

Therefore, a 0.375 mm diameter single-orifice distributor was installed in the small bubble-column reactor (Unit CT-225) prior to Run CT-225-113, which was described in Section IV.B.2.

As was seen, the conversion and methane + ethane selectivity were excellent during the run, illustrating the applicability of such distributors. The pressure drop across the orifice at constant superficial gas velocity was constant during the run and no slurry came through the opening.

The size and shape of the bubbles produced by the orifice is not known, but the gas holdup during the run was similar to holdups produced by a 20 micron SMP distributor using the same wax medium without catalyst.

This is in contrast to the results of our hot-flow bubble-column work, which to date have shown that orifice-type gas distributors generally give lower gas holdups than SMP's. It may be that the small diameter of this reactor causes higher holdups, as indicated by literature reviews. Nevertheless, it appears from this study that the overall F-T reaction may be more kinetically controlled than mass-transfer limited.

C.7. Gas Holdup Studies Using Run CT-256-5 Slurry in BSU Bubble-Column

Gas holdup in the bubble-column reactor of the two-stage BSU was measured by visual observation (at the 610 cm viewport), using nitrogen as the feed gas and end-of-run CT-256-5 slurry. The gas holdup was only 8.6 vol % at 199°C, 101 kPa, 12 wt % catalyst loading, and 4 cm/s superficial gas velocity. This is the lowest gas holdup ever observed at such a velocity in the BSU bubble-column, and is in agreement with results from scoping studies using the short hot-flow bubble-columns.

Increasing the pressure to 1.48 MPa while maintaining the same superficial gas velocity and temperature had no effect on the gas holdup. This is in agreement with results from scoping studies using our small bubble-column reactor reported earlier.

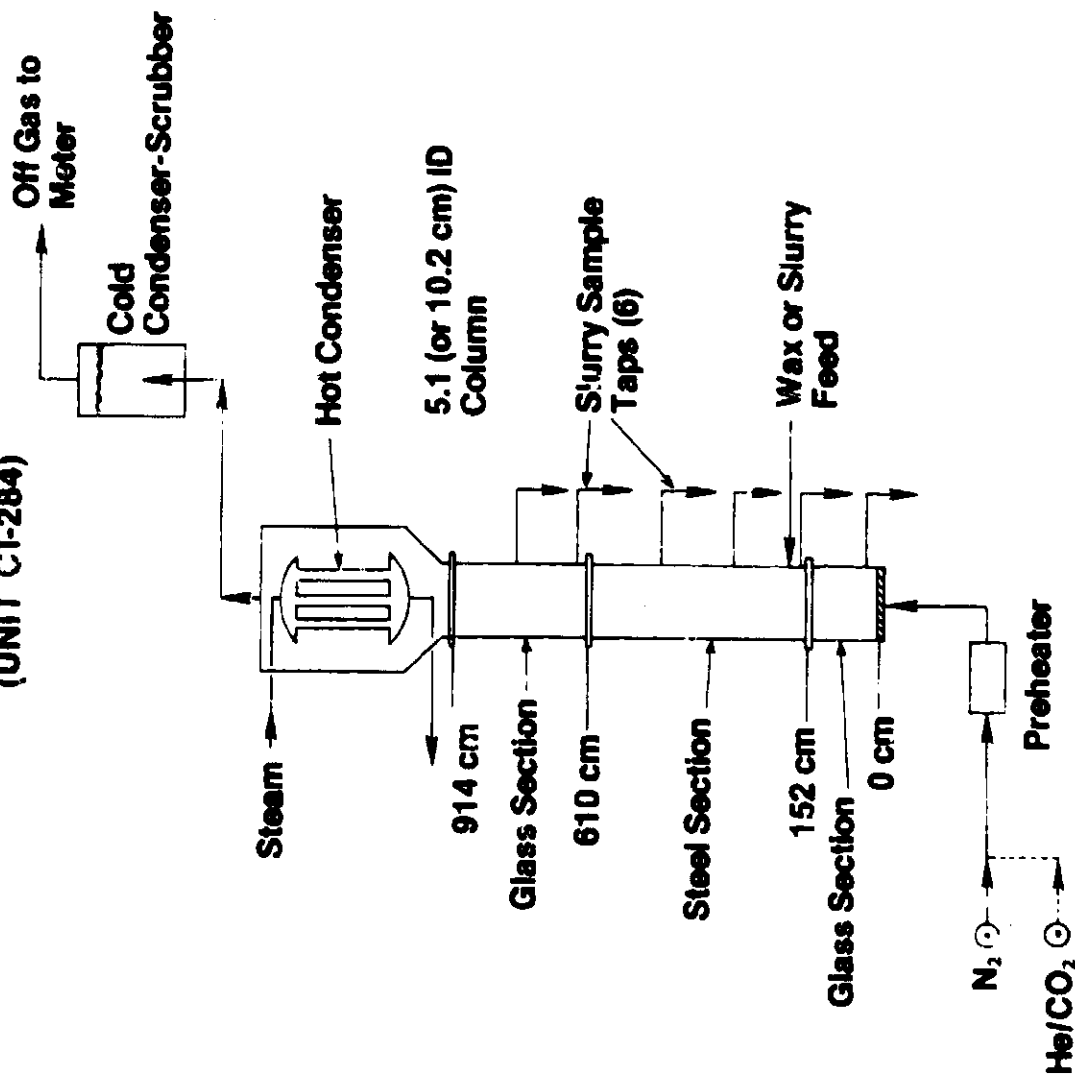
During operation of the BSU, DP-data along the F-T bubble-column were monitored. Part of the gas holdup data derived from them were reported together with the operational data of the BSU runs.

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

D.1. Simplified Flow Diagram and Design Basis

A simplified flow diagram of the two hot-flow non-reacting columns is given in Figure VII-20. The design basis

Figure VII-20
SIMPLIFIED FLOW DIAGRAM OF HOT-FLOW,
NON-REACTING BUBBLE COLUMN
 (UNIT CT-284)



given in Table VII-6, 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 VII-20. 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 VII-20, in-house N_2 or any other gas, such as He or CO_2 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 is mounted between two flanges.

Each column is divided into three sections connected by flanged 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 above 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 ten 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 flux 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. Similarly uniform heating can also be obtained by wrapping heating wires at a distance around the glass section. However, the first method was preferred over the second method because of easier installation and maintenance. 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 were 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 were mounted a short distance away from the column. The insulation around the glass section was fabricated such that small sections (~30 cm long) of the insulation can be swung away from the column for visual observation.

Table VII-6

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

Column Dimensions	<u>Design Range</u>	
	<u>Column 1</u>	<u>Column 2</u>
Inside Diameter, cm	5.1	10.2
Height, cm	914	
Operating Conditions		
Temperature, °C	121-260	
Pressure, kPa	0-207	
Gas Flow Rate, Nm ³ /hr	0-1.4	0-6.8
Other Operating Conditions		
Preheater		
Inlet Temperature, °C	Ambient	
Exit Temperature, °C	121-316	
Pressure, kPa	0-207	
Hot Condenser		
Inlet Temperature, °C	121-316	
Exit Temperature, °C	100-121	
Pressure, kPa	0-207	
Cold Condenser-Scrubber		
Inlet Temperature, °C	100-121	
Exit Temperature, °C	Ambient	
Pressure, kPa	0-207	

The slurry sample taps were provided at 152 cm intervals along the length of the column. At the same locations many pipe couplings were provided which were 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 was mounted inside the disengager. It was maintained at steam temperature, 100-121°C. The exiting gas was further cooled and scrubbed by bubbling it through a solvent which was maintained at chilled glycol temperature. The off-gas was finally metered by a dry gas meter.

Figure VII-21 is a partial Engineering Flow, and Piping and Instrumentation diagram showing the 10.2 cm ID hot-flow non-reacting column. As reported previously, both the 5.1 and 10.2 cm ID columns have identical features. Figure VII-21 gives only the main features of the column. Hence, it does not show the N₂ gas-feed system, the DP-cell arrangement, or the slurry sampling and loading systems. The feed N₂ is passed through the gas preheater E-14 and enters the column below the gas distributor. As shown in Figure VII-21, six slurry sample lines (marked "S") are provided at 152 cm intervals along the column. These sample lines connect to the sample-bombs, not shown in the diagram. The lines marked "DP" are the DP-cell N₂ purge lines used to prevent the plugging of the DP-legs by the wax or slurry. The DP-measurement allowed the estimation of the gas holdup along the column.

The upper glass section between the 610 and 914 cm levels permits the visual observation of the expanded slurry. When the liquid level is below the 610 cm level, it cannot be visually observed. Hence 21 thermocouples were installed in the steel section between the 442 and 602 cm levels at 7.6 cm intervals. These thermocouples allow us to measure the slurry level within 7.6 cm, since the thermocouple readings increase when the expanded slurry reaches there. Also shown in the diagram are the hot-condenser/disengager E-7 at the top of the column and the downstream cold-condenser/scrubber SG-4. The off-gas is bubbled through the scrubber to remove any heavy hydrocarbons stripped by the gas from the column. The scrubbed off-gas is finally metered by gas meter E-30. The control-valve CV-2 just before the gas meter regulates the column back-pressure.

Figur ENGINEERING FLC INSTRUMENTATION

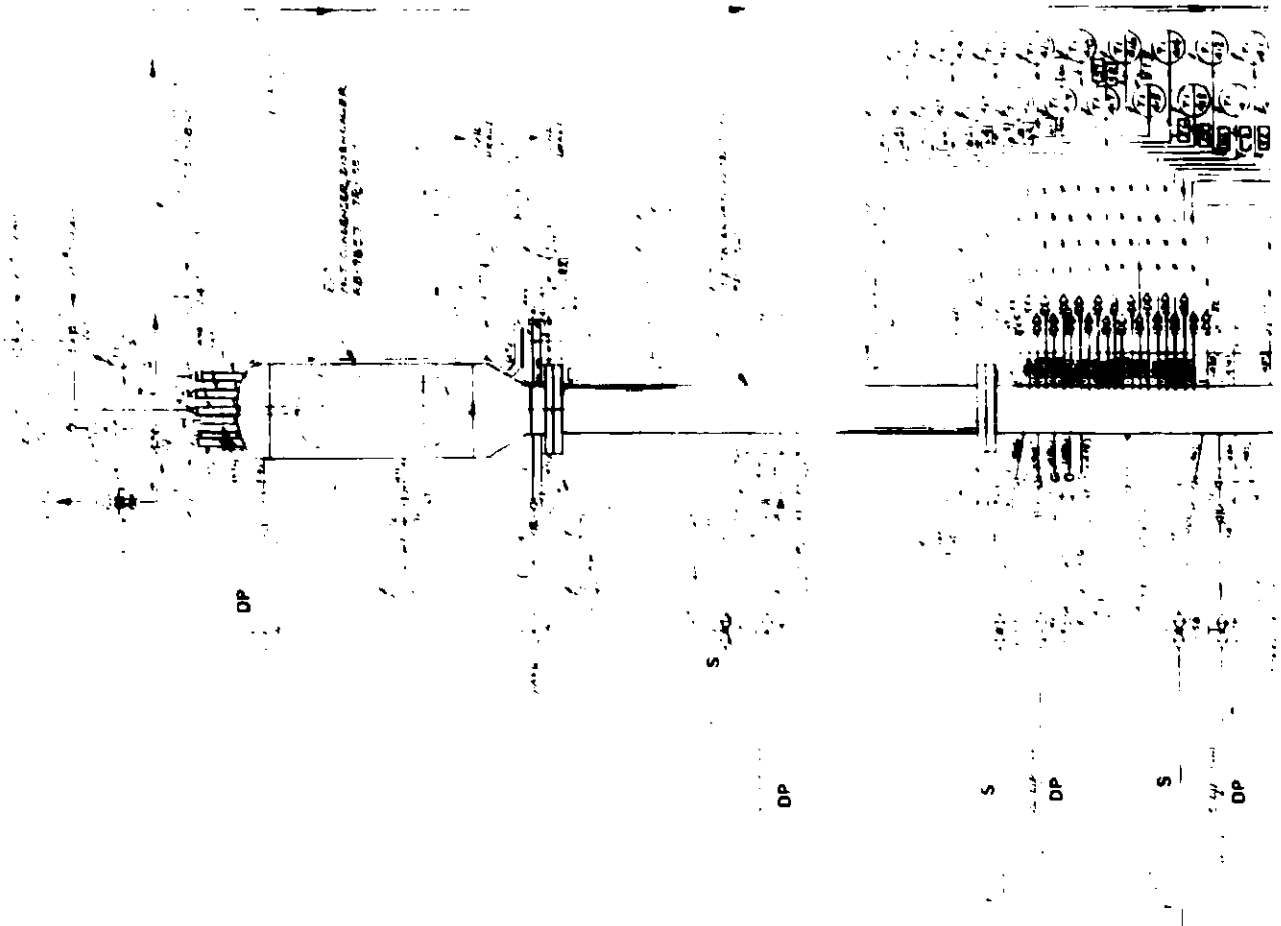
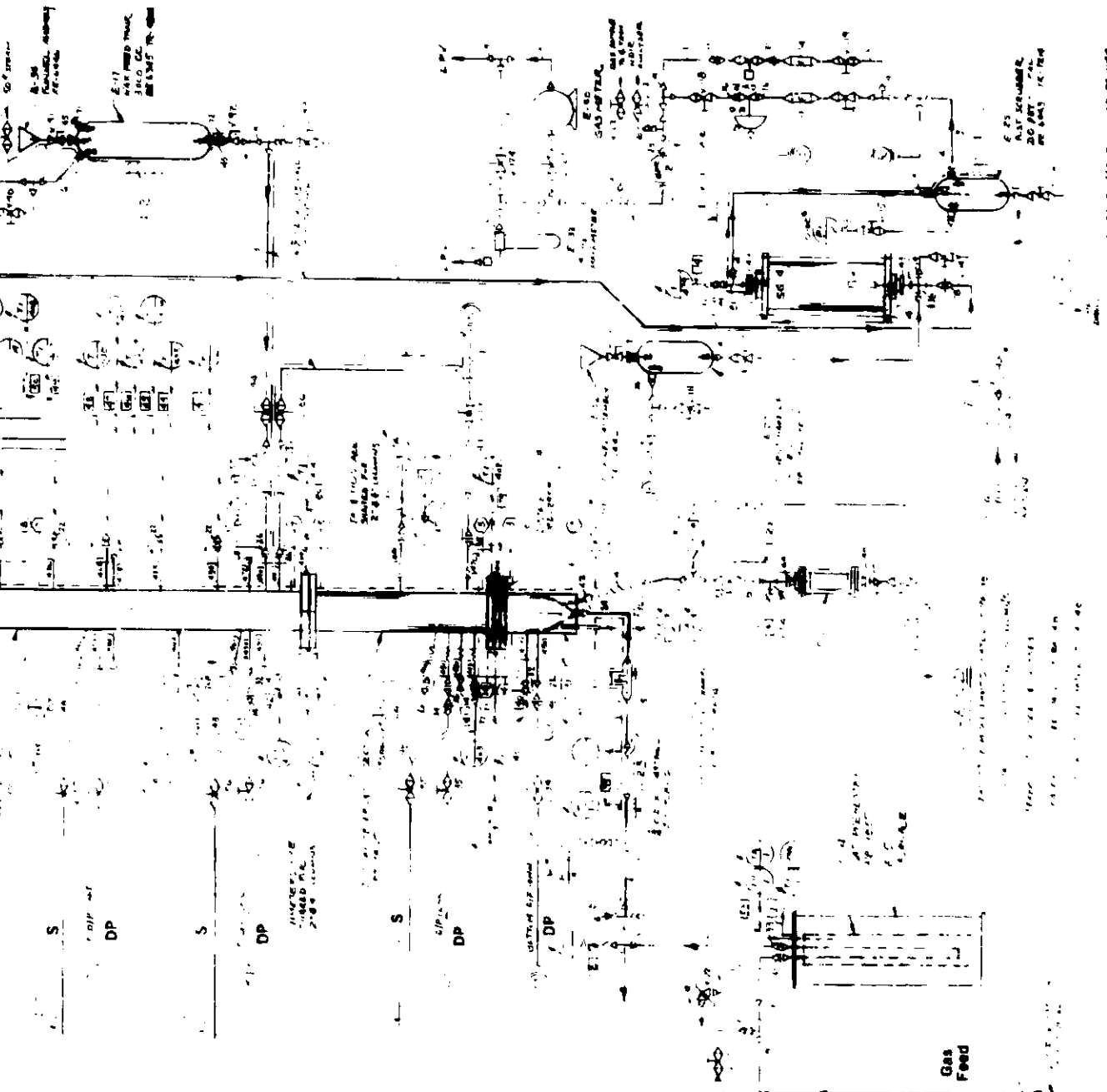


Figure VII-21

FLOW, AND PIPING AND INSTRUMENTATION DIAGRAM, UNIT CT-284



The construction of the two hot-flow columns (Unit CT-284) was completed in the first week of January, 1984. The shakedown of the unit, which was completed in one month, included:

- checking of all piping and valves
- calibration of equipment
- training of operators
- testing of equipment

All shakedown tasks were carried out smoothly as planned. The specific tasks carried out for both columns are listed below:

- Calibrated N₂-purge rotameters for DP-cell.
- Calibrated gas meters and feed N₂ mass-flow meters.
- Cleaned and flushed both columns, cold condenser scrubbers, all slurry sample lines and drop-out pots.
- Pressure-tested the entire system with 308 kPa N₂ at ambient temperature; repaired all leaks.
- Checked all steam tracings for proper operation.
- Tested all temperature indicators and controllers.
- Hot-pressure-testing the entire system (5.1 cm ID column only) with 308 kPa N₂ at 121°C.
- Tested the slurry loading tank operating using n-hexadecane at ambient temperature.

Later, during normal operation, the column was tested at the design temperature of 260°C with 287 kPa N₂.

The 5.1 cm ID column was put into operation first. A shakedown run was carried out using hexadecane as a liquid medium at ambient temperature (23°C) and 204°C. The static liquid height was 653-735 cm. Figure VII-22 gives the gas holdups as a function of average superficial gas velocity, u_g . The gas distributor used was a 20 micron SMP. The effect of temperature can be clearly seen: gas holdups increase substantially as the liquid temperature is increased. Since the viscosity of hexadecane decreases with increase in temperature, based on the

Figure VII-22
GAS HOLDUPS WITH HEXADECANE
IN TALL, HOT-FLOW COLUMN
(5.1 cm ID COLUMN)

