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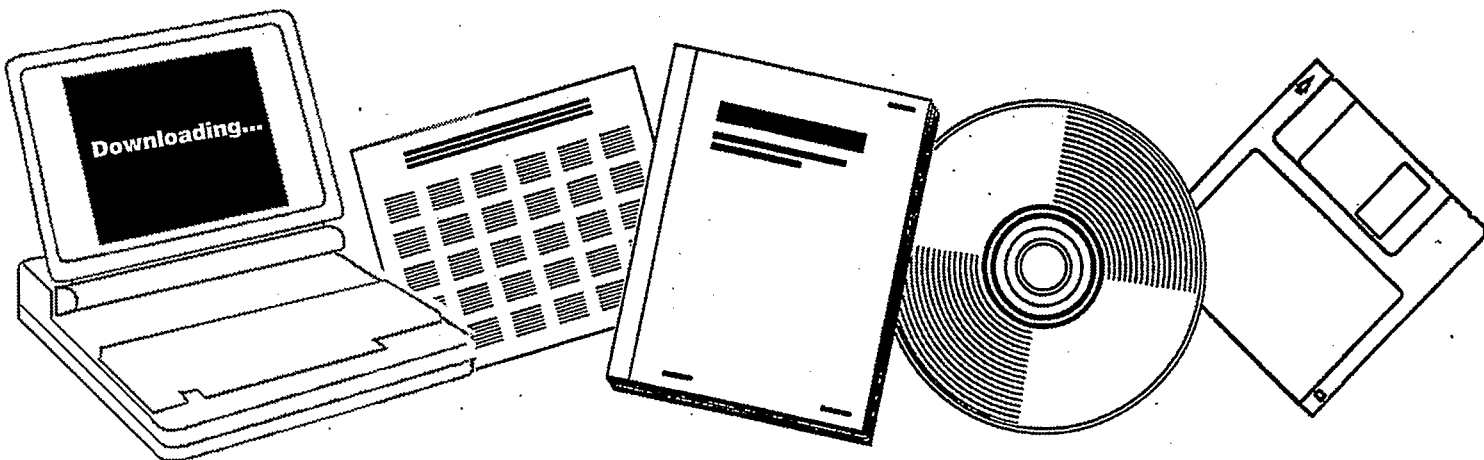
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**TWO-STAGE PROCESS FOR CONVERSION OF  
SYNTHESIS GAS TO HIGH QUALITY  
TRANSPORTATION FUELS. QUARTERLY REPORT, 8  
JUNE-30 SEPTEMBER 1983**

**MOBIL RESEARCH AND DEVELOPMENT CORP.  
PAULSBORO, NJ**

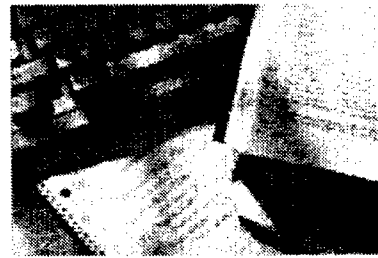
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TWO-STAGE PROCESS FOR CONVERSION OF SYNTHESIS GAS  
TO HIGH QUALITY TRANSPORTATION FUELS

QUARTERLY REPORT FOR THE PERIOD  
8 JUNE - 30 SEPTEMBER, 1983

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## I. Abstract

The design of two large hot-flow models, to be used for slurry bubble-column hydrodynamic studies, is described. Scoping experiments were performed in smaller models to study the effects of feed-gas distributor type, column diameter, and liquid medium on gas holdup and bubble sizes. In addition, a literature review of bubble-column hydrodynamics is presented.

Modifications to improve the operation and flexibility of the existing two-stage pilot plant have been designed and construction initiated. Also, a sample of reactor-wax was fractionated under vacuum in a laboratory still.

## II. Objective and Scope of Work

The general objective of this work is to develop a slurry Fischer-Tropsch/ZSM-5 process for converting low H<sub>2</sub>/CO ratio synthesis gas, of the type produced in a coal gasification system, into maximum yield of transportation fuels. To accomplish this objective, the following tasks will be undertaken.

### Task 1 - Process Studies in Two-Stage Bench-Scale Unit

Operation of the bench-scale unit will be directed toward production of hydrocarbons containing less than 8 wt% of methane plus ethane with high throughput, high conversion, and good catalyst stability. Together with Task 2, high quality liquid fuels, particularly the distillate, will be maximized. At least two tests shall be conducted using at least two different catalysts. One of these catalysts may be provided by DOE's alternate catalyst development projects.

### Task 2 - Scoping Studies of Fischer-Tropsch Reactor-Wax Upgrading

The methods for upgrading the reactor-wax which is withdrawn from the slurry Fischer-Tropsch reactor will be evaluated. These methods should include conventional refinery processes, such as Fluidized Catalytic Cracking, Hydrocracking, Catalytic Selective Cracking, Thermal Cracking, and Hydrodewaxing. Proprietary mathematical models and open literature information will be used to the extent possible for these process evaluations.

Means for separating the reactor-wax from the catalyst fines, if such a separation is needed prior to reactor-wax upgrading, shall be investigated.

### Task 3 - Product Evaluation

The quality of the hydrocarbon liquid products from the two-stage unit and the reactor-wax upgrading processes shall be evaluated. Gasoline octane and distillate cetane quality, as well as pour points should also be determined.

#### Task 4 - Slurry Fischer-Tropsch Reactor Hydrodynamic Studies

The effect of different feed-gas distributor designs on the slurry Fischer-Tropsch reactor performance will be investigated. Tests will be conducted in the BSU slurry reactor, or other bubble-column reactors, to provide guidance for subsequent runs in Task 1 as well as for design and operation of the non-reacting models. For hydrodynamic studies, the design, construction, and operation of hot, non-reacting bubble-column models will be required.

#### Task 5 - Development of Conceptual Process Schemes

A conceptual process scheme to maximize gasoline and distillate yield using a combined system of slurry Fischer-Tropsch/ZSM-5 reactor plus reactor-wax upgrading will be developed. Scoping costs of the plant will be estimated.

### III. Summary of Progress to Date

The two-stage bench-scale unit is currently undergoing modifications. Major items are improvement of the on-line reactor-wax separation hardware, improvement of the DP-cell setup, and installation of hardware for steam co-feeding to the Fischer-Tropsch reactor. The new DP-cell design, which eliminates nitrogen purging, was successfully tested in a small bubble-column reactor.

The design basis of two large hot-flow bubble-columns (5.1 and 10.2 cm ID x 9.1 m height) has been established. The columns, which are to be used for hydrodynamic studies, are each divided into three vertical sections, the middle one being metal and the other two glass. Provisions are made for slurry sampling and probe insertion.

A literature review of bubble-column hydrodynamics was done, with the major conclusions being:

- The data with F-T derived medium in large-size reactor at high gas velocity are essentially nonexistent.
- Gas holdup in Fischer-Tropsch (F-T) derived waxes is significantly larger and bubble sizes significantly smaller than with other liquid media of similar physical properties.
- The type of gas distributor can affect gas holdup and bubble size in small diameter columns operating in the homogeneous bubbly-flow regime.
- Column diameter in the range 4.1 to 10 cm does not affect hydrodynamics in short columns.
- Increased static liquid height reduces the gas holdup.
- Pressure does not affect gas holdup. This was verified by studies in the small bubble-column reactor using the new DP-cell setup.

In addition, hydrodynamic studies were performed in two small hot-flow bubble-columns. Using F-T derived waxes, it was observed that:

- Sintered-metal plate distributors produce high holdups (including foam) and small bubbles, but increasing the average pore size of the plate lessens the effect.



- Single orifice distributors produce non-uniform bubble sizes and generally lower holdups than sintered-metal plate distributors.
- Hexadecane at room temperature, which has properties similar to F-T derived wax at higher temperature, produces much lower holdups and larger bubble sizes.
- Column diameter, in the range 3.2 to 5.3 cm, appears to have some effect on hydrodynamics. The extent of the effect varies with the distributor.

Lastly, vacuum fractionation of a reactor-wax sample from Run CT-256-4 was carried out.

#### IV. Detailed Description of Technical Progress

##### A. Task 1 - Process Studies in Two-Stage Bench-Scale Unit

##### 1. Bench-Scale Unit Modifications - Status

Design work on the modification of the two-stage bench-scale pilot plant is being initiated. The major items are:

- Improved on-line reactor-wax separation hardware
- Improved DP-cell set-up for bubble-column hydrodynamic measurements
- Hardware for steam co-feeding to the Fischer-Tropsch Reactor

The improved on-line reactor-wax separation hardware will include a larger two-liter settling pot instead of the existing one-liter pot to increase the rate of reactor-wax separation. Other details are confidential and will be described in the confidential part of subsequent reports.

The new DP-cell set-up will consist of purgeless DP-cells, with the high pressure side being in contact with the reactor slurry. This will eliminate the plugging which was previously experienced in the purged DP-legs, and also the nitrogen dilution effect.

Steam co-feeding will allow testing of the in-situ water-gas shift concept. In-situ water-gas shift is essential as an inexpensive means for changing a less desirable low  $H_2/CO$  ratio (0.4-0.5) synthesis gas from some advanced gasifiers to a desirable  $H_2/CO$  ratio of 0.6-0.7.

These and a series of other smaller modifications will be described in detail when the final decisions are made.

##### 2. A Study in Support of Process Studies in the Bench-Scale Unit

Before modification of the DP-cell set-up in the two-stage BSU, the concept was successfully demonstrated in Unit CT-225, a small (2.7 cm ID x 1.9 m height) bubble-column reactor. As shown in Figure 1, a DP-cell was mounted at the base of the reactor, with the high-pressure side connected to the existing slurry drain line. The low pressure side was connected to the

product gas line leaving the cold condenser. This prevented buildup of liquid in the DP-line which can cause an erroneous reading. The DP-cell was provided with a nitrogen purge line on the high-pressure side to allow back-flushing of the slurry to the reactor. The entire DP-cell was heated to prevent solidification of reactor-wax inside the chambers.

To test the new arrangement, a fixed amount of 15 wt % solids slurry was loaded into the reactor. The bubble-column was then operated under nitrogen flow at pressures ranging from 0.1 to 1.48 MPa and superficial gas velocities of 0 to 3.3 cm/s. No apparent detrimental effects have been observed on the DP-cell during the test period, which has lasted nearly a month. In fact, it was shown that using the new DP arrangement in the proper manner provided insights into the gas holdup in the column (see Section IV.C.2.3).

The DP-cell was also operated without slurry in the DP-chamber. This was accomplished by back-flushing with nitrogen several times. This configuration provided the most accurate readings by eliminating the leg of slurry leading to the DP-cell. Previously, the height of that leg had to be added to the DP-readings to account for the full slurry height in the reactor. Allowing the slurry to reside in the DP-cell would also permit settling of the trapped catalyst, possibly causing plugging of the lines.

### 3. Future Work

Work in the next Quarter will include:

- Design and modification of the two-stage BSU.
- Scoping evaluation of a F-T catalyst for low methane + ethane mode operation using a small bubble-column reactor.

### B. Task 2 - Scoping Studies of Fischer-Tropsch Reactor-wax Upgrading

#### 1. Vacuum Fractionation of Reactor-wax

A scoping vacuum fractionation of a reactor-wax was conducted. A 20.65 kg sample of reactor-wax from Run CT-256-4. was fractionated using a laboratory vacuum distillation apparatus. The reactor-wax contained about 0.1-0.18 wt % of catalyst fines. The vacuum fractionation was prematurely terminated when a small amount of wax was found on the condenser. This happened when the cooling water temperature in the condenser

was raised to the maximum (66°C). Also, a gradual vacuum loss (pressure increased from 0.5 to 1.2 mm Hg) was experienced. This vacuum loss might indicate some cracking of the reactor-wax during fractionation.

The distillation cut temperature at the termination of fractionation was 461°C. The different fractions collected during the distillation were:

Fraction -----	Wt % (No Loss Basis) -----
216°C <sup>-</sup>	0.02
216-343°C	1.13
343-399°C	2.53
399-454°C	6.95
454-461°C	6.31
461°C <sup>+</sup> (Bottoms)	83.06
	100.00

The total recovery of the material after fractionation was 98.9 wt %. The overhead fractions were very clear and did not contain any catalyst fines. The bottom fraction (461°C<sup>+</sup>) was analyzed for solid content and was found to contain about 0.11-0.16 wt % catalyst. A small amount of catalyst may have been left behind in the still. Overall, both the reactor-wax and the catalyst balances were good.

The laboratory-distillation apparatus, which is widely used in the vacuum fractionation of conventional petroleum wax, could not simulate the commercial vacuum fractionation of the reactor-wax, since a 538°C (1,000°F) cut point can generally be achieved in commercial vacuum fractionation towers. The limitation of the condenser temperature (maximum of 66°C) can be eliminated only by expensive modification of the distillation apparatus.

Another way to estimate the fractions of vacuum separation is to use the carbon-number distribution of the reactor-wax. The 538°C cut point is roughly equivalent to a paraffin of forty-two carbon-number. Based on this equivalence, one can expect that about 93 wt % of the reactor-wax sample shall become an overhead fraction in a commercial vacuum fractionation tower.

## 2. Future Work

Work in the next Quarter will include:

- Preparation of solid-free reactor-wax stocks using a high gradient magnetic separator.
- Scoping evaluation of upgrading reactor-wax by conventional thermal cracking.
- Scoping evaluation of upgrading reactor-wax by conventional fluid catalytical cracking.

C. Task 4 - Slurry Fischer-Tropsch Bubble-Column Hydrodynamic Studies

1. Design and Construction of Two Tall, Hot-Flow Non-Reacting Columns

1.1 Simplified Flow Diagram and Design Basis

A simplified flow diagram of the two hot-flow non-reacting columns is given in Figure 2. The design basis given in Table 1 lists the column dimensions and the design ranges of the major variables of these columns.

Both the 5.1 and 10.2 cm ID columns have identical features. Hence, only one column is depicted in Figure 2. Also, both columns cannot be operated in parallel, since several hardware features are shared by both columns, namely the preheater, all temperature controllers, and the pressure controller.

As shown in Figure 2, in-house N<sub>2</sub> or any other gas, such as He or CO<sub>2</sub> supplied by a bank of cylinders, is metered by a mass-flow meter-controller. The preheated gas enters the column below the feed-gas distributor plate. The distributor plate is mounted between two flanges.

Each column is divided into three sections connected by flange joints. The section between 610 and 914 cm levels is a glass section, permitting visual observation of expanded liquid level and flow patterns. Another glass section is provided between 0 and 152 cm above the distributor to observe the flow patterns developing at the distributor. The third section between 152 and 610 cm levels is made of steel and is heated by two-zone wrapped electric heaters.

Three methods of heating the glass sections were evaluated. The important aspects considered were uniform heating and provisions for visual observation of the liquid level. Based on heat radiation calculations, an arrangement of twelve or more vertical heating rods equally spaced around the glass section with removable insulation was found to give uniform heat

distribution. The ratio of the heat received at a location nearest to the heating rod to that received farthest from the heating rod was estimated to be 1.1, indicating uniform heating. When a heating wire is wrapped around the glass section, the same ratio of heat received at the hot spot/cold spot was also 1.1. But, since the first method has advantages of easier installation, maintenance, and repair, it was preferred over the second method. The third method considered was commercial radiation heaters. These, however, are not available in large sizes, are not usually recommended for vertical installation, and are expensive. Their use was therefore ruled out.

The top and bottom glass sections are thus heated by vertical heating rods (10 for the 5.1 cm column and 20 for 10.2 cm column) equally spaced around the columns. To avoid any local overheating of the glass, these are mounted a short distance away from the column. The insulation around the glass sections is fabricated such that small sections (~30 cms long) of the insulation can be swung away from the column for visual observation.

The slurry sample taps are provided at 152 cm intervals along the length of the column. At the same locations many pipe couplings are provided which can be used for insertion of different probes (e.g. thermocouples, DP-cell purges etc.)

Since there is no reactor-wax formation by F-T synthesis in the non-reacting columns, a continuous loss of reactor-wax or liquid medium is expected. To recover and recycle the lost reactor-wax, a hot condenser is mounted inside the disengager. It is maintained at steam temperature, 100-121°C. The exiting gas is further cooled and scrubbed off by bubbling it through a solvent which is maintained at chilled glycol temperature. The off gas is finally metered by a dry gas meter.

## 1.2 Engineering Design Status

Preparation of the Engineering Flow, and Piping and Instrumentation diagram of the unit is in progress. The detailed fabrication drawings of all major vessels have been completed. Also, several purchase orders for equipment have been issued.

## 2. Gas Holdup and Bubble Size in Bubble-Columns

### 2.1 Literature Data on Gas Holdup and Bubble Size

Literature data for slurry bubble-columns were analyzed to determine the effect of gas distributor design, column diameter, static liquid height, gas and liquid physicochemical

properties, and experimental conditions on gas holdup and bubble size. The results of this work are used as guidelines for current hydrodynamic studies in hot-flow columns

The literature data are divided into three main sections: studies using F-T wax (relatively few); studies using other liquid media; and design criteria for gas distributor design.

The major highlights from studies using F-T waxes are:

- The data on large-size reactors at high gas velocity are essentially nonexistent.
- Gas holdup is significantly larger and bubble size significantly smaller with F-T derived waxes than with other liquid media.
- The type of gas distributor affects gas holdup and bubble size in small diameter (4.1-10 cm) columns operating in the homogeneous bubbly (laminar) flow regime. There are, however, contradictory results as to the direction and extent of this effect.
- For short columns, column diameter in the range 4.1-10 cm does not significantly affect gas holdup and bubble size.
- Increased static liquid height significantly reduces the gas holdup. Thus, studies in tall, hot-flow columns are needed to simulate gas holdup in reactive columns.

Major highlights from studies using liquids other than F-T waxes are:

- The gas distributor design affects gas holdup and bubble size only in the homogeneous bubbly-flow regime.
- Transition from homogeneous bubbly to slug or turbulent flow occurs at higher gas velocities with sintered-metal plates than with perforated plates.
- Column diameter affects gas holdup to a column/bubble diameter ratio of 40-100.
- Increased solids loading decreases the gas holdup, while pressure has no effect (Shah et al., 1982).

- A widely used design criterion for perforated plate distributors is based on the orifice Weber number.

a. Fischer-Tropsch Waxes

Bubble-column hydrodynamic studies with F-T waxes were performed by three groups: in the Sixties in Germany, Koelbel and co-workers (1968); also in the Sixties in Great Britain, Calderbank, and Farley and Ray (Calderbank et al., 1963; and Farley and Ray, 1964). Recently in Germany, Deckwer and co-workers (Deckwer et al., 1979, 1980, 1981; Zaidi et al., 1979; Quicker and Deckwer, 1981).

A summary of the experimental conditions in these studies is given in Table 2. Most of the work was done in small diameter columns - up to 10 cm ID (except Calderbank et al., 1963; and Farley and Ray, 1964 - up to 25 cm ID), at low gas superficial velocities - up to 4.5 cm/s (except Farley and Ray, 1964 - 7 cm/s, and Koelbel et al., 1968 - up to 23 cm/s).

The majority of the data was obtained in the homogeneous bubbly (laminar) flow regime, except for Koelbel et al. (1968), who measured gas holdup in the slug and transition from homogeneous bubbly to slug flow regimes as well. It is not clear what flow regimes were covered in the 25 cm diameter column operated by Calderbank et al. (1963) and Farley and Ray (1964).

The three flow regimes mentioned above are determined mainly by the column diameter and the superficial gas velocity. Deckwer et al. (1980) plotted a flow regime map (Figure 3) which attempts to qualitatively characterize this dependence. At low gas velocities, the gas flow is characterized by bubbles rising rather undisturbed. The bubble size distribution is narrow, and the interaction between bubbles is small. This is the homogeneous bubbly (laminar) flow regime. At high gas velocities, bubble coalescence and disintegration occur and large bubbles may appear. Although the number of large bubbles may be small, they may contribute considerably to the gas flow as they rise fast. This is the heterogeneous or turbulent flow regime. In small diameter columns (<10 cm), the large bubbles may bridge across the column, forming slugs; this is the slug flow regime. In Figure 3, the large gray areas represent the transition between the various flow regimes. The boundaries shown in Figure 3 are used only for qualitative illustration. The exact boundaries may depend on gas distributor design, static liquid height, operating conditions and gas and liquid media. It is expected that commercial F-T reactors will probably operate in the turbulent regime.



The major highlights from literature studies with F-T waxes are:

- The data on large-size reactors at high gas velocity are essentially nonexistent.
- The gas holdup and bubble size depend on the type of gas distributor.
  - Sintered-metal plate distributors give significantly higher gas holdups than single orifice distributors (Koelbel et al., 1968; and Calderbank, 1963).
  - Contrary to the above, Quicker and Deckwer (1981) observed that a single orifice distributor gives higher gas holdups (also somewhat smaller bubbles) than either a multi-hole perforated plate or a sintered-metal plate.
- The gas holdup is independent of column diameter in the range 4.1-10 cm for columns equipped with sintered-metal plate distributors (Deckwer et al., 1980).
- Increased static liquid height significantly reduces the gas holdup (Calderbank et al. 1963). Deckwer et al. (1979, 1980) claim no such effect for short bubble-columns (static liquid heights in the range 60-95 cm).
- The gas holdup increases with: decreasing catalyst loading (Deckwer et al., 1979, 1980; Zaidi et al., 1963; and Farley and Ray, 1964); sometimes decreasing temperature (Deckwer et al., 1980), and is independent of type of gas (Koelbel and Ralek, 1980). Koelbel et al. (1968) observed, however, that hydrogen produced somewhat lower gas holdup than nitrogen.
- Transition from homogeneous bubbly to slug flow regime initiates in a 3.8 cm diameter column equipped with a sintered-metal plate at superficial gas velocities of 2-4 cm/s (Koelbel et al., 1968).
- Foaming occurs at superficial gas velocities  $>4$  cm/s in columns equipped with sintered-metal plate distributors (Deckwer et al., 1979 and 1980).

Many of the above results can be readily rationalized as follows: Quicker and Deckwer (1981) obtained higher gas holdups with a single orifice distributor than with a

nineteen-hole perforated plate distributor, probably because the orifice Weber number for the former was ~7000 at 4 cm/s superficial gas velocity, while that of the latter was only ~10. The orifice Weber number is defined as the ratio of the gas kinetic energy to the surface energy:

$$We_o = u_o^2 d_o \rho_g / \sigma \quad (1)$$

It is reasonable to expect that the higher the orifice Weber number, the better the gas distribution, as shown by the data of Quicker and Deckwer.

An orifice Weber number cannot be defined for a sintered-metal plate, and the higher gas holdup with the single orifice distributor versus that with a sintered-metal plate cannot be similarly rationalized. It also contradicts other available data (Koelbel et al., 1968; and Calderbank et al., 1963) and Mobil data described in Section 2.2.

Deckwer et al. (1979, 1980) showed that the gas holdup is independent of column diameter in the range 4.1-10 cm. This is in agreement with the criterion derived from data with liquids other than F-T waxes (see Section 2.1b) that wall effects are only important for column-diameter/bubble-diameter less than 40-100. If this criterion is translated to bubble sizes in F-T waxes (0.5-0.7 mm; Quicker and Deckwer, 1981; Deckwer et al. 1980), then wall effects, i.e. column diameter, are not important for  $d_R > \sim 5$  cm.

The effect of static liquid height can be explained by postulating the existence of a gas holdup profile composed of three zones (Langemann and Koelbel, 1967): a bottom dynamic zone in which bubbles form and eventually attain an equilibrium size distribution; a middle stable equilibrium zone, where the gas holdup may decrease slightly due to the static pressure drop; and a zone with very high gas holdup at the top, in which the bubbles disengage from the liquid within a finite time. Langemann and Koelbel (1967) showed that in a given column the top and bottom zones are independent of the static liquid height, while the middle stable equilibrium zone is longer for taller columns and may completely disappear for short columns. Hence for short columns the zone of high gas holdup occupies a large fraction of the column, leading to high overall gas holdups. The opposite is true for tall columns.

Increased catalyst loading results in increased slurry viscosity, which in turn results in larger bubbles and smaller gas holdup (see Section 2.1.b for detail on effect of liquid viscosity). This explains the results of Deckwer et al. (1979, 1980), Zaidi et al. (1979), Calderbank et al. (1963) and Farley

and Ray (1964) on the effect of catalyst loading and viscosity. It contradicts, however, the results of Deckwer et al. (1980) on the effect of temperature: higher temperature results in decreased viscosity and should, therefore, result in higher gas holdup; Decker et al. (1980) obtained lower gas holdup at higher temperatures in a 4.1 cm diameter column, and no change with temperatures in a 10 cm diameter column. This behavior cannot be satisfactorily rationalized, and further study is required.

The contradicting results on the effect of the type of gas (Koelbel and Ralek, 1980; and Koelbel et al. 1968) are not understood at this time.

From the above results it is apparent that the few studies on hydrodynamics with F-T waxes result in many contradictions. Also, these studies deal almost exclusively with the homogeneous bubbly-flow regime, and neglect the commercially important turbulent-flow regime.

#### b. Liquids Other Than F-T Derived Waxes

There is a vast amount of bubble-column hydrodynamic studies with liquids that are not F-T derived waxes (e.g. see reviews by Shah et al., 1982; Ostergaard, 1968; Van Landeghem, 1980; and Mashelkar, 1970). The common denominator in the bubble size and gas holdup studies is that most were done in air-water systems. Some use other gases and liquids, but the liquids are either pure hydrocarbons or aqueous solutions of alcohols.

The bubble size and gas holdup correlations developed from these studies account for the liquid and gas properties, gas superficial velocity, and column diameter. Some of the major correlations for gas holdup and bubble size are summarized in Table 3. When applied to F-T wax conditions, however, these correlations fail, predicting significantly larger bubble size (Quicker and Deckwer, 1981) and smaller gas holdup (Deckwer et al., 1980) than observed. Thus, pure hydrocarbons with the same viscosity, density and surface tension of F-T wax produce 2-3 times larger bubbles and about one-half the gas holdup produced in F-T wax. It may be postulated that mixtures of hydrocarbons behave differently from pure hydrocarbons. This is supported by the very high gas holdups obtained by Langemann and Koelbel (1967) using a hydrocarbon mixture they called mineral oil. It follows that results obtained using liquids other than F-T waxes may only be used as qualitative guidelines.

The major highlights from literature studies with liquids other than F-T waxes are:

- Gas holdup is significantly smaller and bubble size significantly larger than those with F-T waxes (Deckwer et al., 1980). Exceptions may be hydrocarbon mixtures (Langemann and Koelbel, 1967).
- The gas distributor design affects gas holdup and bubble size only in the homogeneous bubbly-flow regime: single nozzles produce lower gas holdups than perforated and sintered-metal plates (Ostergaard, 1980).
- Increased static liquid height decreases the gas holdup (Langemann and Koelbel, 1967).
- Column diameter affects gas holdup up to a column/bubble diameter ratio of 40-100 (Shah et al., 1982; Ostergaard, 1980).
- Increased solids loading decreases the gas holdup, while pressure has no effect (Shah et al., 1982).
- Transition from homogeneous bubbly to slug or turbulent flow occurs at higher gas velocities with sintered-metal plate distributors than with perforated plate distributors. When perforated plates with large holes are used, no homogeneous bubbly flow can be observed at all (Shah et al., 1982).

The above results emphasize the need of:

- Studies with F-T wax in tall hot-flow columns, and if possible, in reactive columns.
- Studies in large diameter columns operating in the high gas velocity regime.

### c. Criteria for Gas Distributor Design

General criteria for gas distributor design are given by Mersmann (1977) and Pilhofer et al (1978). For perforated plates or tubes with small diameter orifices, the design criterion is

$$We_o > 2; d_o < d_o^* = 2.32(\sigma/\rho_g)^{.5}(\rho_g/(\rho_L-\rho_g))^{5/8} \quad (2)$$

Physically, Equation (2) implies that good gas distribution is obtained if the gas kinetic energy at the orifice exceeds the surface energy due to surface tension. For larger orifice diameters, good distribution and "rain" through the holes is prevented if

$$Fr_o = (u_o^2/d_o g)(\rho_g/(\rho_L - \rho_g))^{5/4} > .37; \quad d_o > d_o^*, \quad (3)$$

where  $Fr_o$  is the Froude number.

An additional consideration for gas distributor design is the allowed maximum pressure drop across the distributor. The pressure drop across a perforated plate may be estimated by the conventional orifice equation (Fair, 1967).

$$\Delta P = (\rho_g/2g)(u_o/C_v)^2 \quad (4)$$

Equation (4) neglects the velocity of approach to the orifice. According to Fair, a good approximation for  $C_v$  is 0.8.

Literature data on gas distributors used in Fischer-Tropsch bubble-column reactors were analyzed and compared with criteria (2) and (3). The results are shown in Table 4. It is apparent that the designs are very conservative compared to criterion (2). For example, Quicker and Deckwer (1981) used a single orifice with  $We_o = 7220$  at 4 cm/s superficial gas velocity. Even Quicker and Deckwer's perforated plate had  $We_o = 11.4$ , which is ~5 times larger than that required by Equation (2). Data by Farley and Ray (1964) indicate that the Froude number satisfies criterion (3), although this design appears considerably less conservative than that of the smaller orifices used in the other works. Note that Quicker and Deckwer obtained significantly higher gas holdups with a single orifice than with a nineteen-hole perforated plate. This is consistent with the higher  $We_o$  for the single orifice. In general, it would appear that for small orifices, the higher  $We_o$ , the better the gas distribution.

The importance of the distributor design depends on the flow regime, the physical properties of the liquid medium, and even the height of the column. Thus, literature results indicate that distributor design may not be important in the turbulent-flow regime, or for liquid media which favor coalescence and disintegration, or for very tall columns

(e.g. Shah et al., 1982; Mashelkar, 1970). In addition, other criteria may replace criteria (2) and (3) (e.g. Kastanek et al., 1980; Bhavaraju et al., 1978; Kurten and Zehner, 1979; Shah et al., 1982). These shall be further examined.

## 2.2 Scoping Studies Using Small Hot-Flow Columns

Bubble-column hydrodynamics were examined using two small hot-flow columns operating at atmospheric pressure. The effects of distributor type, liquid medium, and column diameter were investigated in this study.

The major highlights of the work are:

- Sintered-metal plate distributors having average pore sizes of 15 and 60  $\mu\text{m}$  produce very small bubbles and a great deal of foam, with holdups as high as 70 vol % at superficial gas velocities above 0.8 and 1.4 cm/s respectively.
- For sintered-metal plate distributors, gas holdup decreases with increasing pore size. This is accompanied by larger bubbles and less foam.
- Single orifice distributors produce smaller bubbles as the orifice diameter is decreased, but slugs form at higher velocities.
- Distributors with small orifices can give holdups similar to large-pore sintered-metal plate distributors. Bubble size distributions, though, are different.
- F-T derived waxes behave much differently than hexadecane, supporting literature findings (Section 2.1).
- Column diameter, in the range 3.2 to 5.3 cm ID, appears to have some effect on hydrodynamics. The extent of the effect varies with the distributors.

### a. Description of Small Hot-Flow Columns

To study the effect of distributor type on the gas holdup, two small hot-flow columns were used, 3.2 and 5.3 cm in diameter. Various types of distributors (sintered-metal plates and single orifices) can be sealed into removable joints and then clamped to the bottom of the columns, with o-rings providing tight seals. The columns are heated with strands of Nichrome heating wire, with the smaller column having three such zones, and the larger column two. Both columns are mounted with glass tubes covering their entire lengths. This serves to insulate the columns, as well as provide safety. Inside, a 6.3 mm diameter thermowell containing four thermocouples runs the length of the columns to record fluid temperatures.

Nitrogen is metered by a rotameter and introduced below the distributor as the feed gas. After leaving a column, the gas is bubbled through a solvent and then passed into a wet-test meter to double-check the flowrate. The bubbles and their flow patterns are visible between the wire strands, and gas holdup is determined by visual observation of the height of the expanded liquid.

#### b. Effect of Distributor Type

Figure 4 is a plot of the gas holdup observed in the 3.2 cm ID column over various sintered-metal plate (SMP) distributors. Also shown is the correlation proposed by Deckwer et al. (1980). The liquid medium used in our experiments was an F-T derived paraffin wax, FT-200 (also known as Vestowax EH-100)<sup>(1)</sup>. This wax is similar in average molecular weight to that used by Deckwer et al. The static (unexpanded) liquid height varied from 50 to 100 cm, while Deckwer et al. reported using static heights of 60 to 100 cm with no significant variation in holdups.

The curves in Figure 4 illustrate the major difficulty we have encountered so far: foam, or regions of very high gas holdups. Foam is undesirable since catalyst loadings per reactor volume would become very low in such systems. The regions of sharpest slope on this plot represents conditions under which the liquid begins to foam at the top, characterized by a sharply visible boundary between the swirling small bubbles below, and the rigidly held, slowly rising bubbles above.

As the gas velocity is increased, the boundary becomes less and less distinct as it moves slowly down the column, while the overall height of the suspension increases dramatically. This is because the mixture is continually being converted to high-holdup foam. This process appears to be self-propagating; that is, beyond a certain superficial gas velocity (approximately 0.8 and 1.4 cm/s for the 15 and 60  $\mu\text{m}$  distributors, respectively) the foam inexorably grows until a maximum holdup is reached where the entire column is only foam. At this point, the holdups are in the 60-75 vol % range in all cases.

It should be noted that although no exact measurements of bubble size were attempted, visual observation of both foam and non-foam regimes showed the bubbles to be very small and densely packed, with little variation in size.

The 100  $\mu\text{m}$  SMP, however, produced significantly less foam than either of the other two distributors. In addition, the

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(1) A F-T paraffin wax probably from SASOL, with an average molecular weight of 600.



bubbles in the bulk liquid appeared to be larger and less densely packed. This produced a stable suspension at all velocities studied. In fact, at the higher velocities, large slugging bubbles could be observed coalescing near the top of the column and rising through the foam layer. This, combined with the larger average bubble size, may have helped keep the foam from propagating. It appears, then, that larger pore size SMP's produce larger average bubble sizes (leading to enhanced coalescence), less foam, and therefore, lower gas holdups.

The results obtained from single orifice distributors were decidedly different (Figure 5). For both 0.5 and 0.32 mm diameter orifices the holdups were substantially lower than those from the sintered-metal plate experiments. In addition, the bubbles varied greatly in size, with large, mushroom-cap bubbles rising quickly past smaller, swirling ones. This is in direct contrast to those observed by Quicker and Deckwer (1981), who reported that the bubbles obtained from a 0.9 mm orifice were both small and uniform, much the same as with SMP results. In that case, however, the inside column diameter was 9.5 cm, so that nearly nine times as high a gas volumetric flowrate was needed to achieve the same superficial gas velocities as our 3.2 cm column. This provides a higher gas jet velocity through the orifice, supplying more kinetic energy for bubble breakup. This, of course, corresponds to higher orifice Weber numbers.

A comparison shows that for a superficial gas velocity of 3.0 cm/s, the Weber number for the 0.57 mm orifice was 143, and 0.39 mm orifice was 447, while that of Quicker and Deckwer's experiment was 3150 (estimated). The gas holdups reported in that study were very similar to the results from the 0.25 mm orifice, also shown in Figure 5. In this case, the Weber number at 3.0 cm/s was 1700. At the higher velocities, there were indeed many more small bubbles than the 0.57 and 0.39 mm orifices produced; however, large bubbles were clearly seen rising through the fine swarm. In addition, foam was observed, though not as much as produced by sintered-metal plates. Again, it seems that the presence of the large bubbles limits the height that the foam can reach.

At the low velocities, flow patterns were similar to those observed with the other two orifices; that is, mostly large, fast-rising bubbles. The gas jet from the orifice was only visible at the low velocities, and could be seen dissipating immediately upon leaving the orifice. At higher velocities the jet was obscured by swirling bubbles.

In conclusion, several general trends were observed:

- For sintered-metal plate distributors, the gas holdup decreases with increasing pore size. This is accompanied by larger bubble sizes and less foam, with some slugging occurring at the higher velocities.
- Single orifice distributors produce smaller bubbles as the orifice diameter is decreased, but slugs form at higher velocities, helping to break the foam.
- Distributors with small orifice can give gas holdups similar to large-pore sintered-metal plate distributors, but with different bubble-size distributions. It may be that the slug-forming mechanism is different in the two cases, i.e. bubble coalescence vs. unsteady jet breakup.

### c. Effect of Liquid Mediums

The consequences of operating a bubble-column with a liquid which does not simulate the actual performance can be very severe. For this reason, the 5.3 cm ID glass column was used to compare the F-T derived paraffin-wax (Vestowax FT-200) which we were using to hexadecane. Hexadecane has been used by other observers in cold-flow studies, due to its similarity to wax in both surface tension and viscosity.

Using the same 15  $\mu\text{m}$  sintered-metal plate distributor for both liquids, gas holdups were determined for FT-200 wax at 200°C, and hexadecane at room temperature. The results were vastly different, as shown in Figure 6. Also shown are the viscosity and surface tension comparisons. It is obvious that the two mediums behave very differently. The wax formed the same small, swirling bubbles reported in Section 2.2.b. In addition, foam was present at all velocities, which contributed significantly to the holdup.

Hexadecane, however, formed relatively large, uniform bubbles which were much less densely packed than the wax case. No foam was observed at any time, but at the higher velocities some coalescence was seen taking place toward the top of the column. The gas holdup was much lower than that in the wax, even if the effect of the foam is removed from the gas holdup data (the dotted line in the figure). The removal of the effect of the foam was done by subtracting the foam height from the overall expanded bed height, and then (assuming that the foam was 70 vol % gas) calculating how much liquid was remaining.

This experiment illustrates the difference that the liquid medium can make when modeling bubble-columns. This evidently makes hot-flow modeling a must for the F-T systems.

#### d. Effect of Column Diameter

To examine the influence of column diameter on hydrodynamics, sintered-metal plates as well as an orifice were used as gas distributors in the 5.3 cm ID hot-flow column. The results of the gas holdup measurements are shown in Figure 7. The curve for the 100  $\mu\text{m}$  SMP is similar to the corresponding curve for the 3.2 cm ID model (Figure 4). The major difference, however, was the apparent absence of slugging in the large column. This helped keep the holdups slightly larger at the higher velocities in the large column. The bubbles in the non-slugging regime seemed to be similar in average size in both cases and foam was present, though again not as much as was produced by the 60  $\mu\text{m}$  SMP.

The 60  $\mu\text{m}$  distributor produced a great deal of foam at gas velocities nearly one-half the rate required in the smaller column to achieve similar holdups. Flow patterns and bubble sizes appeared similar to the smaller column results, with small, swirling bubbles of essentially uniform size occupying the non-foam regions.

We see, then, that for sintered-metal plate distributors the same general trends are present in both columns, but with the larger diameter column providing higher holdups. The magnitude of the holdup difference varies with the distributor.

In addition to SMP's, an orifice of 0.37 mm diameter was used in the larger column to obtain higher orifice Weber numbers. It was calculated that at 3.0 cm/s, the Weber number for the gas jet was 3900, significantly higher than any orifice used in the 3.2 cm ID column. As expected, the holdup produced by this distributor (Figure 7) was much higher than that of any other orifice studied. Also, though the bubbles were very small in size and foam was present when this orifice was used, the holdup was still significantly lower than the 60  $\mu\text{m}$  SMP, which produced similar size small bubbles.

It was possible, however, to observe slug-type motion at the higher velocities. This was visible as an undulation in the foam layer and an occasional rapid motion of the bubbles close to the wall. The column was otherwise too dense to see through. The high holdup produced by the tiny, intensely swarming bubbles was evidently reduced by the presence of large bubbles, much the same phenomenon as was discussed in Section 2.2.b. This suggests that the column diameter was not a major factor under these circumstances. Presumably an orifice which produced similar Weber numbers in the smaller column would show holdups in close agreement with this orifice. This has yet to be proven.

It is interesting to note that the orifice distributor produces holdups significantly higher than does the 100  $\mu\text{m}$  sintered-metal plate distributor at superficial velocities above about 2 cm/s. This shows that two very different flow patterns can produce effectively the same holdups (uniform bubbles vs. a combination of smaller and larger ones). Whether or not these two patterns would give rise to similar conversions in a reactor is yet to be studied.

### 2.3 Scoping Studies Using a Small Bubble-Column Reactor

#### a. Effect of Pressure and Gas Type

As mentioned in Section A.2, the installation of a new DP-cell set-up on the small bubble-column reactor (CT-225) enabled the measuring of gas holdups to a certain extent. This was seen as an excellent opportunity to examine the effect of several parameters which were invariant in the glass models, one of them being pressure.

The new DP-cell set-up was able to provide gas holdup data because of the reactor design. A look back at Figure 1 shows the disengager at the top of the reactor. This is nothing more than an expanded section designed to prevent carryover of slurry to the downstream lines. However, it was realized that if liquid (slurry) was displaced from the 2.6 cm diameter reactor section to the 7.1 cm diameter disengager section by the introduction of gas, then the total hydraulic head recorded by the DP-cell would drop. -Therefore, by knowing the change in the DP reading for given flowrate and the cross-sectional area ratio of the two sections, the gas holdup in the narrow section could be calculated.

Each gas holdup consequently corresponds to a specific amount of liquid in that section. In effect, since the height of the lower section remains fixed, the data can be viewed as being taken at a "constant expanded height." This differs somewhat from the more straightforward technique used with the glass columns, in which static height was constant and the entire expanded bed, including foam, was measured. Nevertheless, gas holdups were measured using a slurry consisting of 15 wt % of standard F-T catalyst in a liquid medium which combined a wax product from a previous run and Mobil base stock F-509.<sup>(1)</sup> The temperature was held at 177°C for all experiments, and the distributor was a 20  $\mu\text{m}$  sintered-metal plate.

The results at different pressures using nitrogen as the feed gas are shown in Figure 8. Also shown is a series of

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<sup>(1)</sup>A proprietary high molecular-weight paraffinic base stock.

points representing hydrogen, in an effort to determine whether the type of gas affects the gas holdup. It can be seen on the plot that all the data seem to fall on the same general curve. The maximum observed holdup is about 59 vol % at 1.75 cm/s superficial gas velocity. Beyond that the holdup falls off, possibly due to the onset of large bubble formation. At all velocities, however, the holdup is substantially higher than the correlation proposed by Deckwer et al. (1980). This correlation, though, is valid only for temperatures above 250°C. Below that, the authors state that the gas holdup (in a paraffin wax) goes up with decreasing temperature. However, even after correcting the correlation as recommended, the predicted holdup is still exceeded by this data.

The close agreement of the data over the wide range of pressures studied, as well as the results for hydrogen, indicate no gas density effect on the holdup and, presumably, the average bubble sizes. A comparison of Figures 4 and 8, meanwhile, shows that the holdups in the short hot-flow columns (which used only F-T derived wax) are higher than that in the reactor. However, note that the entire expanded bed is not seen here, and there might well exist foam which, if the reactor had no disengagement section, might add a good deal of gas holdup to that already reported.

### 3. Future Work

- The construction of the two tall, hot-flow non-reacting columns will be completed.
- Scoping hydrodynamic studies will be continued using the two small, hot-flow non-reacting columns and the small bubble-column reactor of Unit CT-225.

V. NOMENCLATURE

$C_v$	Empirical constant, (cm/s)
$d_B$	Bubble diameter, (cm)
$d_c$	Catalyst particle diameter, ( $\mu\text{m}$ )
$d_o; d_o^*$	Orifice diameter; critical orifice diameter, (mm)
$d_R$	Reactor diameter, (cm)
$g$	Gravitational acceleration, ( $\text{cm/s}^2$ )
$L$	Static slurry height, (cm)
$L_e$	Expanded slurry height, (cm)
$n_o$	Number of orifices or nozzles
$P$	Pressure, (atm or MPa)
$T$	Temperature, ( $^{\circ}\text{C}$ )
$u_g$	Gas superficial velocity, (cm/s)
$u_o$	Gas orifice or nozzle velocity, (cm/s)
$u_L$	Liquid superficial velocity, (cm/s)
$w_c$	Catalyst loading in slurry, (g Cat/g slurry)

Greek Letters

$\epsilon_g$	Gas holdup
$\rho_g$	Gas density, ( $\text{g/cm}^3$ )
$\rho_L$	Liquid density, ( $\text{g/cm}^3$ )
$\mu_L$	Liquid viscosity, ( $\text{g/cm-s}$ )
$\sigma$	Surface tension, (dyne/cm)

Dimensionless Numbers

$Fr_o$	Froude number (orifice), $u_o / (gd_o)^{0.5}$
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$We_o$  Weber number (orifice or nozzle),  $u_o^2 d_o \rho_g / \sigma$

Acronyms

SMP Sintered-Metal Plate

DP Differential Pressure

VI. Literature

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Table 1

Design Basis of Two Tall,  
Hot-Flow, Non-Reacting Columns

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	Design Range	
	Column 1	Column 2
<b>Column Dimensions</b>		
Inside Diameter, cm	5.1	10.2
Height, cm	914	914
<b>Operating Conditions</b>		
Temperature, °C	121-131	121-316
Pressure, kPa	0-207	0-207
Gas Flow Rate, Nm <sup>3</sup> /hr	0-1.4	0-6.8
<b>Other Operating Conditions</b>		
<b>Preheater</b>		
Inlet Temperature, °C		Ambient
Exit Temperature, °C		121-316
Pressure, kPa		0-207
<b>Hot Condenser</b>		
Inlet Temperature, °C		121-316
Exit Temperature, °C		100-121
Pressure, kPa		0-207
<b>Cold Condenser-Scrubber</b>		
Inlet Temperature, °C		100-121
Exit Temperature, °C		Ambient
Pressure, kPa		0-207

Table 2  
Summary of Bubble-Column Hydrodynamic Studies

Ref.	Column		Distributor		Conditions			Liq (2)	Gas	Flow (3) Regime	Quantity Measured			
	dR	L	d <sub>o</sub>	no Type (1)	u <sub>g</sub>	w <sub>c</sub>	d <sub>c</sub>					T	P	
Deckwer et al. (1979, 1980)	4-10	60-100	.0075	-	SP	0-4	0-16	5	143-270	.4-11	MP	N <sub>2</sub>	E	ε, d <sub>B</sub>
Zaidi et al. (1979)	"	"	"	-	"	.7-3.8	2-14	1	250-290	10	"	CO/N <sub>2</sub>	"	"
Quicker and Deckwer (1981a,b)	9.5	135	.9, 1.1	1, 19	PP	0-4	-	-	130-170	NA	FT300	N <sub>2</sub>	"	"
Koalbel et al. (1968)	3.8	300	.0075-.1	-	SP	1-23	0-10	5-60	230-270	2-11	MP	H <sub>2</sub> , N <sub>2</sub> H <sub>2</sub> /CO	B, S	ε <sub>g</sub>
Calderbank et al. (1983)	5.1	230-460	NA	1	SH	0-4.5	NA	NA	265	2	KW	H <sub>2</sub> /CO	NA	"
Farley and Ray (1964)	25.4	920	1.9	1	"	7.3	1.3	1-3	265	"	"	"	"	"
Kuo (1983)	3.2-5.3	46-100	.0016-.0060	-	SP	0-3.5	0-15	NA	200-230	1	FT200 FT300 PW	N <sub>2</sub>	B	"
"	"	"	.32-.50	1	PP	"	"	"	"	"	"	"	"	"
"	5.1	305-762	.001-.002	-	SP	0-4	2-14	"	260-267	11.2-25	FT200 PW	H <sub>2</sub> /CO	NA	"

(1) SP - Sintered Plate  
PP - Perforated Plate  
SN - Single Nozzle

(2) MP - Molten Paraffin  
KW - Krupp Wax  
PW - Product Wax

(3) B - Homogeneous Bubbly  
S - Slug

TABLE 3: CORRELATIONS FOR GAS HOLDUP AND BUBBLE SIZE  
(Shah et al, 1982)

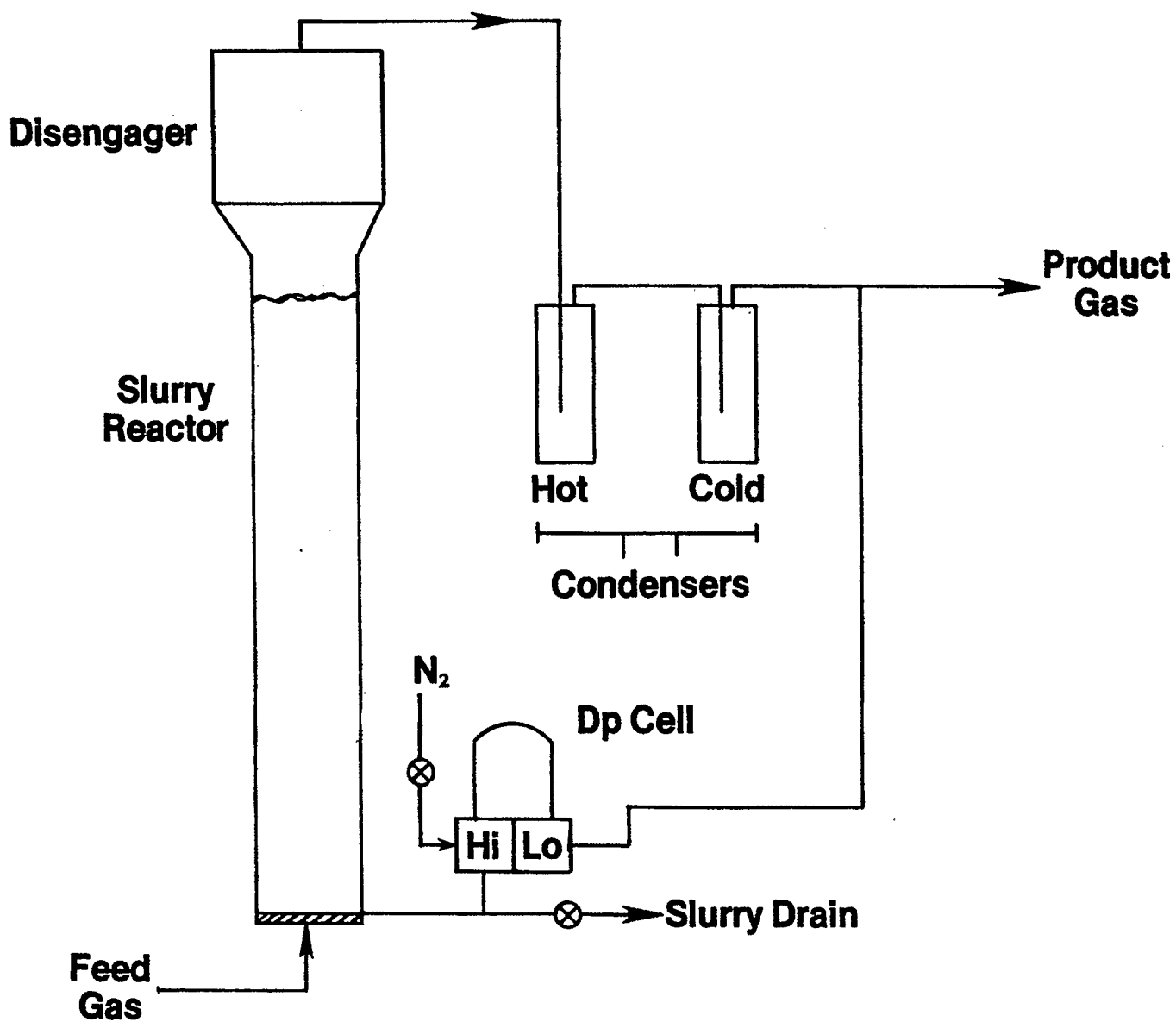
<u>System</u>	<u>Range of Parameters</u>	<u>Correlation Proposed</u>	<u>Reference</u>
Air-Water Air-Kerosene Air-Na <sub>2</sub> SO <sub>3</sub> aq. soln.	u <sub>g</sub> , cm/s: 4-45 d <sub>R</sub> , cm: >10	$\epsilon_g = \frac{1}{2 + (0.35/u_g)(\rho_L/72)^{1/3}}$	Hughmark (1967)
Air-Glycerol Air-Light Oil Air-ZnCl <sub>2</sub> aq. soln.	u <sub>g</sub> , cm/s: 0.3-40 u <sub>L</sub> , cm/s: 0-4.4 d <sub>R</sub> , cm: 15.2-60 L, cm: 126-350	$\frac{\epsilon_g}{(1-\epsilon_g)^4} = \left( \frac{gd_R^2 \rho_L}{\sigma} \right)^{1/8} \left( \frac{gd_R^3}{v_L} \right)^{1/12} \left( \frac{u_g}{gd_R} \right)^{1/2}$	Akita and Yoshida (1973)
Air-Different Liquids	u <sub>g</sub> , cm/s: 1-8 d <sub>R</sub> , cm: 7.56-61 L, cm: 2-350	C=0.2 for pure liquids and non-electrolytes C=0.25 for electrolytes $\frac{d_b}{d_R} = \frac{d_R^2 g \rho_L}{26 \left( \frac{\sigma}{v_L} \right)^{-0.5} \left( \frac{gd_R^3}{v_L} \right)^{-0.12} \left( \frac{u_g}{gd_R} \right)^{-0.12}}$	Gestrich and Rahse (1975)
Air-Alcohols Air-Halogenated Hydrogens	u <sub>g</sub> , cm/s: 0-10 d <sub>R</sub> , cm: >10 L, cm: >120	$\epsilon_g = .89 \left( \frac{L}{d_R} \right) 0.036 (-15.7 + \log K) \left( \frac{d_b}{d_R} \right) 0.3 \left( \frac{u_g}{d_b g} \right)^{2+0.025(2.6+\log K)}$ $K = \rho_L \sigma^3 / \mu_L^4 g$ d <sub>b</sub> = .3 cm $\frac{\epsilon_g}{(1-\epsilon_g)} = 0.115 \left( \frac{u_g^3}{v_L g (\rho_L \rho_g) / \rho_L} \right)^{0.23}$	Bach and Pilhofer (1978)

Table 4

## Gas Distributor Designs in F-T Bubble-Columns

	Quicker and Deckwer (1981)		Farley and Ray (1964)
Column	$d_R$ , cm	9.5	24.8
Orifice	$h_o$	1	1
	$d_{o\ddagger}$ , mm	0.9	19.1
	$d_o$ , mm	2	2
	$u_o$ , cm	4	7.2
Conditions	T, °C	130-170	275
	P, MPa	0.1-1.0	1.14
	$\Delta P$ , MPa	0.007	0.014
	$We_o$	7220	550
	$Fr_o$	-	1.05

FIGURE 1

SCHEMATIC DIAGRAM OF Dp CELL  
ARRANGEMENT ON UNIT CT-225

# FIGURE 2

## SIMPLIFIED FLOW DIAGRAM OF HOT-FLOW, NON-REACTING BUBBLE COLUMN

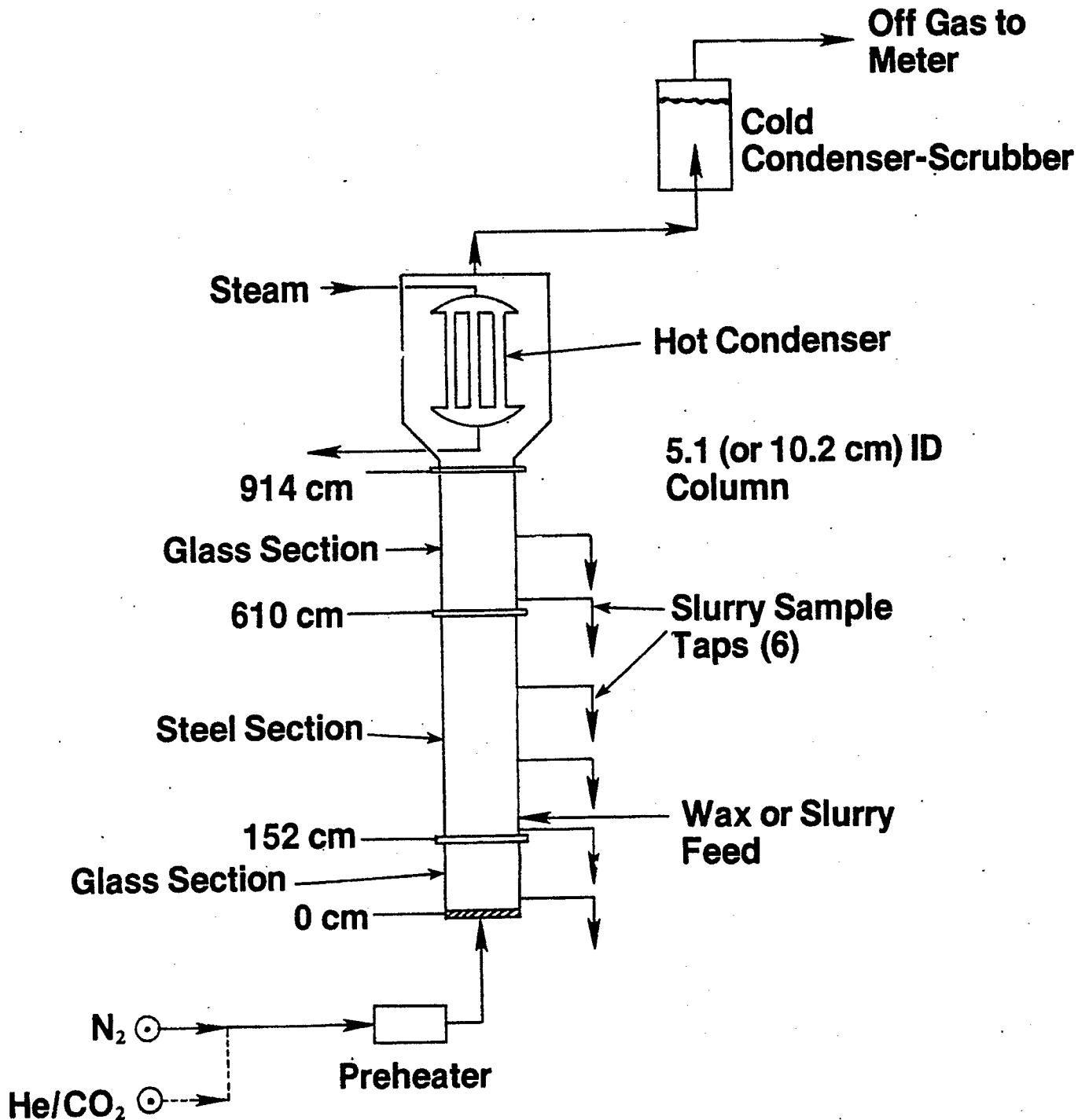


FIGURE 3

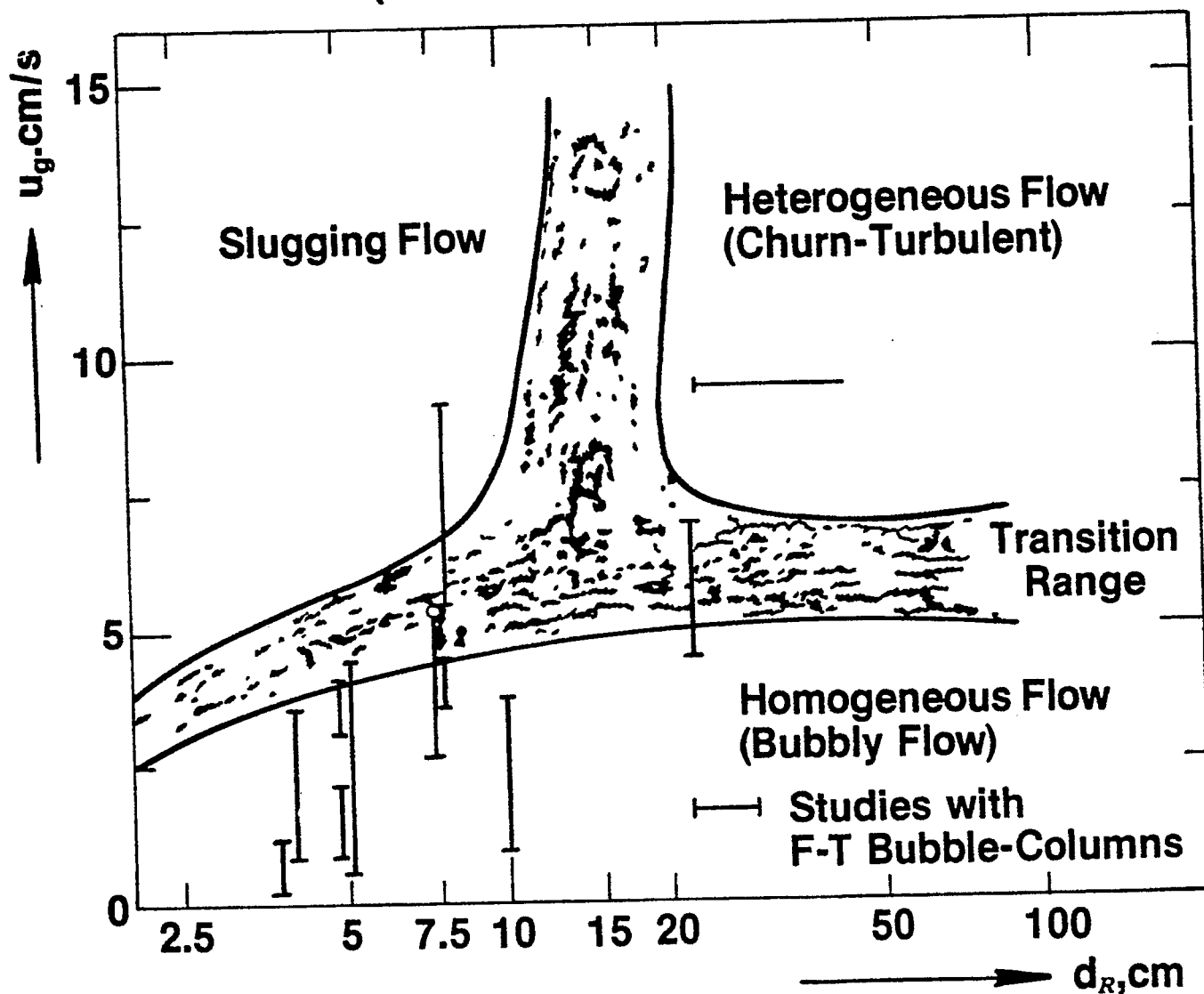
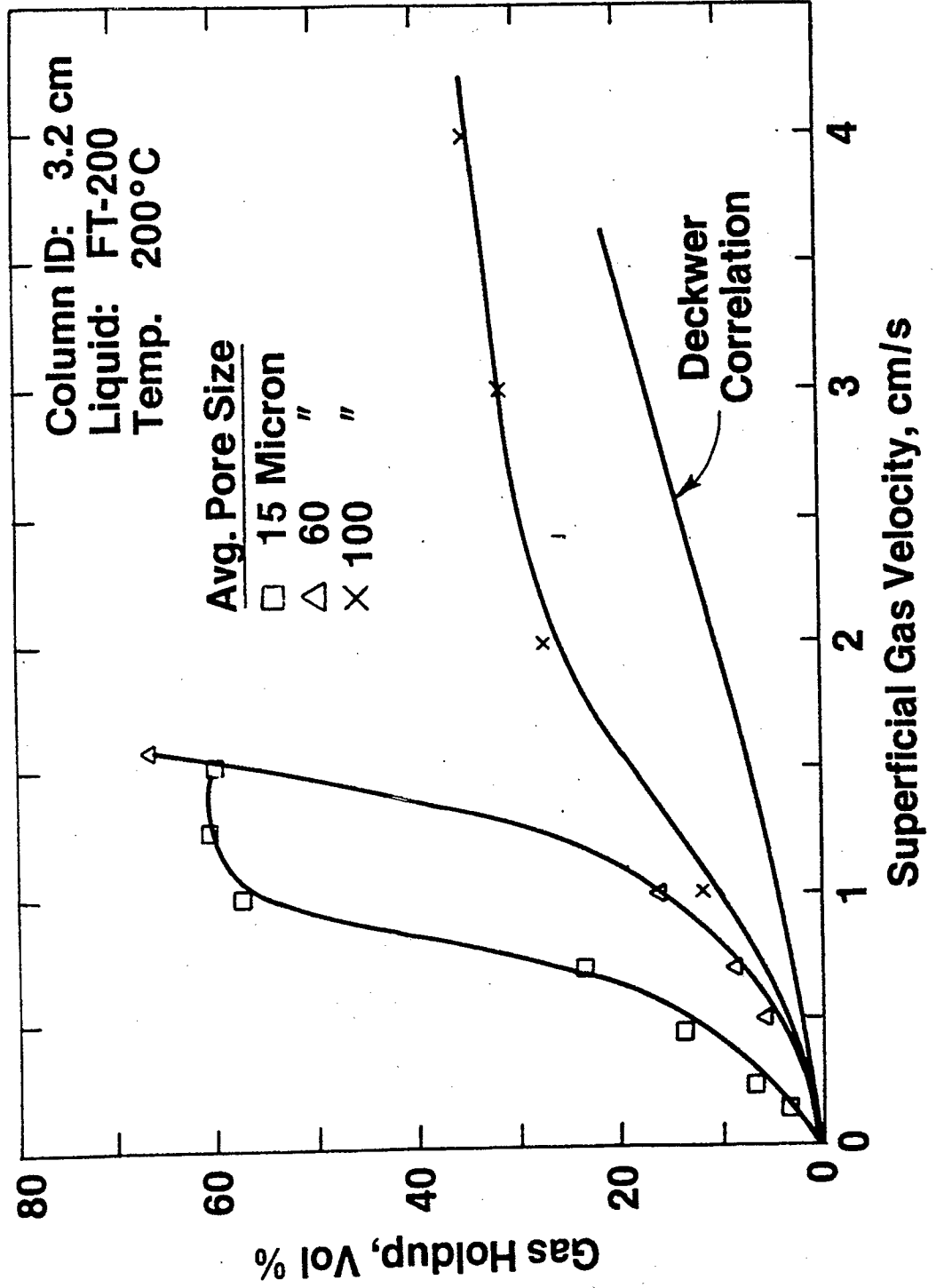
**BUBBLE-COLUMN FLOW-REGIME MAP  
(Deckwer, et al 1980)**

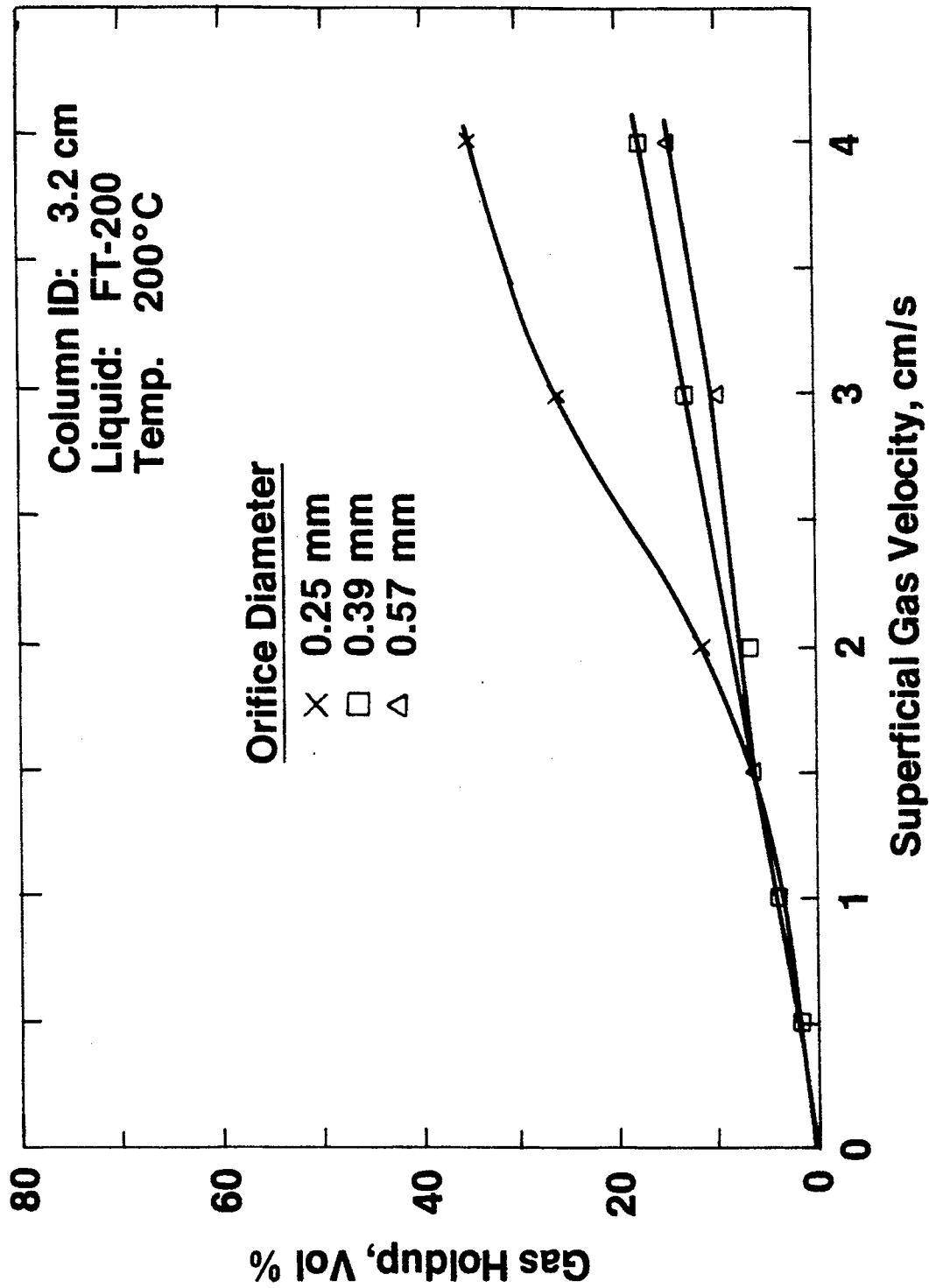


FIGURE 4

EFFECT OF SINTERED-PLATE PORE SIZE  
ON GAS HOLDUP

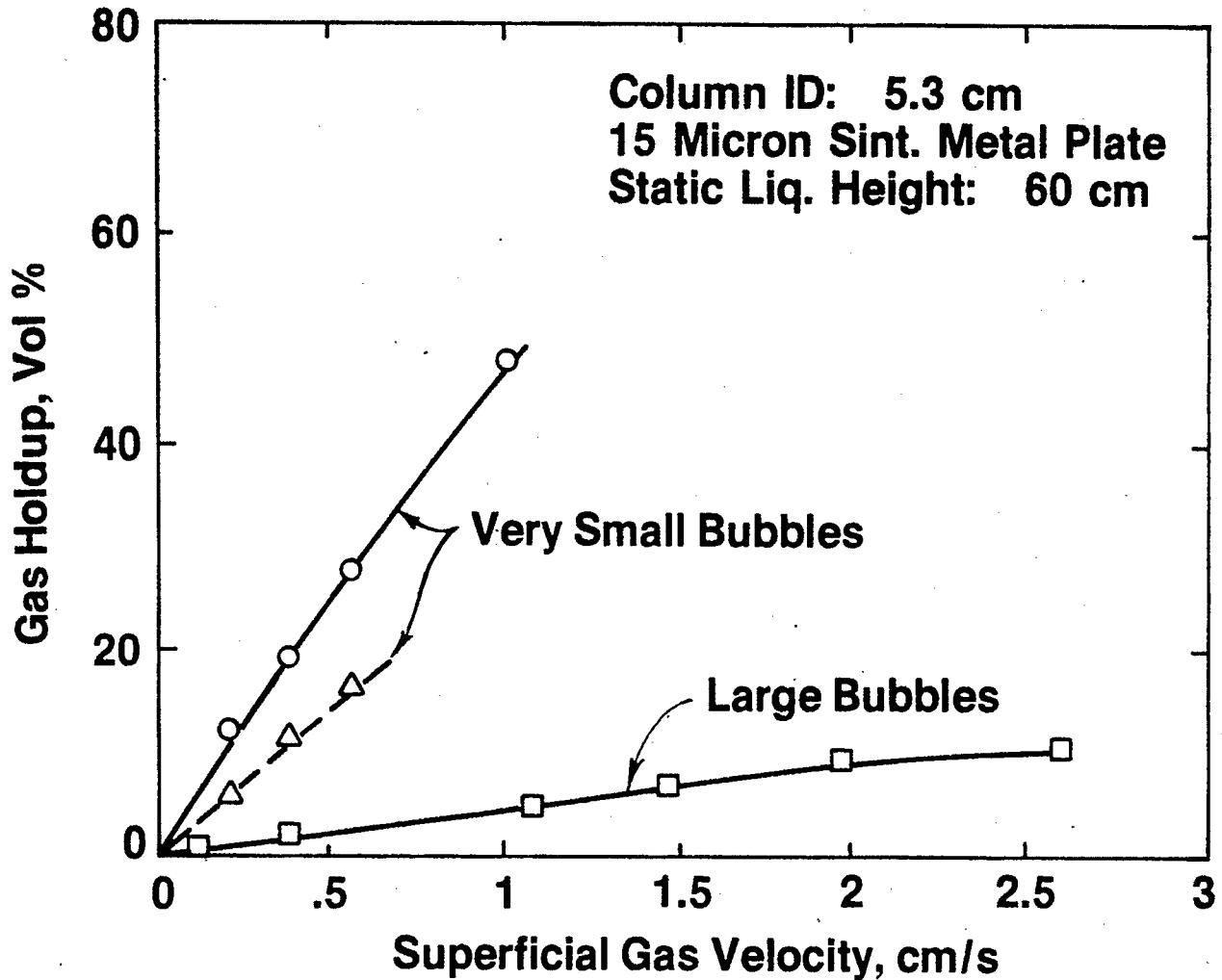


**FIGURE 5**  
**EFFECT OF ORIFICE DIAMETER ON GAS HOLDUP**  
**(Single-Orifice Distributor)**



# FIGURE 6

## EFFECT OF LIQUID MEDIUM ON GAS HOLDUP

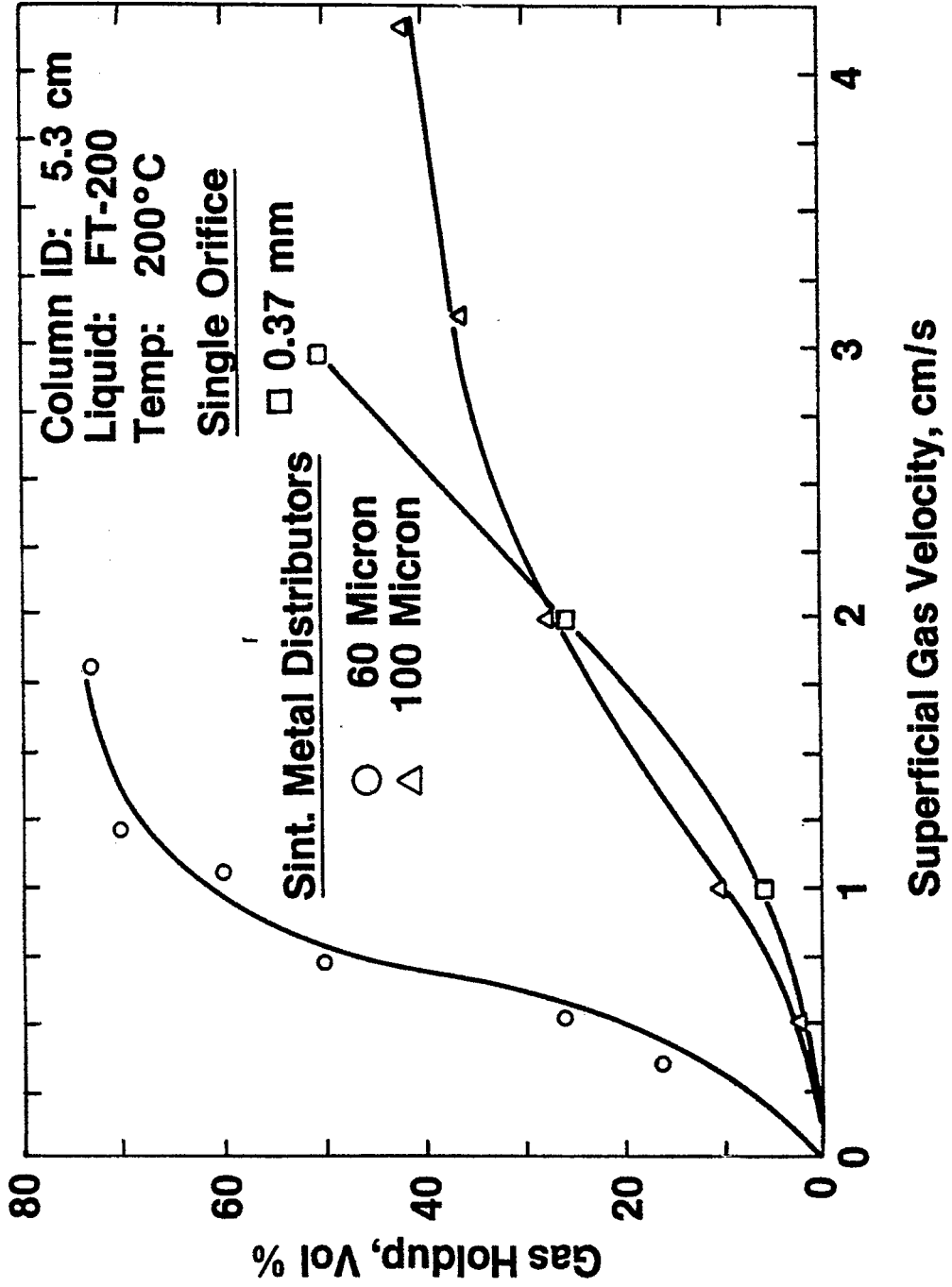


△ FT-200 Excluding Foam

	○	□
Liquid	FT-200	Hexadecane
Temp., °C	200	20
Visc., cp	2.1	3.5
Surf. Tension, Dynes/cm	26.0	27.5

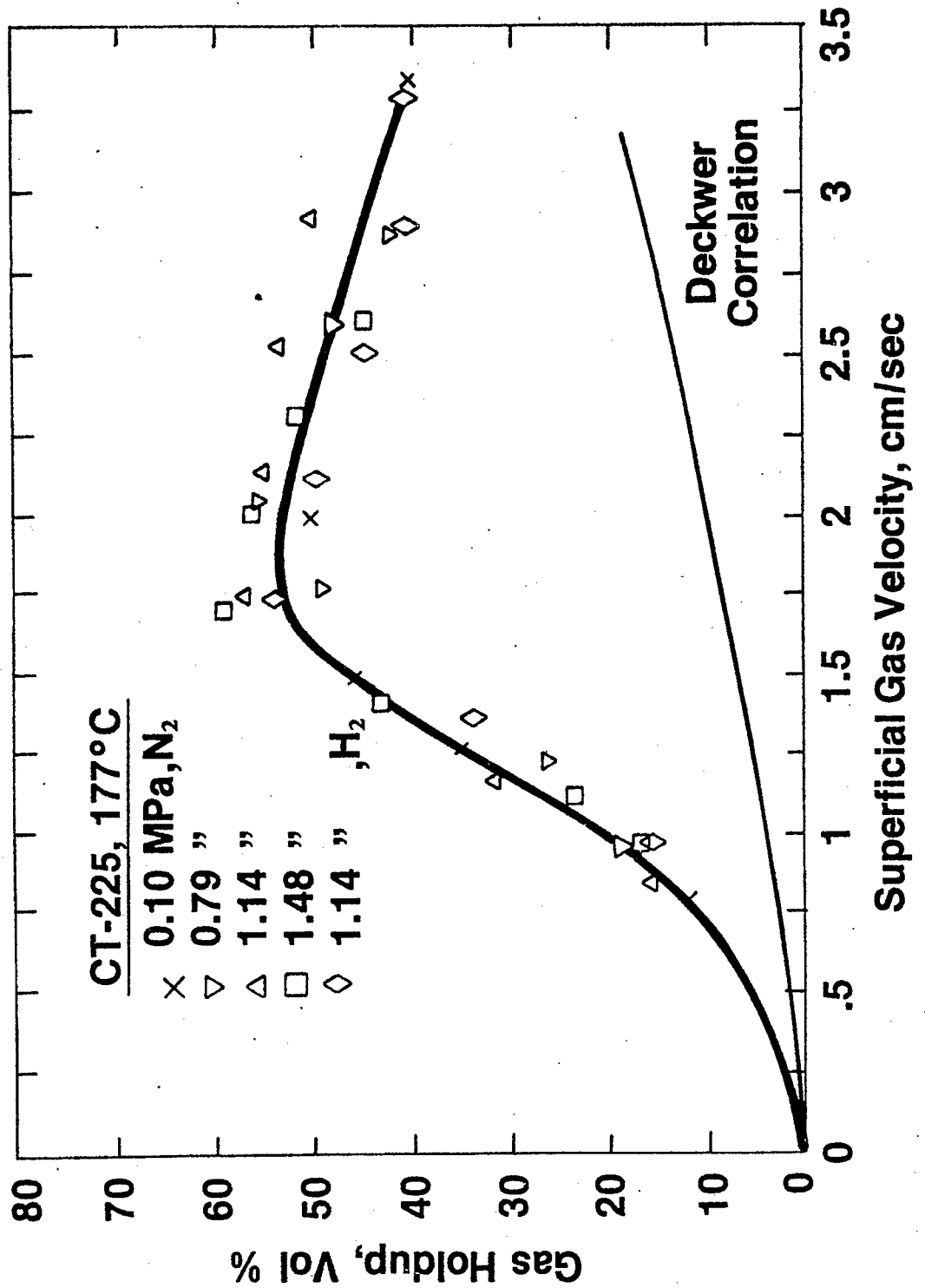
FIGURE 7

**EFFECT OF DIFFERENT GAS DISTRIBUTORS  
ON GAS HOLDUP  
(Small Hot-Flow Column)**



**FIGURE 8**

**EFFECT OF PRESSURE & GAS TYPE  
ON GAS HOLDUP**



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