II. MEASUREMENT OF GAS HOLDUPS BY CONVENTIONAL TECHNIQUES

Gas holdups and solids concentration profiles were measured using conventional techniques. In particular, gas holdup was calculated from visual observations of the expanded and static liquid height in the glass columns, and from measurements of differential pressures and solids concentrations in the stainless steel columns. The experimental apparatus, operating conditions, data reduction procedures, and results from both two–phase and three–phase experiments are described below. Also, empirical correlations which may be used to predict overall (or average) gas holdup in a Fischer–Tropsch slurry bubble column reactor are presented.

Experimental Apparatus and Operating Procedure

Figure 2.1 is a schematic representation of the slurry bubble column apparatus which was constructed for these studies. The majority of experiments were conducted in 0.05 and 0.21 m ID by 3 m tall stainless steel columns. Experiments in both the batch mode (i.e. without slurry circulation) and continuous mode (i.e. with slurry circulation) of operation were conducted in the stainless steel columns. Five pressure transducers (Valydine Model DP 15) and five slurry sampling valves (1/4" Whitey ball valves) with pneumatic actuators were located along the column (see Figure 2.9 for their locations).

The flow rate of prepurified nitrogen from gas cylinders was measured and controlled by a Brooks Model 5816 mass flow meter for experiments conducted in the 0.05 m ID column. A Sierra Series 840 mass flow meter was used to measure the gas flow rate during experiments conducted in the 0.21 m ID column. For the 0.21 m ID column, the flow rate was controlled manually by adjusting the outlet pressure from the nitrogen cylinder (cryogenic). Prior to each series of experiments, the mass flow

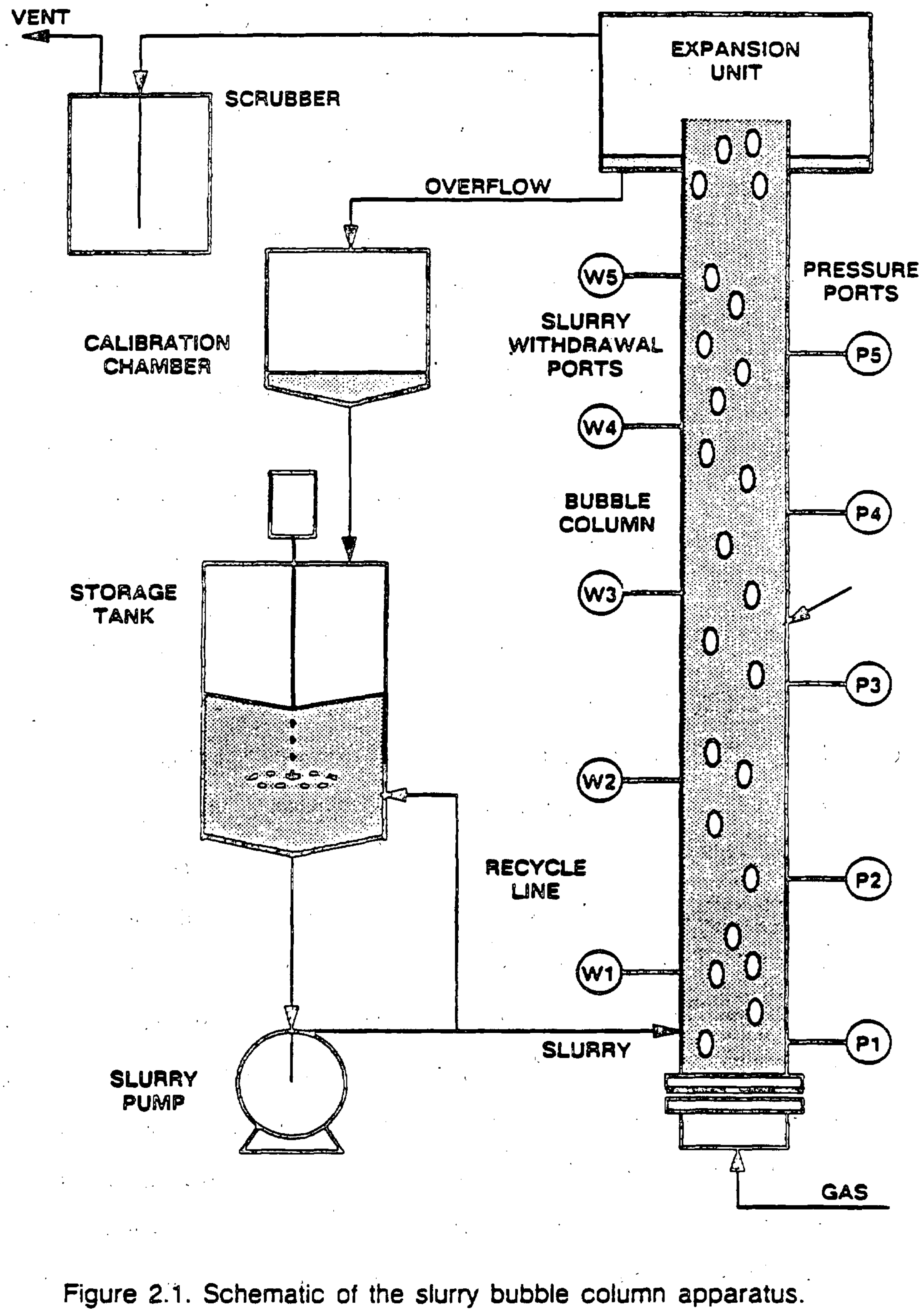

Figure 2.1. Schematic of the slurry bubble column apparatus.

meters were calibrated. The Brooks mass flow meter was calibrated using a wet test meter, and the Sierra mass flow meter was calibrated using a flow prover (i.e. an orifice meter). The metered gas entered the bubble column through the distributor which was located between two flanges at the bottom of the column. For experiments in the 0.21 m ID column, the gas was passed through an electrically heated U–shaped preheater before entering the column at the distributor. The gas inlet temperature was manually controlled using two variable voltage transformers. The temperature of the gas was monitored by three thermocouples (one located in the middle of the preheater – 0.21 m ID column only; one located after the preheater; and one located just below the distributor). The thermocouples were connected to an Omega (Model 199) ten channel temperature indicator.

The wax was charged in the storage tank and the tank was electrically heated to bring the wax to the desired temperature. The wax storage tank for the large diameter column was 0.61 m in diameter and 0.91 m long; and the wax storage tank for the small diameter column was 0.3 m in diameter and 0.46 m long. The slurry inlet systems for the large and small diameter columns are shown in Figures 2.2 and 2.3, respectively. Once the solid wax was melted ($\approx$ 150 °C), the stirrer was switched on to improve the heating process. For experiments conducted with solids, the solids were added to the storage tank once the wax was at the desired temperature (220 °C for batch experiments and 265 °C for continuous experiments). The column was heated to the desired operating temperature (265 °C) before the slurry was introduced. The column temperature was controlled using two temperature controllers, one for the bottom half of the column and one for the top half of the column. For all experiments, batch and continuous, the wax was transported to the column using a slight nitrogen overpressure in the storage tank. For the continuous mode experiments, the pump (Pulsafeeder, Model G12 – 0.05 m ID column; Tuthill Corporation, Model 3A – 0.21 m ID column) was not switched on

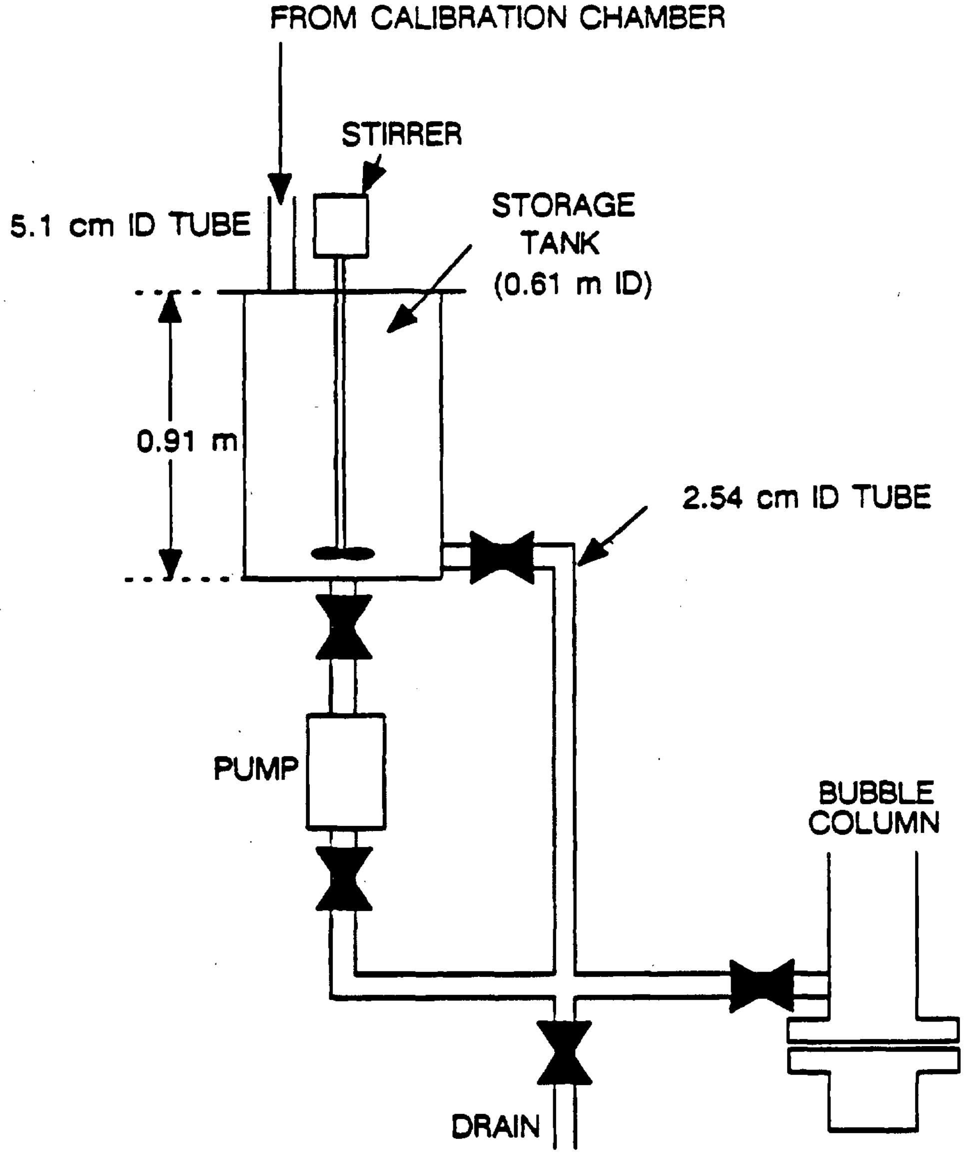

Figure 2.2. Schematic representation of the slurry inlet system for the large diameter stainless steel column.

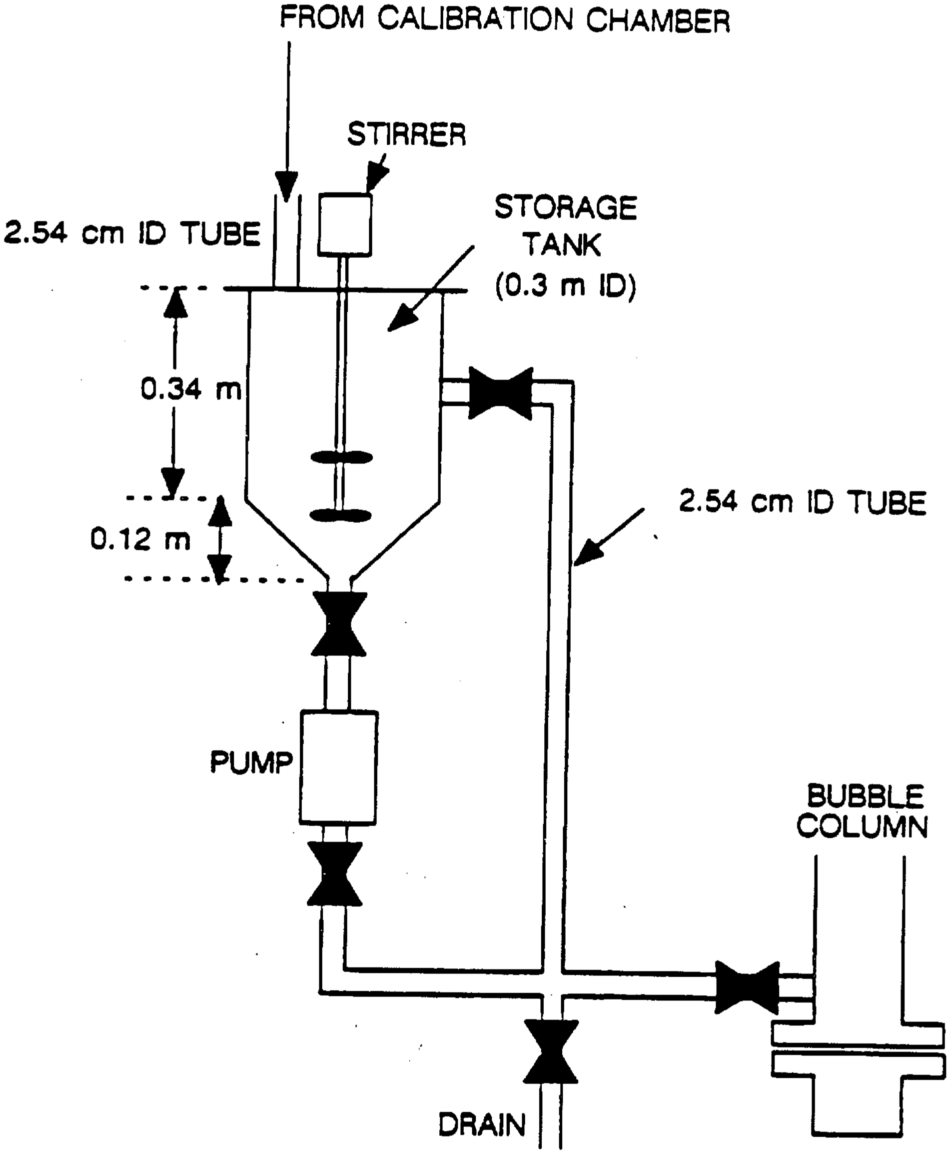

Figure 2.3. Schematic representation of the slurry inlet system for the small diameter stainless steel column.

until the column was at least half filled with wax. This was done to prevent clogging of the pump by solids which might have settled in the storage tank. Throughout the preheating period and during the transportation of wax to the column, nitrogen flowed through the column. Once the wax was in the column, the temperatures of the various units were allowed to stabilize before the actual run was started. For experiments in the batch mode of operation, only the column was maintained at the desired operating temperature. The exit lines and expansion unit were maintained at a temperature of approximately 200 °C. The hot gas leaves the separator and passes through the scrubber which is filled with Varsol (mineral spirits), before it is vented to the atmosphere. The scrubber is used to recover components of the wax that evaporate from the column and is maintained at approximately 70 °C. The lines connected to the pressure transducers and slurry sampling valves were maintained at 200 °C. For experiments in the continuous mode of operation, all lines and vessels carrying the slurry were maintained at the operating temperature. The remaining temperatures were the same as those used for batch experiments. All temperatures were monitored regularly, every half hour during the preheat period and every hour during the experiment.

Once the column reached the desired temperature, the experiment was initiated. Superficial gas velocities in the range 0.02 – 0.12 m/s were employed in all runs. A duration of at least one and a half hours was used for each velocity. Pressure measurements were made three times for every gas velocity (i.e. approximately every half hour), with the first measurement made one half hour after the gas velocity was changed. Slurry samples were withdrawn at the five different locations after the final pressure measurement. The gas flow rate was then changed to the next setting. For experiments conducted in the continuous mode of operation, the superficial slurry velocity was monitored using the calibration chamber. The calibration chamber for the large diameter column was a 0.46 m ID cylindrical tank with an internal volume of approximately

50000 cm^3, and for the small diameter column, the calibration chamber was a 0.23 m ID cylindrical tank with an internal volume of approximately 4000 cm^3. Figures 2.4 and 2.5 are schematic representations of the circulation loops associated with the large and small diameter columns, respectively. The desired slurry flow rate was set by varying the pump speed, and slurry flow rate checks were made prior to each pressure reading (i.e. three times per gas velocity).

For three–phase experiments, slurry samples were withdrawn from the storage tank at the beginning and end of each experiment; as well as, at the end of each gas velocity for experiments conducted in the continuous mode of operation. In order to determine the volume of slurry in the storage tank, a dipstick, similar to that used to determine the oil level in an automobile, was designed (see Figure 2.6). The dipstick assembly consisted of a casing (2.54 cm diameter tube), which was welded to the lid of the storage tank, and the dipstick (0.635 cm diameter shaft). The casing extended half way into the storage tank and had vent holes at the top to allow any gas which might be trapped in the casing to disengage.

Following the completion of a run, the slurry was withdrawn into the storage tank using a slight vacuum (the pump was switched off for runs conducted in the continuous mode of operation). After each run, solids and wax inventories were made to check for any losse losses, particularly losses in solids due to settling in the various lines and process vessels. Solids and wax inventories are discussed in Chapter IV.

Following the completion of a series of experiments, the bubble column apparatus was cleaned . Any slurry which may have remained in the system was collected and weighed, so that an overall mass balance (solids + wax) could be obtained (see Chapter IV).

Two–phase experiments were also conducted in two columns (0.05 m ID and 0.23 m ID by 3 m tall) made of borosilicate glass. A detailed description of these columns has

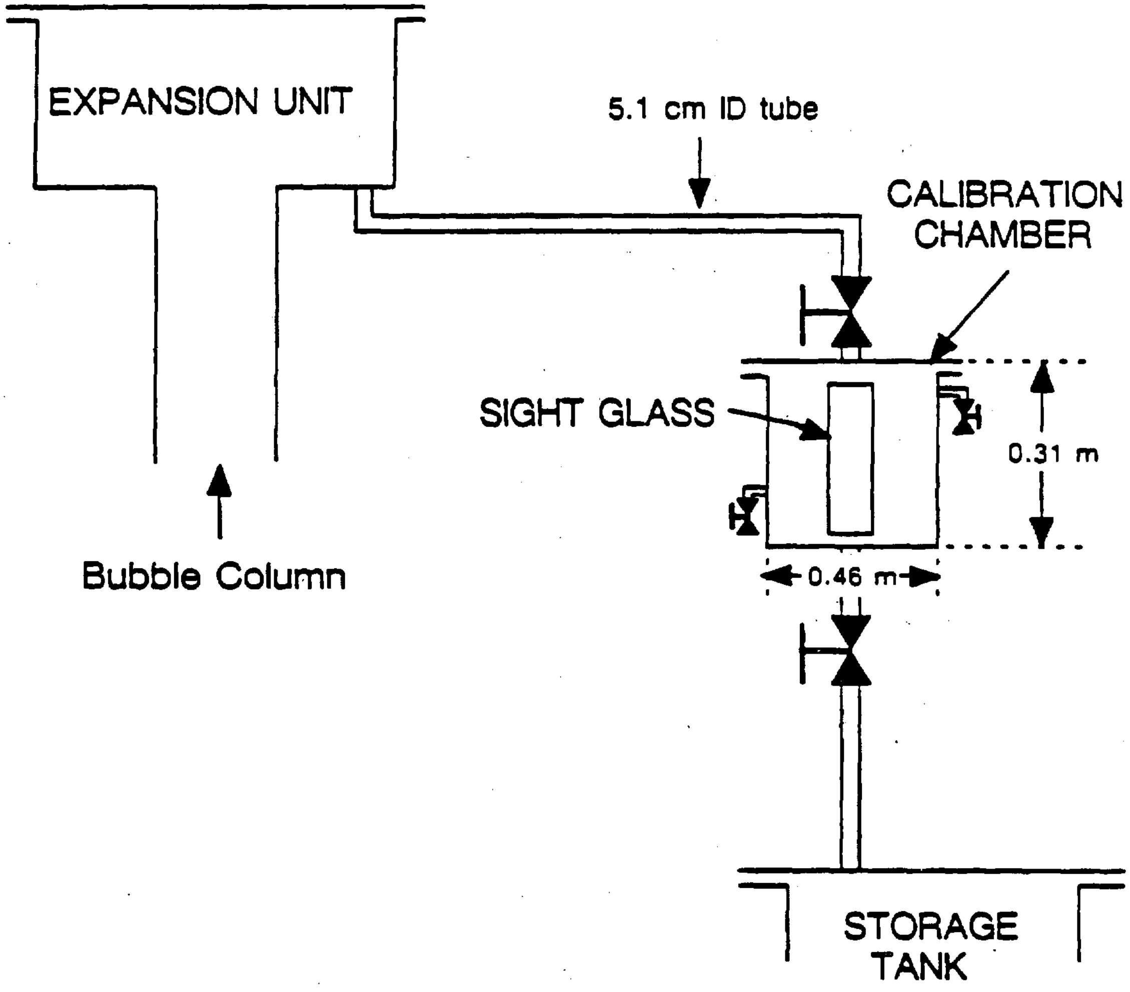

Figure 2.4. Schematic representation of the circulation loop for the large diameter stainless steel column.

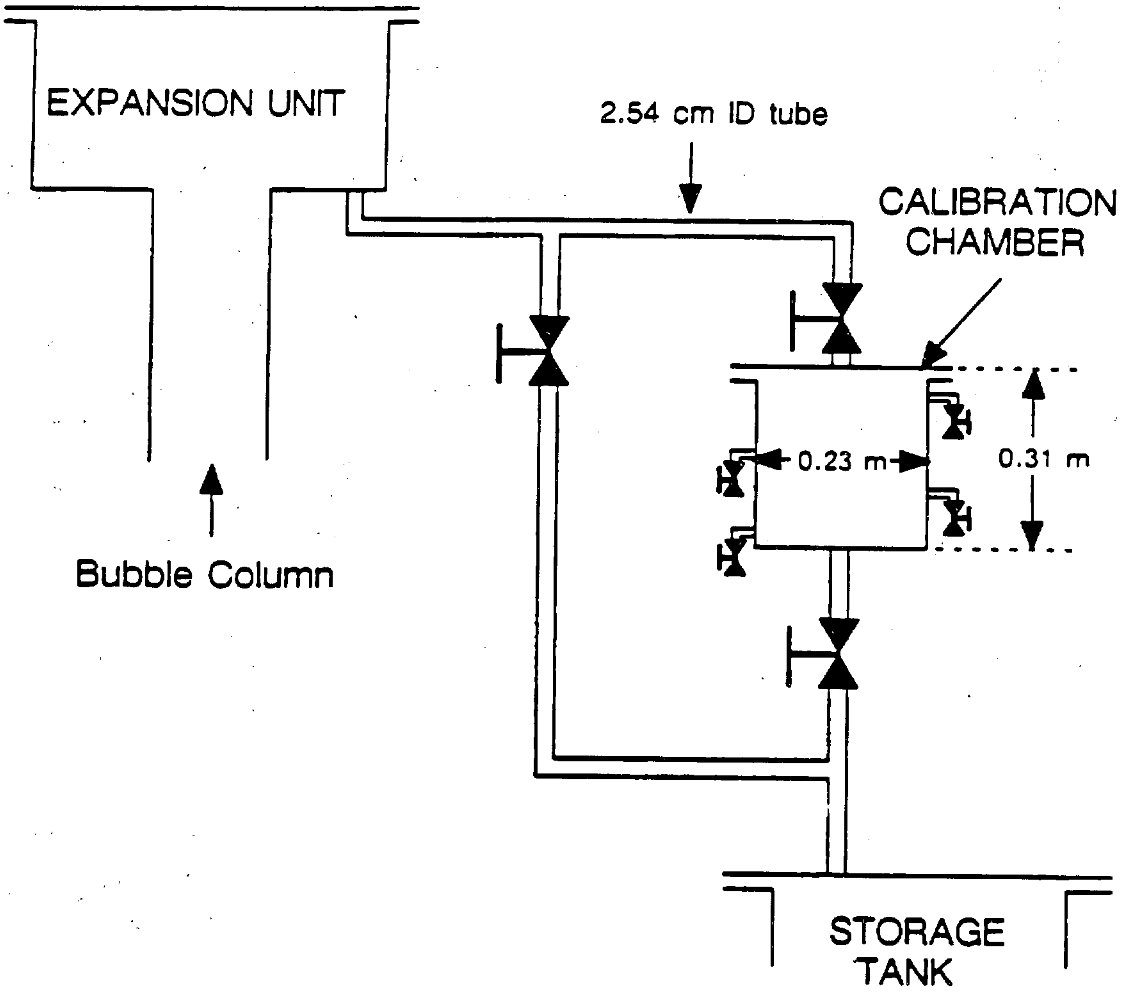

Figure 2.5. Schematic representation of the circulation loop for the small diameter stainless steel column.

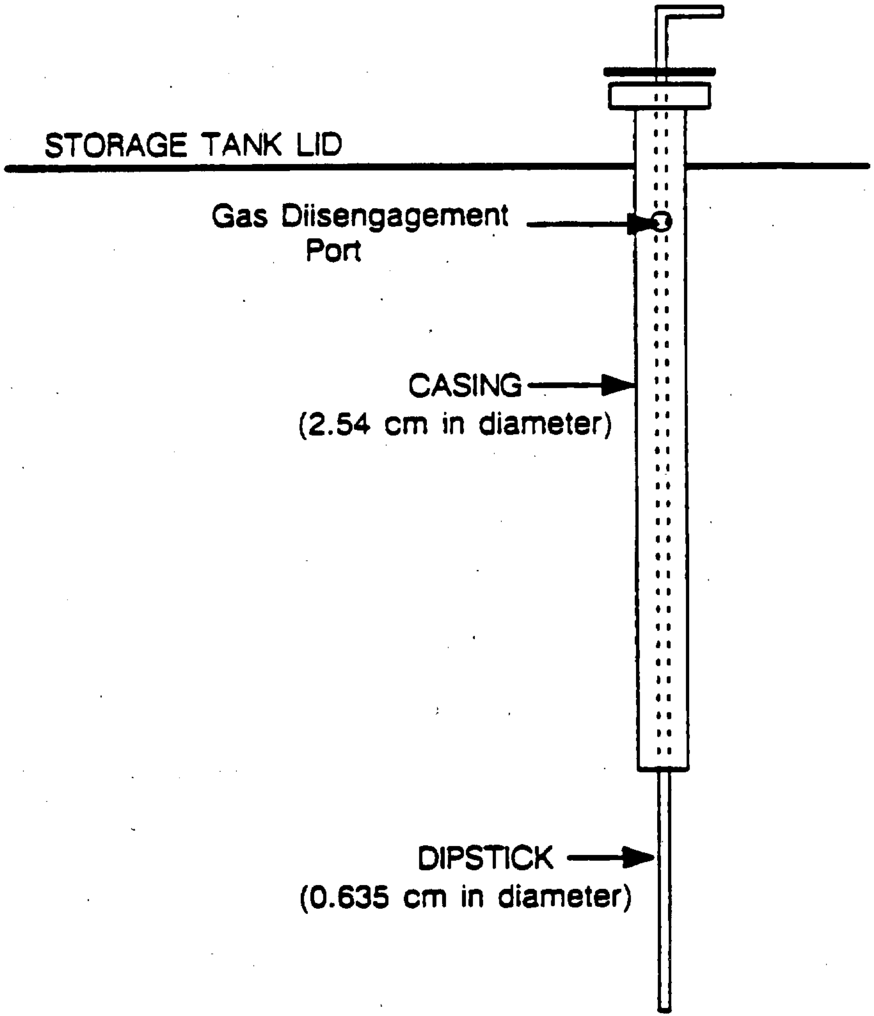

Figure 2.6. Schematic representation of the dipstick assembly.

been given elsewhere (Bukur et al., 1987a,b,c). All experiments in the glass columns were conducted in the batch mode of operation. The glass columns were used to obtain average gas holdups and bubble size distributions.

Experimental Conditions

The effects of operating conditions (slurry and gas superficial velocity), gas distributor design, column diameter, and solids concentration, type, and size were studied. Table 2.1 summarizes the experimental conditions employed in the stainless steel bubble columns. Experiments were conducted with SASOL's Arge reactor wax and FT–300 wax. SASOL reactor wax consists of high molecular weight products of the Fischer–Tropsch synthesis. FT–300 wax (also known as SH–105) is a hard paraffin wax obtained by hydrotreating and fractionation of reactor wax and it has an average molecular weight of 730 (Dura Commodities, New York).

Nitrogen was used as the gas for all experiments because it is inert, non–toxic, and inexpensive. Also, in earlier studies, it was found that the effect of gas type on the average gas holdup is small (Deckwer et al., 1980; Kuo, 1985). Superficial gas velocities in the range 0.02 – 0.12 m/s were employed in all experiments. With this range of gas velocities in the two columns, all important flow regimes were observed. A superficial gas velocity of 0.095 m/s was employed in the Rheinpreussen demonstration plant unit (Kolbel and Ralek, 1980), and thus, the higher velocities (0.08 – 0.12 m/s) chosen in this study are representative of gas velocities employed in large diameter reactors.

All of the experiments in the stainless steel bubble columns were conducted at a temperature of 265 °C, which is a typical temperature for the Fischer–Tropsch synthesis with precipitated iron catalysts. In our previous studies (Bukur et al., 1987a,b,c) some experiments were conducted in the small diameter column at other temperatures (160-280 °C) in order to study the effect of liquid viscosity on gas holdup.

Table 2.1. Bubble Column Dimensions and Experimental Conditions

COLUMN DIMENSIONS		
DIAMETER (m)	0.05	0.21
HEIGHT (m)	3.0	3.0
GAS DISTRIBUTOR	2 mm ORIFICE	PERFORATED PLATE BUBBLE CAPS
GAS	NITROGEN	NITROGEN
LIQUID	FT-300 and SASOL	FT-300 and SASOL
SOLIDS	IRON OXIDE (< 44 μm) SILICA (< 44 μm)	IRON OXIDE (< 44 μm) SILICA (< 44 μm)
VARIABLES		
PRESSURE (atm)	1	1
TEMPERATURE (^{o}C)	265	265
SUPERFICIAL GAS VELOCITY (m/s)	0.02 - 0.12	0.02 - 0.12
LIQUID UPFLOW VELOCITY (m/s)	0.0 - 0.02	0.0 - 0.02
SOLIDS CONCENTRATION (wt%)	0 - 30	0 - 30

A 2 mm single hole orifice plate distributor was used for experiments conducted in the 0.05 m ID column. Whereas, for experiments in the 0.21 m ID column, both a 19 x 2 mm perforated plate and bubble cap distributor were used (see Figures 2.7 and 2.8, respectively). Perforated plates and bubble caps are commonly used in slurry bubble columns.

Solids concentrations in the range 0 – 30 wt% were employed throughout this work. This range of concentrations encompasses the range of catalyst concentrations used in slurry bubble column reactors for Fischer–Tropsch synthesis. Iron oxide particles (0 – 5 μm and 20 – 44 μm) were used as the primary solid. Some experiments were also conducted with silica particles (0 – 5 μm and 20 – 44 μm) to study the effect of solid density on the hydrodynamics of the system. The two types of solids used, iron oxide and silica, simulate typical catalysts and supports, respectively, employed in Fischer–Tropsch synthesis.

Data Acquisition and Reduction Procedures for Gas Holdups and Solids Concentration Profiles

Average gas holdups, axial gas holdups, and axial solid concentration profiles were measured experimentally. Experiments in the glass column were limited to two–phase (i.e. liquid/gas systems) batch studies; whereas, experiments in the stainless steel columns were conducted using both two–phase and three–phase systems with and without slurry circulation.

Average Gas Holdup – Glass Columns

For experiments conducted in the glass columns, only average (or overall) gas holdups were measured. The average gas holdup, which is the volume fraction of gas in the suspended slurry, is calculated from visual observations of the expanded and

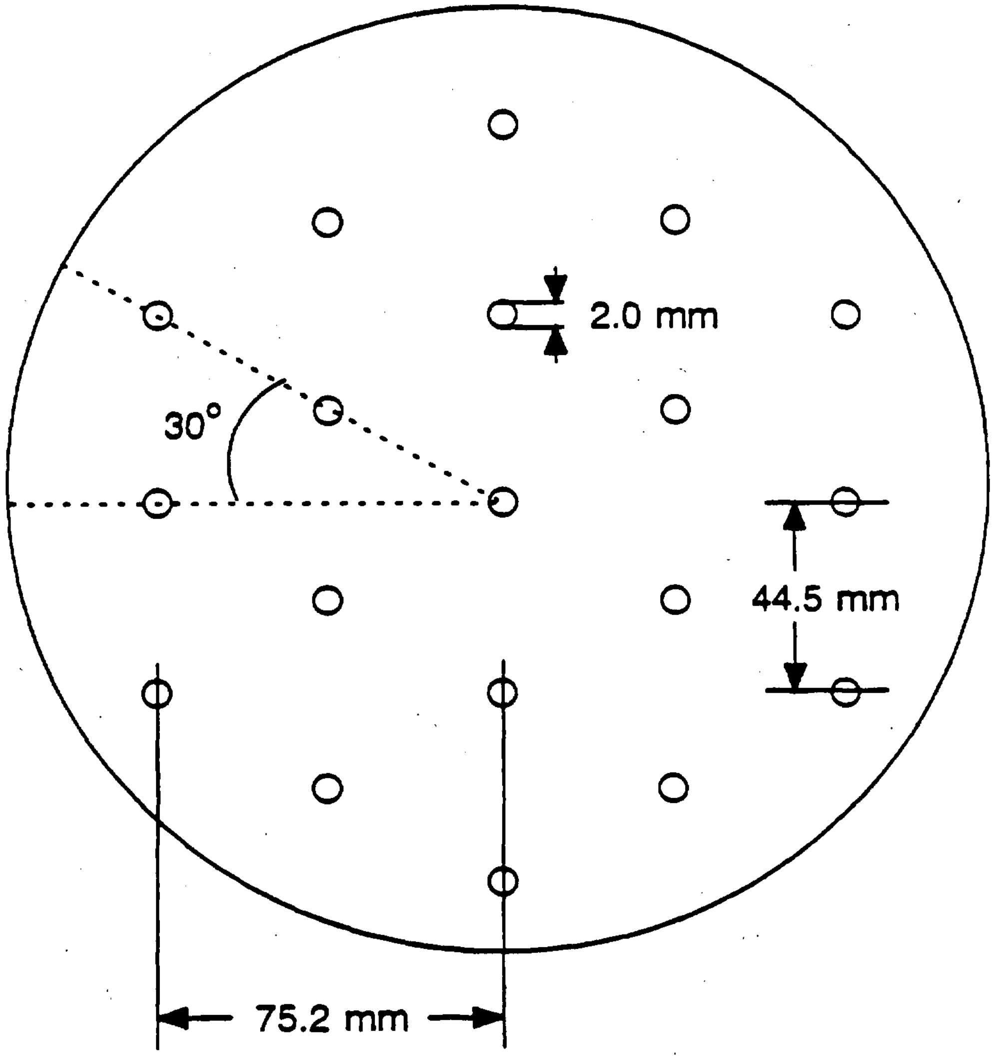

PLATE THICKNESS = 3.2 mm

Figure 2.7. Schematic representation of the perforated plate distributor.

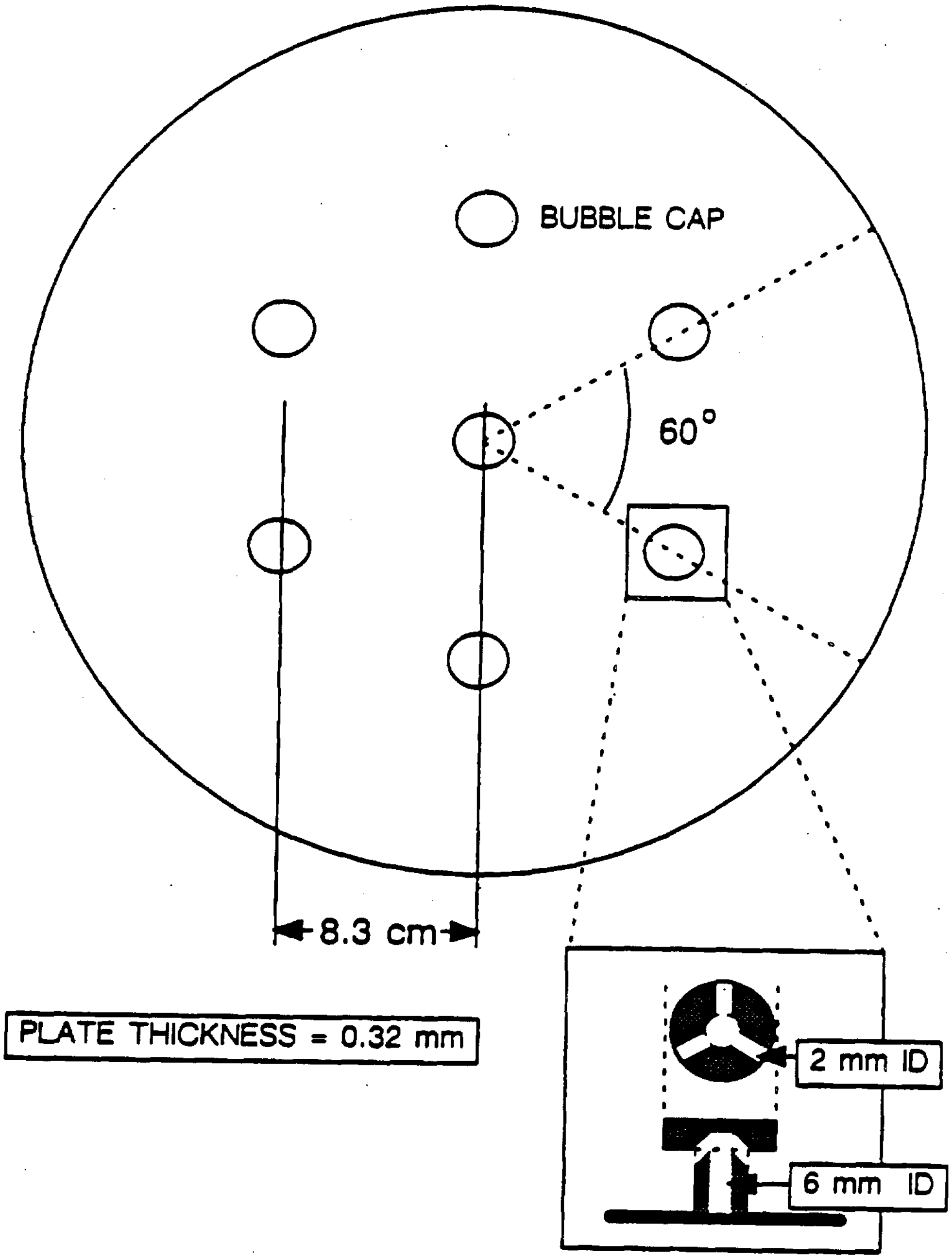

Figure 2.8. Schematic representation of the bubble cap distributor plate.

static liquid heights, i.e.

$$\epsilon_g = 1 - \frac{h_s}{h_{exp}} \tag{2.1}$$

where h_s is the static liquid height and h_{exp} is the expanded height of the slurry, and the quantity h_s/h_{exp} is the volume fraction of liquid in the gas/liquid dispersion. The expanded height was recorded three times per gas velocity at intervals of approximately 30 minutes. Once the expanded height was recorded three times, the gas flow was shut off and the static liquid height was recorded.

Phase Fractions – Stainless Steel Columns

In the stainless steel columns, axial gas holdups, axial solids concentrations, and average gas holdups were obtained. Axial pressure measurements and axial solids concentrations were used to calculate gas holdups (average and axial). Figure 2.9 is a schematic representation of the locations of the five pressure transducers and five slurry sampling ports.

Pressure Measurements

During experiments in the stainless steel columns, the pressure drop across the column and the weight fractions of solids were measured at various axial locations. A purgeless pressure transducer system was designed which prevents hot slurry from coming in contact with the DP cell (see Figure 2.10). The system consisted of a 0.635 cm diameter tube attached to the column wall and a 20 cm^3 chamber. When the column is filled with slurry, the nitrogen trapped in the chamber serves as a buffer between the hot slurry in the column and the low temperature DP cell. The chamber also serves as a trap for any slurry that flows into the 0.635 cm tube. A drain located at the bottom of the chamber is used to clean the trap between runs. A ball valve located in the 0.635 cm line serves to isolate the system from the column, in case the

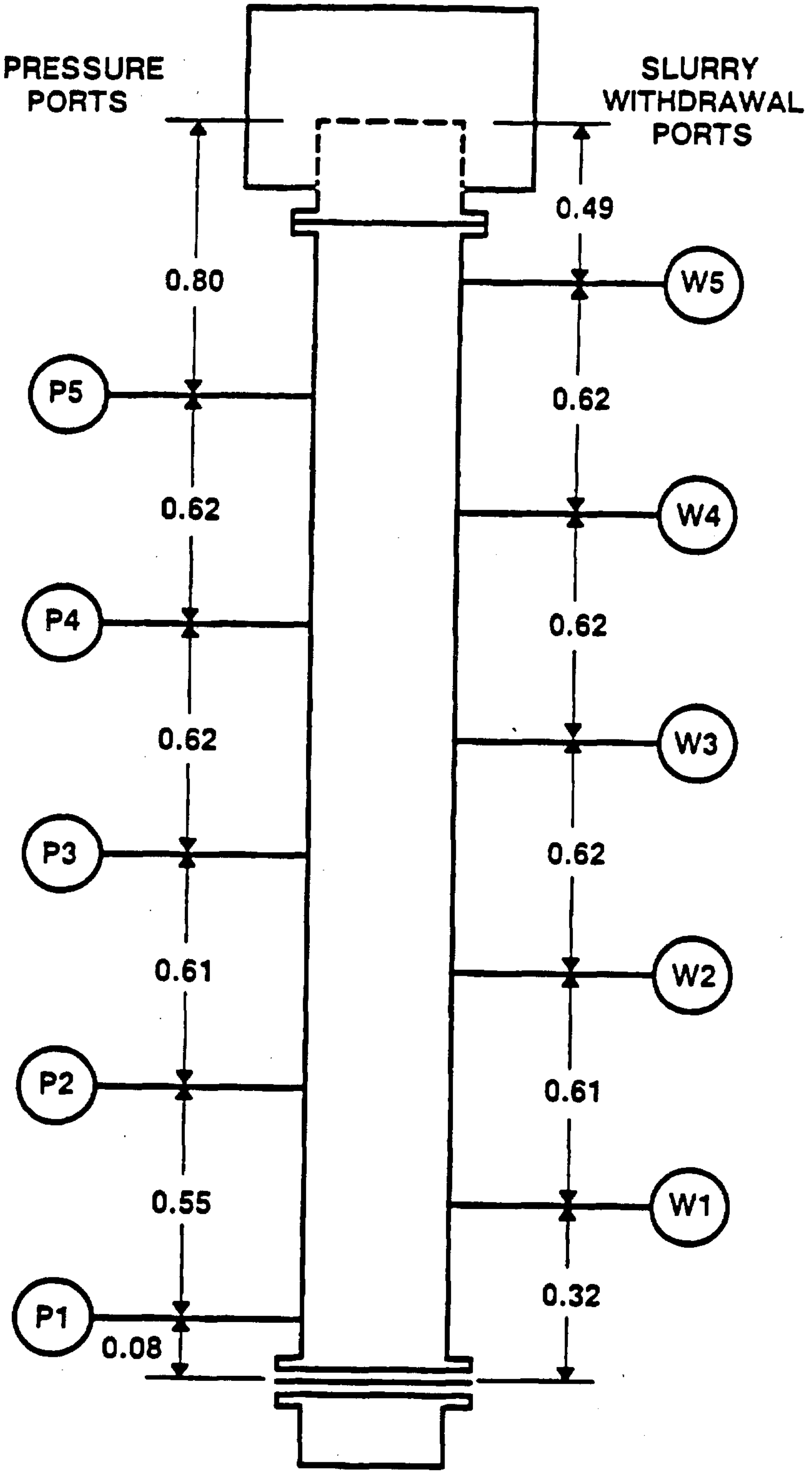

Figure 2.9. Schematic diagram of the pressure ports and slurry sampling ports locations (all dimensions in m).

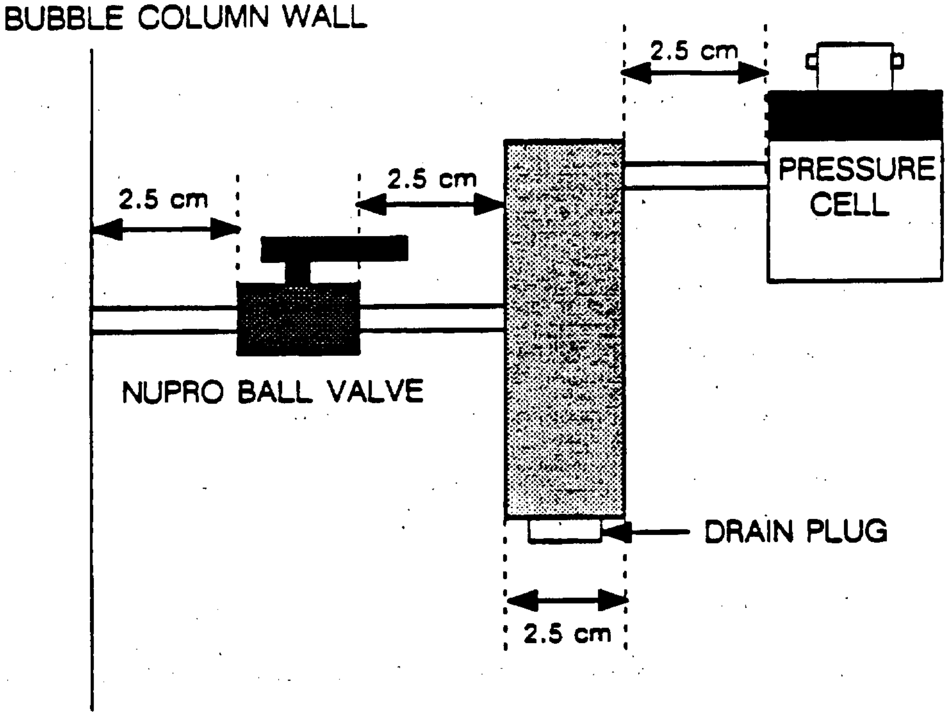

Figure 2.10. Schematic representation of the pressure transducer system.

trap has to be emptied during a run. The 0.635 cm line and chamber are heat traced and insulated to prevent solidification of slurry in this section.

The pressure transducers (Valydine Model DP–15) were connected to dual channel indicators (Valydine Model CD–223). The indicators have a digital display, as well as a 0 – 15 volt DC output. A data acquisition system which consisted of an A/D converter (Metrabyte Model DAS–16G), associated software, and a Zenith 286 personal computer was used to record the output voltage from the pressure transducer indicators. The pressure transducer indicators were adjusted so that the output voltage (proportional to pressure in inches of water) was scaled down by a factor of 10 before being sent to the data acquisition system. Thus, an output voltage of 1 corresponded to a height of approximately 10 inches of water. The calibration procedure for a single pressure transducer is outlined below. All pressure transducers were calibrated using the same procedure.

Before beginning each series of experiments, the pressure transducers were calibrated using tap water. The density of water was assumed to be 1000 kg/m^3. Initially, the output voltage from the pressure transducer indicator was set to zero. In order to calibrate a pressure transducer, the column was filled with water. The height of water above the transducer is the height of water in the column minus the height of the pressure transducer (both measured from the bottom of the column). The output voltage from the transducer indicator was forced to be 1/10th of the height of water (in inches) above the transducer by adjusting the span. The column was then drained and the zero was readjusted if necessary. Next, the column was filled with water again, and the output voltage was recorded. By making several measurements with different amounts of water in the column once the zero and span were set, a calibration curve of height of water (in inches) above the pressure transducer versus output voltage from the pressure transducer indicator was obtained. Figure 2.11 is a sample calibration curve. The form of the calibration equation for a given pressure transducer is:

$$\text{Pressure (inches of water)} = \text{slope} * (\text{output voltage}) + \text{intercept} \qquad (2.2)$$

For all pressure transducers, the slope of the calibration curve was in the range 9.9 to 10.1 and the intercept was in the range -0.6 to 0.6.

Data acquisition software was written which would display a "running" average of the pressure indicator output voltage. During the experiments, data was collected at a rate of 50 Hz for a period of 2 to 3 minutes. As previously described, measurements were made three times per gas velocity (i.e. approximately every 30 minutes), and the average of the three values (output voltage) was used to calculate the pressure, in inches of water, above a given pressure transducer using Eq. 2.2.

Solid Concentration Measurements

For three–phase systems, both pressure measurements and weight fractions of solids are needed to determine the phase holdups (i.e. gas, liquid, and solids holdup). The weight fraction of solids in the slurry samples withdrawn at the various axial locations (see Figure 2.9) was measured using the Archimedean principle (the apparent loss in weight of a solid body, when completely immersed in a liquid, equals the weight of the displaced fluid). The procedure used is outlined below.

The slurry withdrawn into the sampling cylinder is allowed to cool and solidify. The solid slurry plug is then removed for solids fraction determination. The sample is first weighed on a precision balance ($m_{s\ell}$). It is then suspended with a thin wire from a support structure placed on the balance and the combined weight of the support structure and sample recorded (m_1). The sample, while still suspended from the support structure, is then completely immersed in a beaker of acetone and the balance reading recorded (m_2). The three measured quantities, along with the known densities of

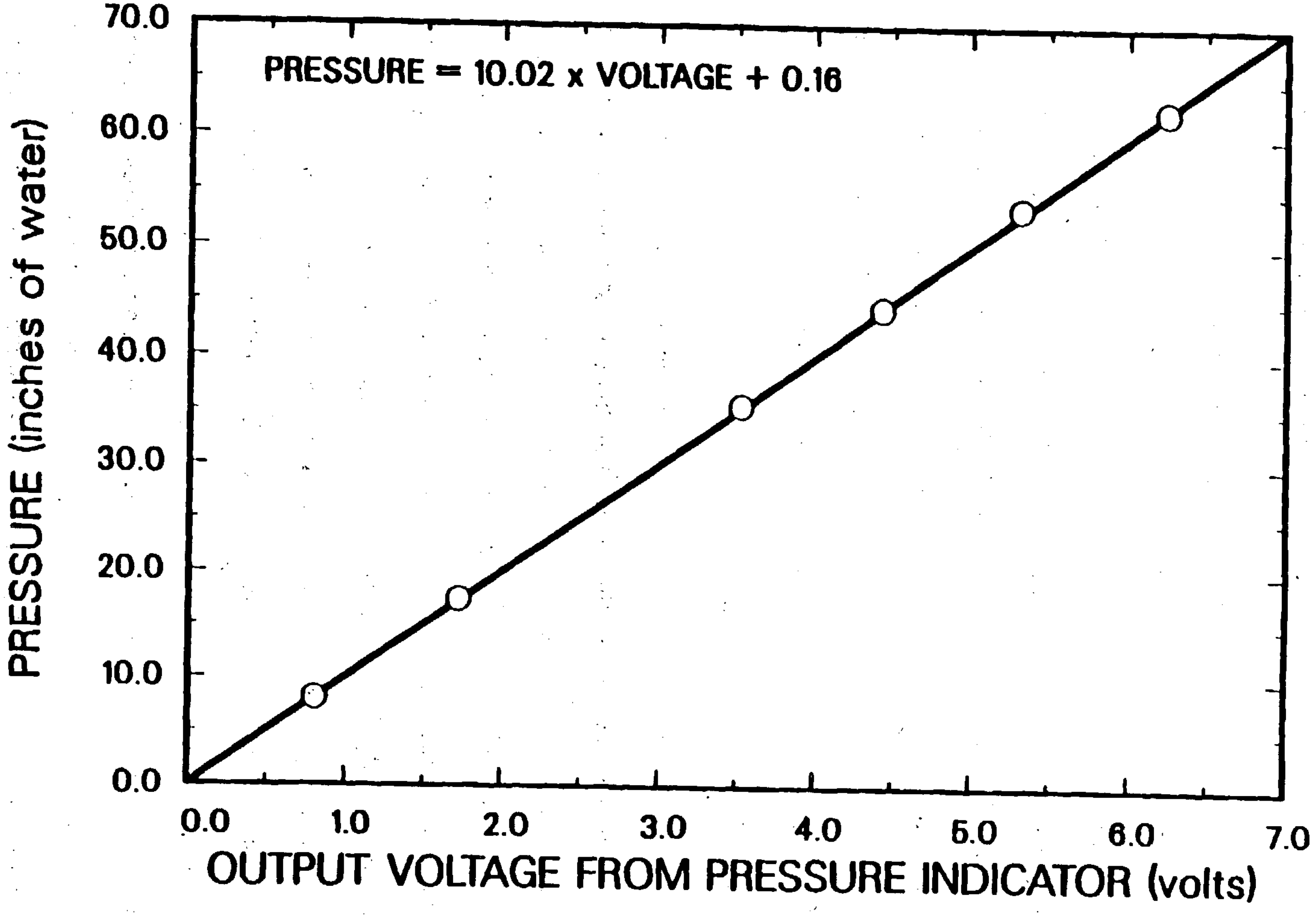

Figure 2.11. Typical pressure transducer calibration curve.

solidified wax (ρ_w), solids (ρ_s), and acetone (ρ_{acet}), were used to determine the weight fraction of solids (ω_s) in the slurry sample. By the definition of the Archimedean principle,

$$W_{acet} = m_1 - m_2 \tag{2.3}$$

where W_{acet} is the weight of acetone displaced. The volume of acetone displaced, which is the same as the volume of the slurry sample, is given by

$$V_{s\ell} = \frac{W_{acet}}{\rho_{acet}} \tag{2.4}$$

By substituting Eq. 2.3 into Eq. 2.4 and dividing the weight of the slurry sample ($m_{s\ell}$) by the volume of the sample ($V_{s\ell}$), the following expression is obtained for the density of the slurry sample

$$\rho_{s\ell} = \frac{m_{s\ell}\rho_{acet}}{m_1 - m_2} \tag{2.5}$$

The density of the sample may be expressed in terms of ω_s as follows

$$\rho_{s\ell} = \frac{1}{\frac{\omega_s}{\rho_s} + \frac{(1-\omega_s)}{\rho_w}} \tag{2.6}$$

Equating Eqs. 2.5 and 2.6 to eliminate $\rho_{s\ell}$, and rarranging the terms, yields the following expression for ω_s

$$\omega_s = \frac{\rho_s - (\frac{\rho_w \rho_s}{\rho_{acet}})(\frac{m_1 - m_2}{m_{s\ell}})}{\rho_s - \rho_w} \tag{2.7}$$

The density of solidified wax was determined experimentally. The density of fresh FT–300 wax is 950 kg/m^3, and the density of fresh SASOL wax is 930.5 kg/m^3. Acetone was selected as the liquid medium for this procedure because it has a lower density than wax (ρ_{acet} = 792 kg/m^3), it evaporates quickly from the sample surface, and the solubility of wax in acetone at room temperature is negligible. The procedure was tested using both FT–300 and SASOL wax containing known quantities of solids. Samples containing 3, 7, 10, 15, 20, 25, 30, and 35 wt.% solids in wax were prepared. Samples with 0–5 μm iron oxide, 20–44 μm iron oxide and 0–5 μm silica were used with FT–300, and samples with 0–5 μm iron oxide and 20–44 μm silica were used with SASOL wax. Solid densities of 5100 kg/m^3 for iron oxide and 2650 kg/m^3 for silica were employed. Tables 2.2a, 2.2b, and 2.2c show results obtained with slurries of 0 – 5 μm iron oxide particles, 20 – 44 μm iron oxide particles, and 0 – 5 μm silica particles in FT–300 wax, respectively. For these samples, the relative error between the calculated (Eq. 2.7) and the actual weight fraction of solids was highest for the sample containing 3 wt% 0 – 5μm silica particles (6.5 %). For all other solids concentrations, the relative error between the actual and calculated weight fractions was less than 2.2 %. Tables 2.3a and 2.3b show results from measurements with 0 – 5 μm iron oxide and 20 – 44μm silica particles in SASOL wax, respectively. For all samples analyzed, the calculated weight fractions of solids were within 1.02 % (relative) of the actual weight fraction of solids.

Sensitivity analysis of Eq. 2.7 revealed that the results were very sensitive to small variations in the density of solidified wax. A variation of only 1 % in the density of wax caused a 12 % change in ω_s; whereas, a 5 % change in the density of solids caused only a 2 % change in ω_s for slurries containing iron oxide, and a 4 % change in ω_s for slurries containing silica. Because of the high sensitivity to wax density, we determined the density of fresh wax and used wax. For FT–300, the density of fresh wax and used wax (100 hours on stream) was the same. However, the density of SASOL wax varied with time on stream. The density of fresh SASOL wax was 930.5 kg/m^3 and the density of used SASOL wax (72 hours on stream) was 941.2 kg/m^3. There was less than 0.07 % difference in the density of SASOL wax between 72 hours on stream and 144 hours on stream. The change in density between fresh and used SASOL wax was probably caused by changes in the composition of SASOL wax with time on stream. SASOL wax contains a significantly higher concentration of lower molecular weight components

Table 2.2a. Results from Archimedean Procedure (0 – 5 μm iron oxide in FT–300)[a]

Nominal	Actual wt.%	Measured wt.%	% Error
3	2.94	2.96	0.74
7	6.61	6.60	-0.13
10	9.15	9.09	-0.61
15	13.13	12.92	-1.59
20	20.20	20.26	0.31
25	25.15	24.95	-0.78
30	30.25	30.04	-0.68
35	35.22	34.90	-0.91

[a] densities used: ρ_w = 0.9495 g/cc, ρ_s = 5.1 g/cc, ρ_{acet} = 0.792 g/cc

Table 2.2b. Results from Archimedean Procedure (20 – 44 μm iron oxide in FT–300)[b]

Nominal	Actual wt.%	Measured wt.%	% Error
3	3.09	2.89	-6.54
7	7.11	7.25	2.02
10	10.24	10.01	-2.21
15	15.13	15.17	0.26
20	20.00	20.02	0.10
25	25.13	25.25	0.49
30	30.33	30.43	0.32
35	35.03	35.07	0.11

[a] densities used: ρ_w = 0.9495 g/cc, ρ_s = 5.1 g/cc, ρ_{acet} = 0.792 g/cc

Table 2.2c. Results from Archimedean Procedure (0 – 5 μm silica in FT–300)[c]

Nominal	Actual wt.%	Measured wt.%	% Error
3	3.04	3.09	1.54
7	7.06	7.04	-0.39
10	10.04	10.07	0.27
15	15.10	15.07	-0.23
20	20.09	20.00	-0.44
25	25.09	24.93	-0.67
30	30.15	30.05	-0.32
35	35.05	34.87	-0.51

[a] densities used: ρ_w = 0.9495 g/cc, ρ_s = 2.65 g/cc, ρ_{acet} = 0.792 g/cc

Table 2.3a. Results from Archimedean Procedure (0 – 5 μm iron oxide in SASOL)[a]

Nominal	Actual wt.%	Measured wt.%	% Error
3	3.03	3.02	-0.34
7	6.78	6.74	-0.65
10	9.81	9.89	0.77
15	14.94	14.99	0.31
20	20.26	20.05	-1.02
25	24.63	24.45	-0.72
30	28.95	29.04	0.32
35	35.21	34.90	-0.89

[a] densities used: $\rho_w = 0.9305$ g/cc, $\rho_s = 5.1$ g/cc, $\rho_{acet} = 0.792$ g/cc

Table 2.3b. Results from Archimedean Procedure (20 – 44 μm silica in SASOL)[b]

Nominal	Actual wt.%	Measured wt.%	% Error
3	2.97	2.97	0.0
7	7.02	6.97	-0.71
10	9.94	10.00	0.06
15	15.03	14.91	-0.83
20	19.94	19.86	-0.41
25	24.81	24.91	0.39
30	29.88	29.93	0.18
35	35.12	35.08	-0.10

[a] densities used: $\rho_w = 0.9305$ g/cc, $\rho_s = 2.65$ g/cc, $\rho_{acet} = 0.792$ g/cc

than FT–300 wax. These low molecular weight components evaporate when the slurry is held at 265 °C for extended periods of time. As mentioned previously, slight errors (or changes) in wax density result in large errors of the estimated solids concentration. In order to compensate for changes in wax density (SASOL wax) with time on stream, the density of used wax (i.e. 941.2 kg/m^3) was used to calculate the solids concentration once the wax had been on stream for over 72 hours, and the density of fresh wax (930.5 kg/m^3) was used prior to that.

Holdup Calculations

The system constants used to determine the gas holdup, liquid holdup, and solid holdup include the densities of solids (ρ_s), liquid (ρ_ℓ), solidified wax (ρ_w) and acetone (ρ_{acet}), heights of the five pressure ports above the distributor (h_1 to h_5), and the distance between the distributor (bottom of the column) and the top of the column (h_t). The measured quantities include the meter readings (i.e. average output voltages, OV_1 to OV_5), the weights of solidified slurry samples ($m_{s\ell_{ij}}$), the weight of the slurry sample suspended in air ($m_{1_{ij}}$), and its weight when immersed in acetone (m_{2ij}). For simplicity, the measured quantities have been replaced with the primary derived quantities, i.e. the differential pressure across section i–j (ΔP_{ij}), and the average weight fraction of solids for the section i–j ($< \omega_s >_{ij}$).

The differential pressure is defined by

$$\Delta P_{ij} = P_i - P_j = (a_i OV_i + b_i) - (a_j OV_j + b_j) \qquad i = 1 \text{ to } 5 \text{ and } j = i + 1 \qquad (2.8)$$

where a_i and a_j are the slopes of the calibration curves relating the meter readings to pressure (in inches of water) for transducers at ports i and j, respectively, and b_i and b_j are the intercepts of the two curves (see Eq. 2.2). Note, j = 6 corresponds to the top of the column, and the pressure at the top of the column is assumed to be atmospheric pressure. The distance between pressure ports i and j is defined as

$$\Delta h_{ij} = h_j - h_i \qquad i = 1 \text{ to } 5 \text{ and } j = i + 1 \tag{2.9}$$

The average weight fraction of solids (see Eq. 2.7) between pressure ports i and j is given by

$$< \omega_s >_{ij} = \frac{\rho_s - \left(\frac{\rho_w \rho_s}{\rho_{acet}}\right)\left(\frac{m_{1_{ij}} - m_{2_{ij}}}{m_{s\ell_{ij}}}\right)}{\rho_s - \rho_w} \qquad i = 1 \text{ to } 5 \text{ and } j = i + 1 \tag{2.10}$$

The gas holdup in a three-phase (gas/liquid/solid) system may be expressed in terms of the slurry (liquid/solid) density, $\rho_{s\ell}$ and the density of the gas-liquid-solid dispersion, ρ_d (i.e. density of the expanded slurry) as,

$$\epsilon_g = \frac{\rho_{s\ell} - \rho_d}{\rho_{s\ell} - \rho_g} \approx 1 - \frac{\rho_d}{\rho_{s\ell}} \tag{2.11}$$

since the density of the gas, ρ_g is small in comparison to the density of the slurry at low pressures.

The density of the expanded slurry between any two pressure ports, i and j may be calculated from the measured pressure drop ΔP_{ij} and the known distance between the pressure taps, Δh_{ij},

$$s_{d_{ij}} = \frac{\Delta P_{ij}}{\Delta h_{ij}} \text{ and } \rho_{d_{ij}} = s_{d_{ij}} \rho_{water} \qquad i = 1 \text{ to } 5 \text{ and } j = i + 1 \tag{2.12}$$

where $s_{d_{ij}}$ is the specific gravity of the dispersion between pressure ports i and j. Substituting this expression into Eq. 2.11, yields:

$$< \epsilon_{g_{ij}} > = 1 - \frac{\Delta P_{ij}}{s_{s\ell_{ij}} \Delta h_{ij}} \tag{2.13}$$

where $s_{s\ell_{ij}}$ is the specific gravity of the slurry (liquid/solid) in the i-j section. The specific gravity of the slurry between pressure ports i and j can be calculated from the weight fraction of solids between pressure ports i and j, the density of the solid, and th density of the liquid using the following expression

$$s_{s\ell_{ij}} = \frac{1}{\rho_{water}} \frac{1}{\left[\frac{<\omega_s>_{ij}}{\rho_s} + \frac{1-<\omega_s>_{ij}}{\rho_\ell}\right]} \qquad i = 1 \text{ to } 5 \text{ and } j = i+1 \tag{2.14}$$

Substituting Eq. 2.14 into Eq. 2.13 yields

$$< \epsilon_g >_{ij} = 1 - \left(\frac{\Delta P_{ij}\rho_{water}}{\Delta h_{ij}}\right) \left[\frac{< \omega_s >_{ij}}{\rho_s} + \frac{1- < \omega_s >_{ij}}{\rho_\ell}\right] \qquad i = 1 \text{ to } 5,\ j = i+1 \tag{2.15}$$

The latter expression was used to calculate the gas holdup between pressure ports i and j.

The liquid holdup may be expressed in terms of ρ_ℓ, ρ_d, ρ_s, ϵ_g, and ϵ_s as follows:

$$\epsilon_\ell = \frac{\rho_d - \epsilon_g\rho_g - \epsilon_s\rho_s}{\rho_\ell} \tag{2.16}$$

where ϵ_s is the volume fraction of solids (i.e. solids holdup) in the dispersion. Assuming $\epsilon_g\rho_g$ is negligible, Eq. 2.16 may be rewritten as:

$$\epsilon_\ell = \frac{\rho_d - \epsilon_s\rho_s}{\rho_\ell} \tag{2.17}$$

The volume fraction of solids in the dispersion may be expressed in terms of the weight fraction of solid in the dispersion using

$$\epsilon_s = \frac{\omega_s\rho_d}{\rho_s} \tag{2.18}$$

Substituting Eq. 2.18 into Eq. 2.17 upon rearrangement yields the following expression for ϵ_ℓ

$$\epsilon_\ell = \frac{\rho_d(1 - \omega_s)}{\rho_\ell} \tag{2.19}$$

Thus the liquid holdup in the section i-j is given by

$$< \epsilon_{\ell_{ij}} > = \frac{\rho_{d_{ij}}(1- < \omega_{s_{ij}} >)}{\rho_\ell} \qquad i = 1 \text{ to } 5 \text{ and } j = i+1 \tag{2.20}$$

Substituting the expression for $\rho_{d_{ij}}$ (Eq. 2.12) into Eq. 2.20 the following expression is obtained for the average liquid holdup between pressure ports i and j

$$<\epsilon_\ell>_{ij}=\left(\frac{\Delta P_{ij}\rho_{water}}{\Delta h_{ij}}\right)\left(\frac{1-<\omega_s>_{ij}}{\rho_\ell}\right) \qquad i=1 \text{ to } 5,\ \ j=i+1 \qquad (2.21)$$

Since the sum of the volume fractions of gas, liquid and solids equals one, the volume fraction of solids in the dispersion between pressure ports i and j may be expressed as:

$$<\epsilon_{s_{ij}}>=1-<\epsilon_{\ell_{ij}}>-<\epsilon_{g_{ij}}> \qquad i=1 \text{ to } 5 \text{ and } j=i+1 \qquad (2.22)$$

Substituting Eqs. 2.15 and 2.21 into Eq. 2.22 yields the following expression for the average volume fraction of solids in the i–j section

$$<\epsilon_s>_{ij}=\left(\frac{\Delta P_{ij}\rho_{water}}{\Delta h_{ij}}\right)\left(\frac{<\omega_s>_{ij}}{\rho_s}\right) \qquad i=1 \text{ to } 5,\ \ j=i+1 \qquad (2.23)$$

Equations 2.15, 2.21, and 2.23 were used to estimate holdups of the three phases in the section between any two adjacent pressure ports; however, for the equations to be valid the entire section must be filled with the dispersion. For runs conducted in the continuous mode of operation, all five sections are always full, since the dispersion fills the entire column. For runs conducted in the batch mode of operation, Eqs. 2.15, 2.21, and 2.23 may be used for those sections that are full, i.e. all sections below the top most non-zero pressure port. The section just above this pressure port (say section n) is only partially full, therefore the height of the dispersion in this section (Δh_n) is not known. However, the differential pressure for this section (ΔP_n) is known. If a slurry sample was not withdrawn from this section, $<\omega_s>_n$ would also be an unknown quantity. For such cases, the gas holdup and if necessary, the weight fraction of solids in this section are either estimated by extrapolation of the $<\epsilon_g>$ and $<\omega_s>$ profiles or they are assumed to have the same values as in the section below (i.e. $<\epsilon_{g_{n,n+1}}>=<\epsilon_{g_{n-1,n}}>$

and $< \omega_{s_{n,n+1}} >=< \omega_{s_{n-1,n}} >$). The height of the dispersion in this section is then calculated using (see Eq. 2.15)

$$\Delta h_n = \left(\frac{\Delta P_n \rho_{water}}{1- < \epsilon_g >_n}\right) \left[\frac{< \omega_s >_n}{\rho_s} + \frac{1- < \omega_s >_n}{\rho_\ell}\right] \tag{2.24}$$

Equations 2.21 and 2.23 can then be used to estimate the liquid and solids holdups for this section. For all runs, no measurements are made between the distributor and pressure port 1 (see Figure 2.9). It is assumed that the volume fractions of the three phases in this section are the same as those in the section above (i.e. section 1-2).

The average gas holdup for the entire dispersion can be obtained using a weighted (volume) average of the gas holdups in the individual sections and is expressed as

$$< \epsilon_g >= \sum_{i=1}^{n} f_{d_{ij}} < \epsilon_{g_{ij}} >, \qquad j = i+1 \tag{2.25}$$

where $f_{d_{ij}}$ is the volume fraction of the dispersion between pressure ports i and j and is given by

$$f_{d_{ij}} = \frac{\Delta h_{ij}}{\sum_i \Delta h_{ij}} \qquad i = 1 \text{ to } 5 \text{ and } j = i+1 \tag{2.26}$$

Substituting Eqs. 2.15 and 2.26 into Eq. 2.25 yields

$$< \epsilon_g >= 1 - \frac{\sum_{i=0}^{n} \left(\Delta P_{ij} \rho_{water}\right) \left[\frac{< \omega_s >_{ij}}{\rho_s} + \frac{1- < \omega_s >_{ij}}{\rho_\ell}\right]}{\sum_{i=0}^{n} \Delta h_{ij}} \qquad j = i+1 \tag{2.27}$$

where $\Delta P_{01} = \Delta P_{12} \Delta h_{01} / \Delta h_{12}$, and $< \omega_s >_{01} = < \omega_s >_{12}$. For the continuous mode of operation n = 5; whereas, for the batch mode of operation, n is dependent on the location of the top of the dispersion.

For two–phase experiments in the stainless steel column, the same procedure was used to calculate gas holdups and liquid holdups. However, the weight fraction of solids, ω_s, was set equal to 0.