

A plausible explanation for the higher bed expansions observed in the PDU-10 runs is that the gas holdup in the PDU reactor was significantly higher because of smaller gas bubbles generated under high-pressure conditions. This is consistent with initial results<sup>(5)</sup> obtained in the 250 tons/day EDS Coal Liquefaction Pilot Plant which indicate that the high pressure conditions in the reactor increases the gas holdup significantly.

Another parameter that affects the size of the gas bubbles is the surface tension of the liquid. The lower surface tension of the PDU fluids tends to result in smaller bubbles and higher gas holdup. As a results, the bed expansion tends to be higher.

Based on a study reported in the literature<sup>(6)</sup> an expression has been developed to correlate the bed expansion data measured for extrudate catalyst. The relative bed expansion ( $\delta$ ), defined as the ratio of the volume increase of the catalyst bed to the volume of the settled bed, can be predicted using the following correlation:

$$\delta = 1390 (V_L/V_T)^{1.0} (\mu_L V_G / g C \sigma)^{0.21} \times 100\%$$

where

$$V_T = [4gD_p (\rho_p - \rho_L) / 3C_D \rho_L]^{1/2}$$

$$C_D = 24/Re_T \quad \text{for } 0.1 \leq Re_T$$

$$= 18.5/Re_T^{3/5} \quad \text{for } 0.1 < Re_T \leq 500$$

$$= 0.44 \quad \text{for } 500 Re_T \leq 2 \times 10^5$$

and

$$Re_T = D_p V_T \rho_L / \mu_L$$

(5) EDS Coal Liquefaction Process Development Phase V, Annual Tech. Prog. Report for July 1, 1980 - June 30, 1981.

(6) Dakshinamurty, P., V. Subramanian, and J. N. Rao, Ind. Eng. Chem. Proc. Des. Devel., Vol. 11, No. 2, 1972.

In the above equations,

- $\delta$  = relative bed expansion
- $V_L, V_G$  = superficial liquid (or slurry) velocity and gas velocity, respectively
- $V_T$  = terminal velocity of single particle in liquid (or slurry)
- $\mu_L$  = liquid (or slurry) viscosity
- $\sigma$  = liquid surface tension
- $g$  = gravitational acceleration
- $g_C$  = conversion factor, 32.2 lbm-ft/sec<sup>2</sup>-lbf
- $D_p$  = Heywood particle projected diameter
- $\rho_p, \rho_L$  = liquid-saturated particle density and liquid (or slurry) density, respectively
- $C_D$  = drag coefficient
- $Re_T$  = Reynolds number

The correlation takes into account both the liquid (or slurry) and catalyst properties. The effect of the flow regime for the particle motion in the liquid is included implicitly in the determination of the drag coefficient.

A computer program has been used to determine the constants in the correlation by the method of multiple linear regression. Based on the cold flow data obtained in this study and the PDU-10 high-pressure data, a correlation has been obtained which has a standard error of estimate of 0.22. A comparison of the predicted values with the observed values for the percent relative bed expansion is shown in Figure 17. Except for a small number of points, most of the data are within a  $\pm 25\%$  band.

We have also compared the predicted values from this correlation with the bed expansion data reported by Amoco<sup>(7)</sup> for the kerosene/N<sub>2</sub> system with or without fines. For the system without fines, almost all of the data points are within the same error band, as can be seen in Figure 17. For the system with fines, however, only a few points are outside the error band but the errors are still comparable to those of the present data with fines. One of the causes for the discrepancies is the uncertainty in the estimation of the apparent viscosity of the slurry. Since the bed expansion is approximately proportional to the 2/3 power of the viscosity, about 2/3 of the error in the estimation of the viscosity will contribute to the overall error in the predicted bed expansion.

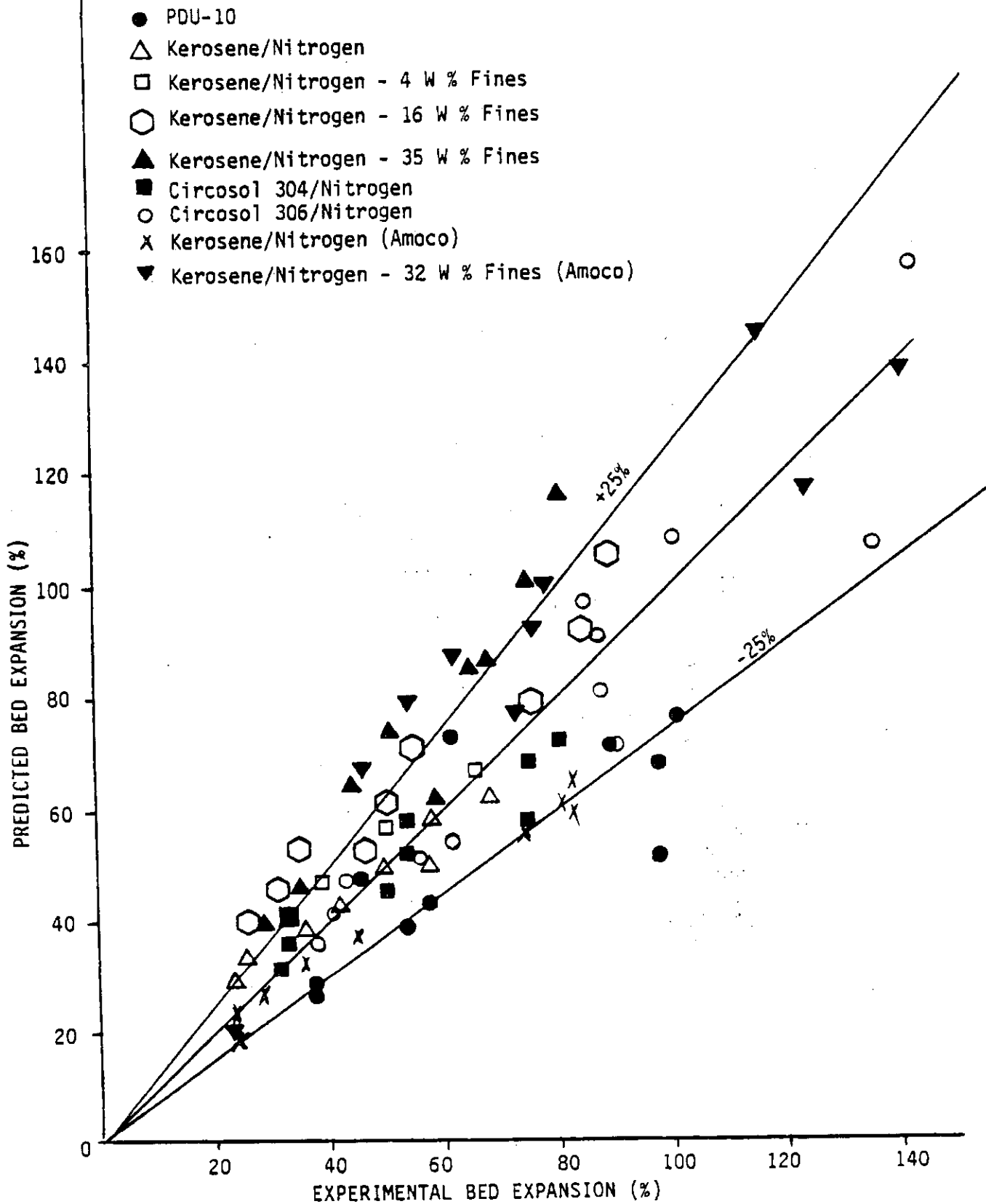
(7) "H-Coal Fluid Dynamics Final Technical Progress Report"  
Aug. 1, 1977 - Dec. 31, 1979, Report No. Amoco M80-21  
April 16, 1980

BED EXPANSION DATA  
FROM PDU-10

<u>Period</u>	<u>V<sub>L</sub> (ft/sec)</u>	<u>V<sub>G</sub> (ft/sec)</u>	<u>Bed Expansion (%)</u>
41B	0.09	0.07	97
42A	0.07	0.06	57
42B	0.04	0.07	37
43A	0.04	0.09	53
43B	0.04	0.04	37
44A	0.07	0.09	97
44B	0.07	0.05	45
45A	0.08	0.09	101
45B	0.09	0.04	61
46A	0.09	0.07	89

FIGURE 17

COMPARISON OF PREDICTED AND EXPERIMENTAL BED EXPANSIONS



## DEVELOPMENT OF IMPROVED INLET DISTRIBUTION SYSTEM

### BACKGROUND

The reactor inlet flow distribution system in the Pilot Plant involves in essence pumping the mixture of coal slurry feed, makeup and recycle hydrogen, and ebullating flow through a straight pipe entering the reactor plenum on the east side at an angle of  $46^\circ$  with the vertical. Except for impingement of the incoming jet on the internal recycle pipe, which disperses some flow to the two sides and bottom, the momentum of the inlet stream tends to carry the majority of the flow to the west side of the reactor. As the distributor grid cannot be expected to correct grossly uneven flow in the plenum, significant flow maldistribution is likely to exist in the catalyst bed above the grid.

Pilot Plant Run No. 6, a 45-day continuous operation on Illinois No. 6 coal which ended in December 1980, appeared to exhibit the above symptom. When the inside of the vessel was inspected coke and loosely agglomerated catalyst were found on the east side, where the inlet pipe enters the reactor plenum. The coke layer was about 18 inches thick and extended from several inches above the distributor grid to the thirteen-foot level. The coked material had high catalyst concentration. It is very likely that this coking problem was caused by insufficient flow in the inlet side of the vessel which is related to flow maldistribution in the reactor plenum. Reduced flow generally results in lower bed expansion, and in some cases, causes the bed to slump. In reactor systems involving exothermic reactions such as those occurring in the H-Coal reactor, reduced flow in a slumped bed increases the effective residence time, leading to a higher degree of completion of reactions locally, and as a result, this region becomes hotter, which in turn further increases the reaction rate. The local temperature can eventually become so high so as to initiate formation of coke.

In order to confirm flow maldistribution in the Pilot Plant reactor, a cold-flow simulation was carried out in the 5'-diameter model similar to the Pilot Plant reactor. The only major difference between the two is that the inlet pipe in the cold-flow model enters the plenum at an angle of  $15^\circ$  with the vertical and a solid deflection plate making a  $45^\circ$  angle with the vertical has been added to make the direction of the inlet flow similar in the two cases. It should be pointed out that the cold-flow model is expected to produce slightly more uniform flow than the Pilot Plant reactor because the deflection plate disperses some fluid to the north and south sides of the vessel.

As shown in Section III, the cold-flow study confirmed flow maldistribution in both the plenum and the region immediately above the distribution grid.

#### MODIFIED INLET DESIGN

To minimize flow maldistribution problems in the Pilot Plant reactor, we have recommended an immediate solution which consists of adding disc-doughnut type dispersion plates to the plenum inlet pipe. The configurations and dimensions of this device are detailed in Figure 18. The dispersion plates are to be made of erosion-resistant material to minimize erosion by the coal slurry.

The purpose of the added plates is to disperse the inlet flow across the reactor plenum and minimize "jetting" of the inlet stream. About 50% of the flow is to be deflected by the first plate and about 25% deflected by the middle plate. The slight angle between the plates and the plane normal to the pipe axis has been provided to account for the somewhat larger fluid volume on the west side of the inlet pipe.

To evaluate the performance of this type of inlet design, a cold-flow simulation was again been carried out using the 5' diameter model. The solid deflection plate was removed and a disc-doughnut type distributor as shown in Figure 19 was installed. For simplicity, the plates were placed normal to the

pipe axis. In this simulation, the flow in the cold-flow model is expected to be less uniform than in the Pilot Plant reactor since the uneven fluid volumes on the east and west sides of the inlet pipe is more severe in the cold-flow model. This being the case, a satisfactory performance of the inlet design in the cold-flow model would suggest an even better performance of that in the Pilot Plant reactor.

The results from a highly successful, 131-day operation (Run No. 8) at the Pilot Plant have become available. This run, also on Illinois coal, ended in December 1981 and post-run inspection revealed only a small amount of coke on the east wall above the grid, indicating that the modified plenum inlet design has greatly reduced, if not completely eliminated flow maldistribution in the reactor. This is consistent with the findings from the cold-flow simulation as presented in Section III.



IMPROVED INLET PLENUM WITH NEW DIFUSSER  
PILOT PLANT REACTOR

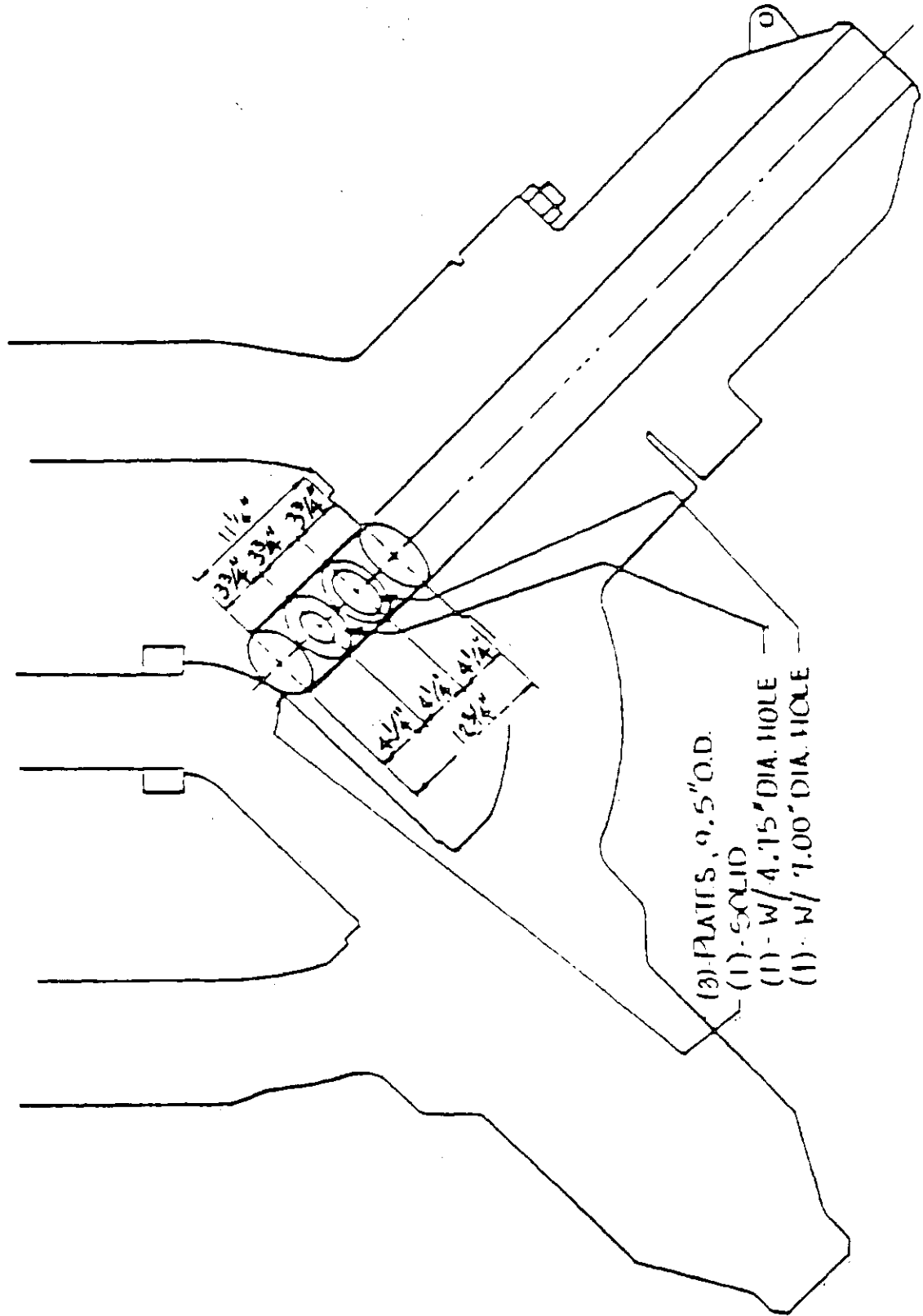
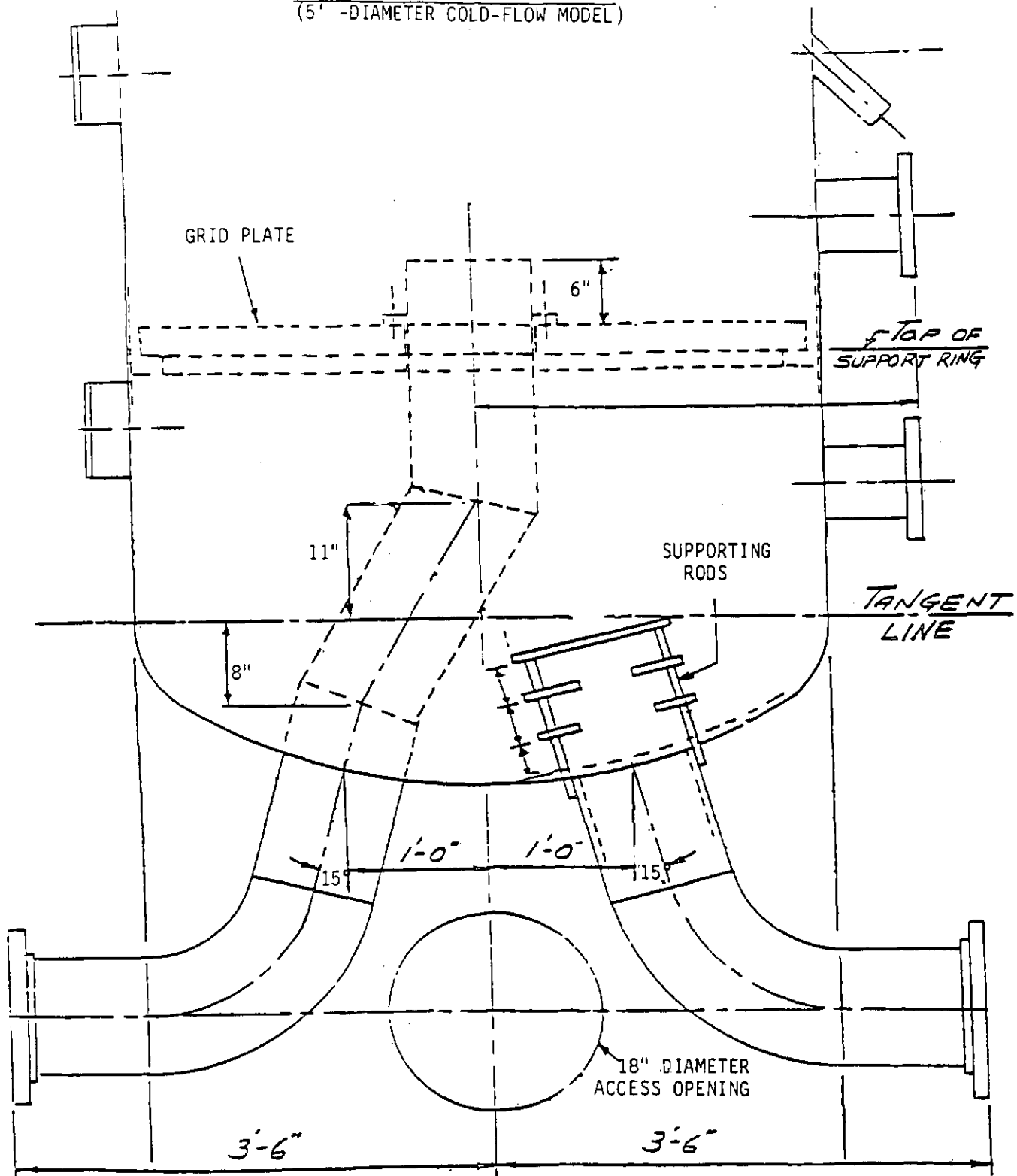


FIGURE 19

MODIFIED PLENUM INLET DESIGN  
(5' -DIAMETER COLD-FLOW MODEL)



APPENDIX A

CALCULATION PROCEDURES

## CALCULATION PROCEDURES

### CATALYST BED EXPANSION

Percent bed expansion is defined as:

$$\text{percent expansion} = 100 \times \frac{\text{bed height at conditions}}{\text{settled bed height}}$$

The settle bed height was visually measured after the liquid-filled vessel was charged with catalyst and before each day's operation. Adjustments to the settled-bed height were made whenever there was a catalyst carryover, by subtracting the amount of carryover from the amount of catalyst in the bed. The bed height at conditions was measured either visually or by a Texas Nuclear gamma-ray density detector.

### VOLUMETRIC HOLDUPS

Volume percents of the four phases (catalyst, liquid, gas and fines) were calculated from pressure-drop and bed-height measurements. The catalyst volume percent was calculated from the catalyst bed height and the mass of the oil-filled catalyst in the vessel.

$$\Sigma_C = \frac{M}{\rho_C AH}$$

where:

- M = mass of oil-soaked catalyst
- $\rho_C$  = density of oil-soaked catalyst
- A = cross-sectional area of the vessel
- H = catalyst bed height

The liquid volume percent was defined as:

$$\Sigma_L = \frac{-\Delta P_i}{H_i \rho_L} \quad (\text{for gas-liquid systems})$$

$$\Sigma_L = \frac{-\Delta P_i - \rho_C \Sigma_C}{H_i \rho_L} \quad (\text{for gas-liquid-catalyst systems})$$

$$\Sigma_L = \frac{-\Delta P_i}{H_i - \rho_C \Sigma_C} \quad (\text{for gas-liquid-catalyst-fines systems})$$

$$\rho_L + \frac{W_f \rho_L}{(100 - W_f)}$$

$$\Sigma_L = \frac{1 - \Sigma_C}{1 + \frac{\rho_L W_f}{\rho_f (100 - W_f)}} \quad (\text{for liquid-catalyst-fines systems})$$

where:

- $\Delta P_i$  = Pressure drop over vertical distance  $H_i$  in reactor
- $\rho_L$  = Liquid density
- $W_f$  = W % fines
- $\rho_f$  = Fines density

The fines volume percent was calculated by a weight balance and defined as:

$$\Sigma_f = \frac{\Sigma_L \rho_L W_f}{\rho_f (100 - W_f)}$$

The gas volume percent was calculated by difference for all four systems. Therefore:

$$\Sigma_g = 1 - \Sigma_L - \Sigma_C - \Sigma_f$$

### WEIGHT PERCENT FINES MEASUREMENT

The weight percent of fines in the slurry was calculated from the liquid density, the fines density and the measured slurry density. A nominal 200 cc sample of slurry was measured in a graduated cylinder and weighed on a triple beam balance. Then the W % fines is given by

$$W \% \text{ Fines} = \frac{100 V_s \rho_l \rho_f - 100 \rho_f}{\rho_l - \rho_f}$$

where:

$V_s$  = Specific volume of the slurry.

## PARTICLE SIZE ANALYZER

A Leeds and Northrup Microtrac particle size analyzer, Model No. 7991, was purchased by HRI in March 1980. This apparatus is capable of determining the volume-average diameter of a particle between 1.9 and 176 microns. It detects, but cannot distinguish between, particle diameters ranging from 1.2-1.9 and 176-250 microns.

The instrument reports a sample particle size distribution by determining the cumulative volume percents for particles which are smaller than thirteen preselected sizes based on diameter. It also calculates the volume mean diameter as well as the 10th, 50th and 90th numerical percentiles. These capabilities make this instrument an excellent tool for fast, accurate determinations of the particle size characteristics of multiple samples. Four samples can be analyzed within one hour when the same immersion fluid is used. The manufacturer's description of the analyzer is appended to this section.

The analyzer determines the particle diameter by measuring the Fraunhofer diffraction pattern of a particle illuminated by a plane of monochromatic light. The light intensity distribution is related to the particle radius by the airy formula:

$$I(w) = Ek^2a^4 \left[ \frac{J_1(kaw)}{kaw} \right]^2$$

where,

- I(w) = The intensity distribution
- w = The sine of the angle relative to the direction of the incident beam
- E = The flux per unit area of the incident beam
- k =  $2\pi$  divided by wavelength of the beam
- a = Particle radius
- J<sub>1</sub> = The first-order Bessel function of the first kind

Additional information on the theory is contained in an article in Applied Optics, V15, 1616 (1976) by A.L. Wertheimer and W.L. Witcock.

APPENDIX B  
UNIT DESCRIPTIONS



## UNIT DESCRIPTIONS

### EBULLATED CATALYST BED

A unique feature of the H-Coal Process is the ebullated catalyst bed which allows for intimate contact between the reactants and the catalyst particles. As the liquid flow through the catalyst bed is increased, the bed begins to fluidize and expand slightly as the catalyst particles separate and begin to move. A further increase in the liquid flow will cause the bed to expand even more as the motion of catalyst particles increases, but there is very little increase in the pressure drop.

Theoretically, the smaller the expansion, the better the reactor space utilization but in practice, an expansion of about 50% is needed to ensure sufficient catalyst motion for good mixing and heat distribution. The choice of 50% has been based on the operating experience of several bench-scale and larger ebullated-bed units.

HRI's ebullated bed is basically a liquid fluidized bed with the characteristics of particle fluidization. It differs, however, in several important respects from the better known gas fluidization system. The ebullated-bed system has a sharp demarcation between the fluidized catalyst bed and the solids-free fluid above the bed. The solid catalyst particles within the bed are in random, churning motion, which results in excellent solid-liquid contact for reaction and heat transfer. In contrast, a gas fluidized system is characterized by violent motion, bubbling and streaming of particles within the bed with very poor definition of the upper bed interface. In an ebullated-bed reactor, although some amount of gas and vapor is necessary for reaction, it should be limited to a superficial velocity of approximately 0.2 ft/sec at reactor inlet conditions. Above this rate the bed has a tendency to exhibit characteristics of aggregative fluidization with bubbling and channelling in the bed, and with a migration of catalyst to the top of the reactor. This condition is avoided by minimizing the gas and vapor at the inlet.

### SIX-INCH UNIT (176)

A six-inch diameter, Plexiglas, cold-flow column was used to study the ebullation characteristics of HRI's PDU. A schematic diagram of the system is shown in Figure B-1. Figures B-2 and B-3 are detailed sketches of the recycle cup and bubble caps used in the column. During operations, the liquid/slurry phase is pumped either from a 165 gallon clear oil tank or from a 110 gallon slurry mix tank into the column's plenum chamber. Nitrogen is mixed with the other incoming material in the inlet chamber forming a well-mixed multiphase mixture which enters the catalyst zone of the column through the distributor plate. The effluent from the column then enters a flash vessel from which the nitrogen is vented. The bottoms from the flash separator are returned to the appropriate holding vessel depending on the mode of operation. An internal liquid/slurry recycle, characteristic of H-Coal/H-Oil™ reactors, aids in increasing back mixing and in controlling the catalyst bed height.

This unit operates between pressures of 0 to 20 psig and between temperatures of 80 to 100°F, as controlled by a water-cooled heat exchanger. Sufficient pump capacity is available to operate the unit at superficial liquid/slurry velocities of as much as 0.21 ft/sec. Nitrogen is used to supply gas velocities of as much as 0.30 ft/sec.

There are several pressure taps in the column for measuring pressure drops across the grid plate, within the catalyst bed and across the recycle cup.

### FIVE FOOT UNIT (UNIT 216)

A five-foot-diameter, carbon-steel, cold-flow vessel (shown in Figure B-4\*) was used to investigate the distribution characteristics in the H-Coal Pilot Plant reactor. The system is similar in design to the six-inch unit. However, a separate nitrogen compressor is used to achieve gas velocities up to 0.22 ft/sec. A single reservoir tank is used as either a slurry mix tank or as a clean oil tank depending on the mode of operation.

The unit can be operated at pressures between 0 and 100 psig and at temperatures up to 400°F. Sufficient pump capacity is available to operate at superficial liquid velocities up to 0.12 ft/sec. The separator design limits the vessel's effluent flow to approximately 300 gpm.

This unit has a number of pressure taps to measure pressure drops across the grid plate, within the catalyst bed (at three locations 120° apart) and across the recycle cup. Eighteen sight glasses were installed in the vessel wall in order to observe various locations of the vessel. Figure B-4 shows the approximate location of 300 psig 6-inch pressure-product sight glasses. Six of them are located at intervals of about 60° around the vessel, just below the distributor plate. Another six are located 2 feet above the distributor plate. Three are staggered between 8 and 9 feet above the distributor plate. Finally, three are located just above the internal recycle cup.

\* Original plenum inlet design shown in this sketch.

176 UNIT SCHEMATIC DIAGRAM

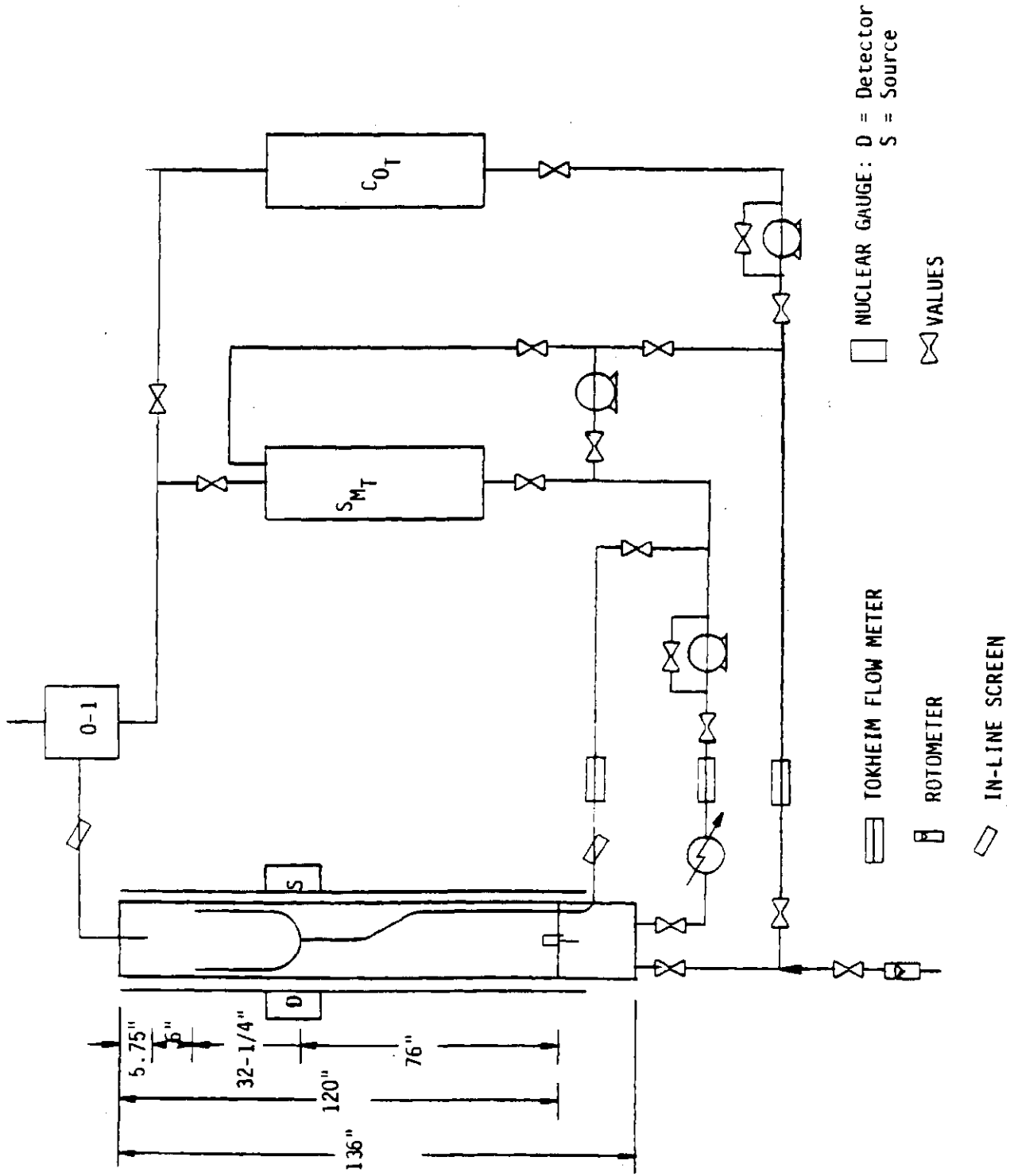


FIGURE B-2

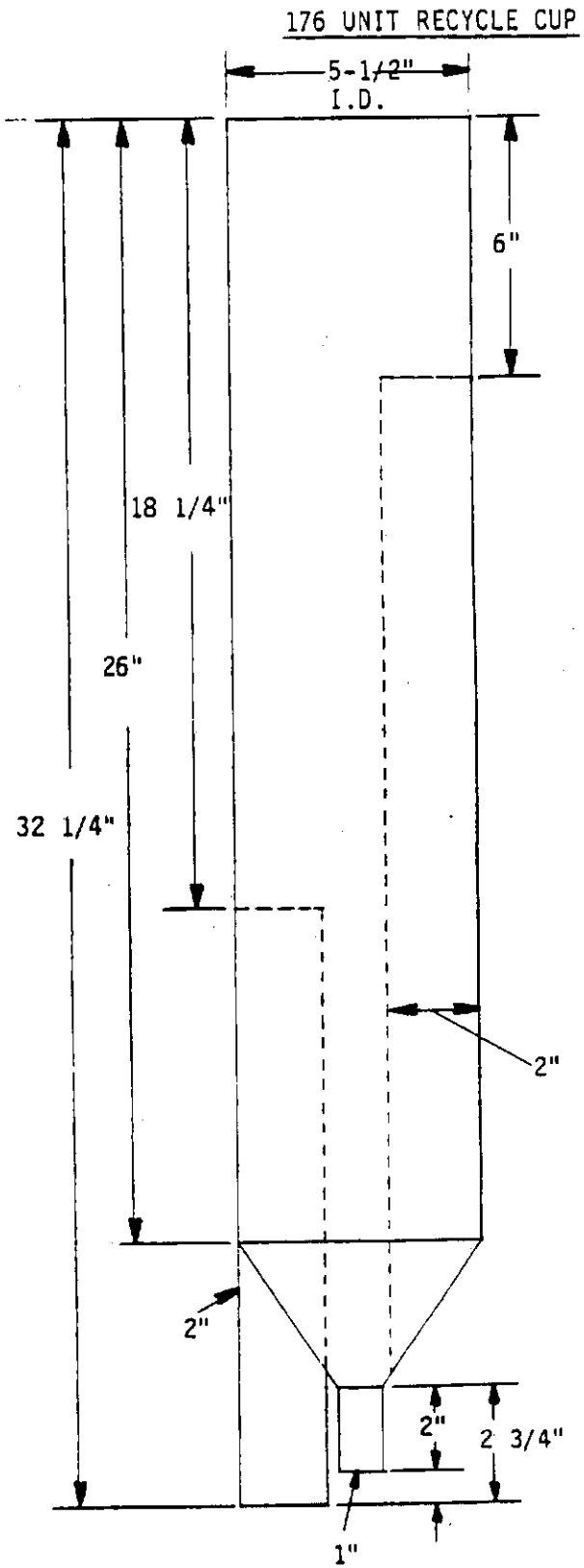


FIGURE B-3

176 UNIT BUBBLE CAP

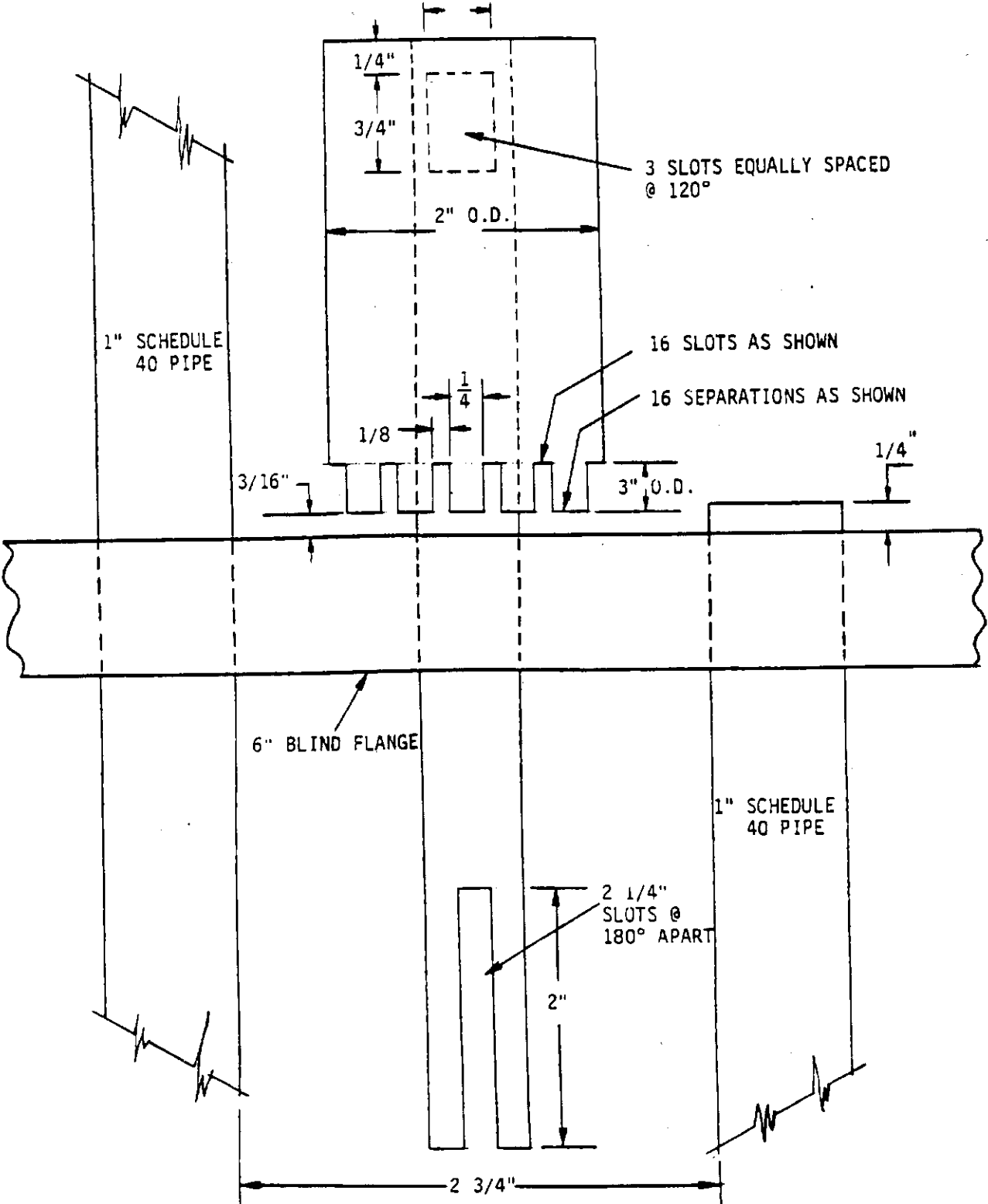


FIGURE B-4

COLD FLOW MODEL (5' -DIAMETER X 23' -HEIGHT) OF UNIT 216

