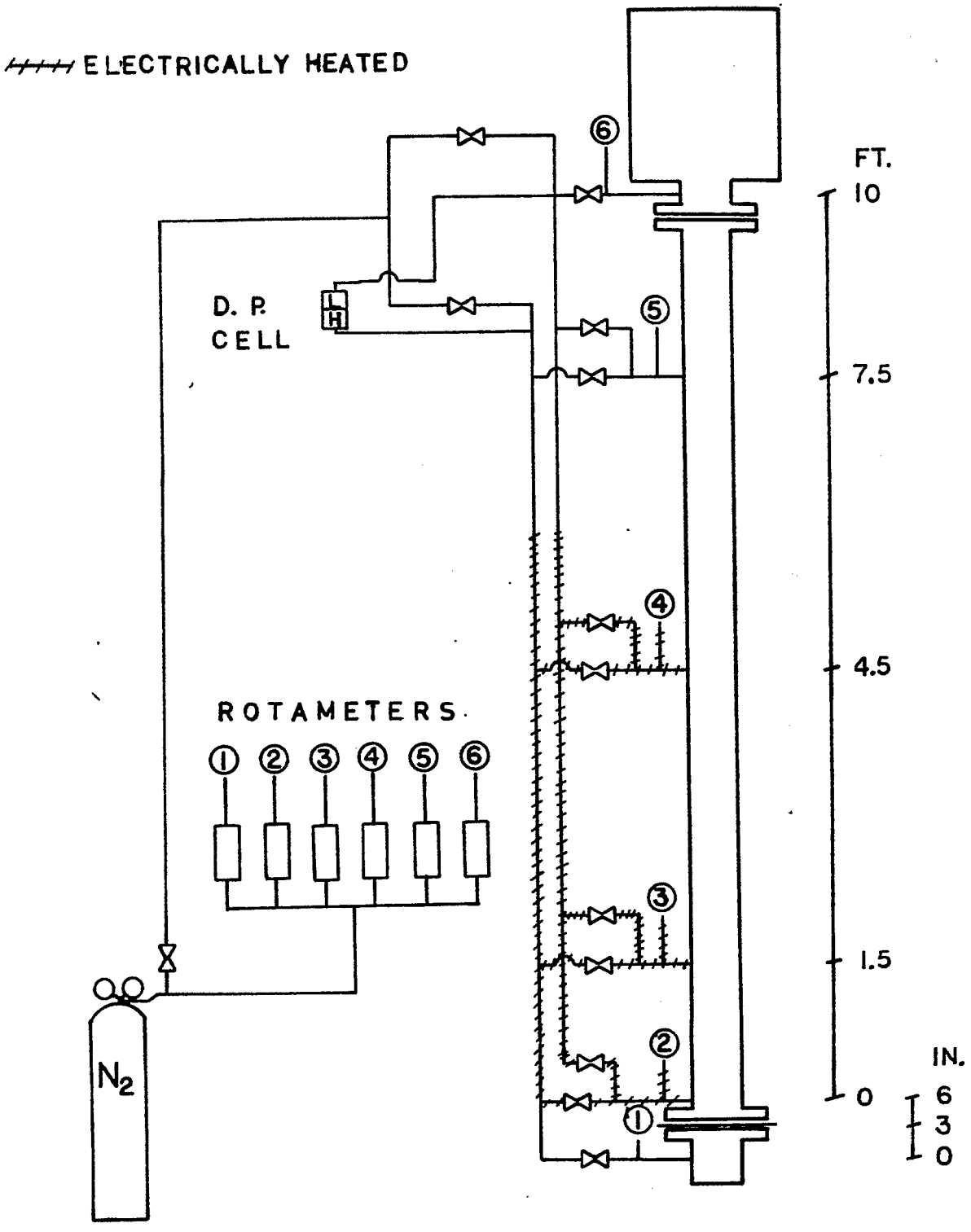

Figure 11. Pressure tap system for axial gas holdup measurement by differential pressure method

APPENDIX

Description of start-up procedure required to attain the "nonfoamy" regime.

NOT FOR PUBLICATION

(Contains potentially patentable material)

Appendix. Description of start-up procedure required to attain the "nonfoamy" regime.

In order to operate the column in the virtual absence of foam the following procedure was employed with all three distributors (i.e. 40 μm SMP, 1.85 mm and 4mm orifice plate).

The nitrogen flow corresponding to the superficial gas velocity of about 9 cm/s is maintained during the transport of wax from the storage tank to the column, and during the entire heating period of the column. Once a desired operating temperature is reached (230-280°C), the measurements are taken as described in the Section 1a. of the report. The system is in the "nonfoamy" regime, and experiments are usually continued by decreasing the superficial gas velocity in order to determine an approximate range of superficial gas velocities over which this flow regime can be maintained.

We found that it is essential to maintain relatively high nitrogen flow rate (u_g = 7-9 cm/s) while the column is being heated in order to attain the "nonfoamy" regime. If the small gas flow rate is used during the heating period, and the switch to high superficial gas velocity is made when the desired operating temperature is reached, a large amount of foam will be formed (i.e. the "foamy" regime will be attained).

The reason for this behavior is that during the heating period the flow rate is small, and a large number of small bubbles is present in the liquid. These small bubbles do not coalesce, and they accumulate in the upper zone of the column forming a stable foam layer. The sudden increase of the gas flow rate at the end of the heating period can not break this foam (at least not over relatively short periods of time, 1-2 hrs., that

were investigated in our study), and the "nonfoamy" regime can not be attained. On the other hand, if one starts with the high flow rate from the very beginning, the bubbles in the liquid are larger and coalesce faster so that the foam does not form on the top of liquid. The intensive turbulence and mixing patterns, also may promote coalescence of very small bubbles, and thus prevent the formation of foam.

At the present time we have not had opportunity to review all relevant papers on hysterisis behavior observed in other gas-liquid systems, and to see whether other authors have described procedures to attain the low branch on the ε_g vs. u_g diagram (i.e. the "nonfoamy" regime). The literature review will show whether our procedure is a novel one or not, i.e. whether there is a merit to patent it.