4.0 EXPERIMENTAL SECTION

4.1 Program Plan

This program consisted of the following tasks:

Task 1 - Experimental Planning
Task 2 - Two-Phase and Three-Phase Flow in Vertical Columns
Task 3 - Effect of Distribution on Flow
Task 4 - Solids Removal Study
Task 5 - Organic Fluid Phase
Task 6 - Effect of Liquid Properties
Task 7 - Final Report

4.2 Cold Flow Model Equipment

Both a 5-in.-diameter by 5-ft-long Plexiglas column and a 12-in.-diameter by 25-ft-long glass column were used in the cold-flow simulation study. These two columns are described separately in the following sections.

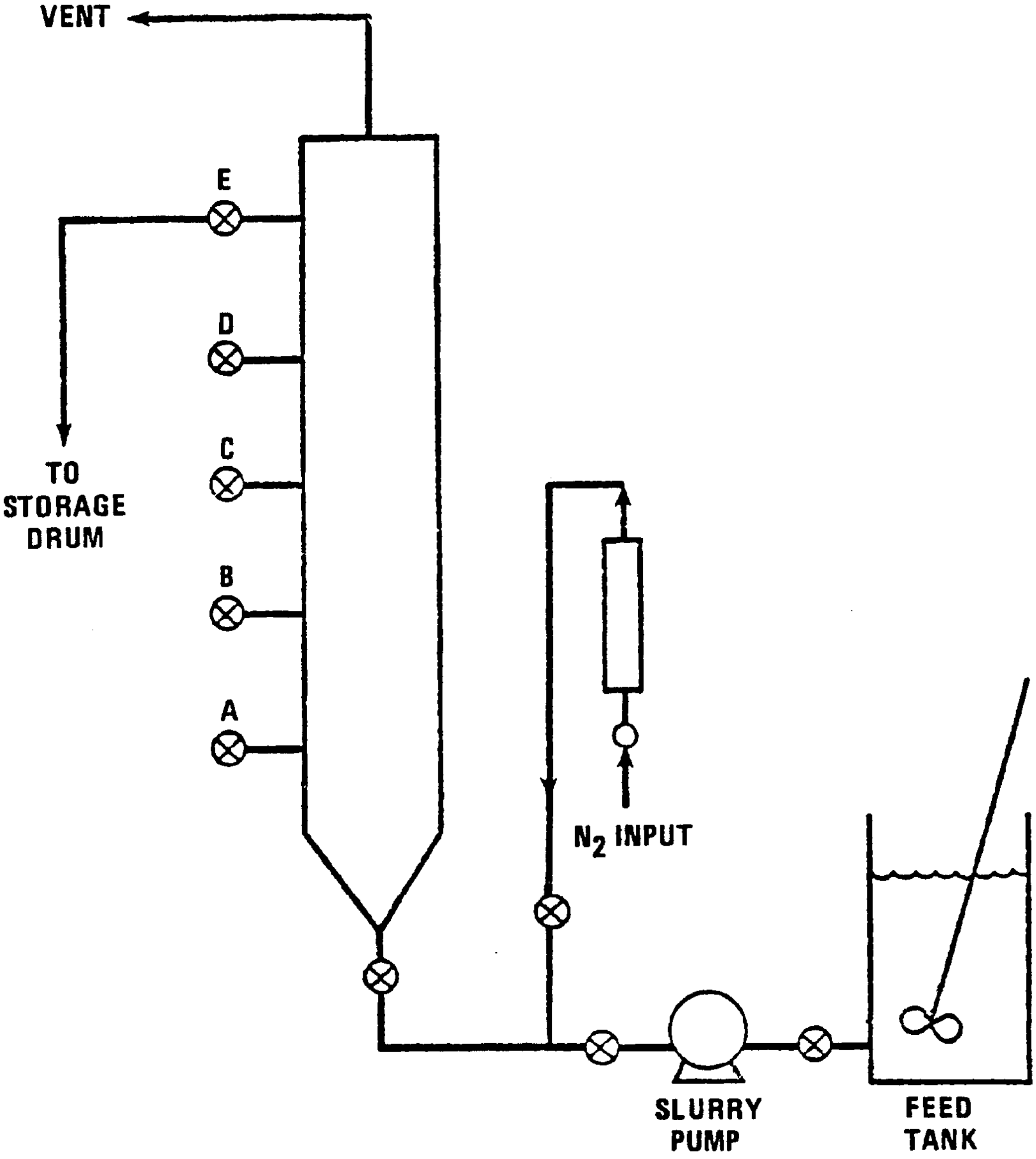
4.2.1 5-Inch Diameter Column

A schematic of the 5-in. x 5-ft column is shown in Figure 1. Both the column and the conically shaped bottom inlet (60 degree angle with the horizontal) were fabricated from Plexiglas. The bottom was designed so that it would accept a distributor plate. Five sampling taps (A-E) were located along the column. In typical operation, slurry exited through the topmost opening (E) and gas vented through the uppermost top central opening.

Slurries were prepared by mixing sand and water in a 110-gallon feed tank equipped with a Chemineer agitator (10 in. propeller). Slurry was pumped into the column with a Moyno progressing cavity pump. Gas was co-fed into the bottom through the common opening. Slurry exiting

Figure 1

Schematic Of 5-Inch Diameter Plexiglass Column



the column was stored separately in a second drum to avoid any complications due to partitioning of solids of different particle sizes in the column. (Recycling into the feed vessel under certain conditions would result in a feed composition depleted in the larger particles.) A 10 cm^3 syringe was also attached to the column at the bottom in order to inject tracer into the feed stream for dispersion studies.

4.2.2 12-Inch Diameter Column

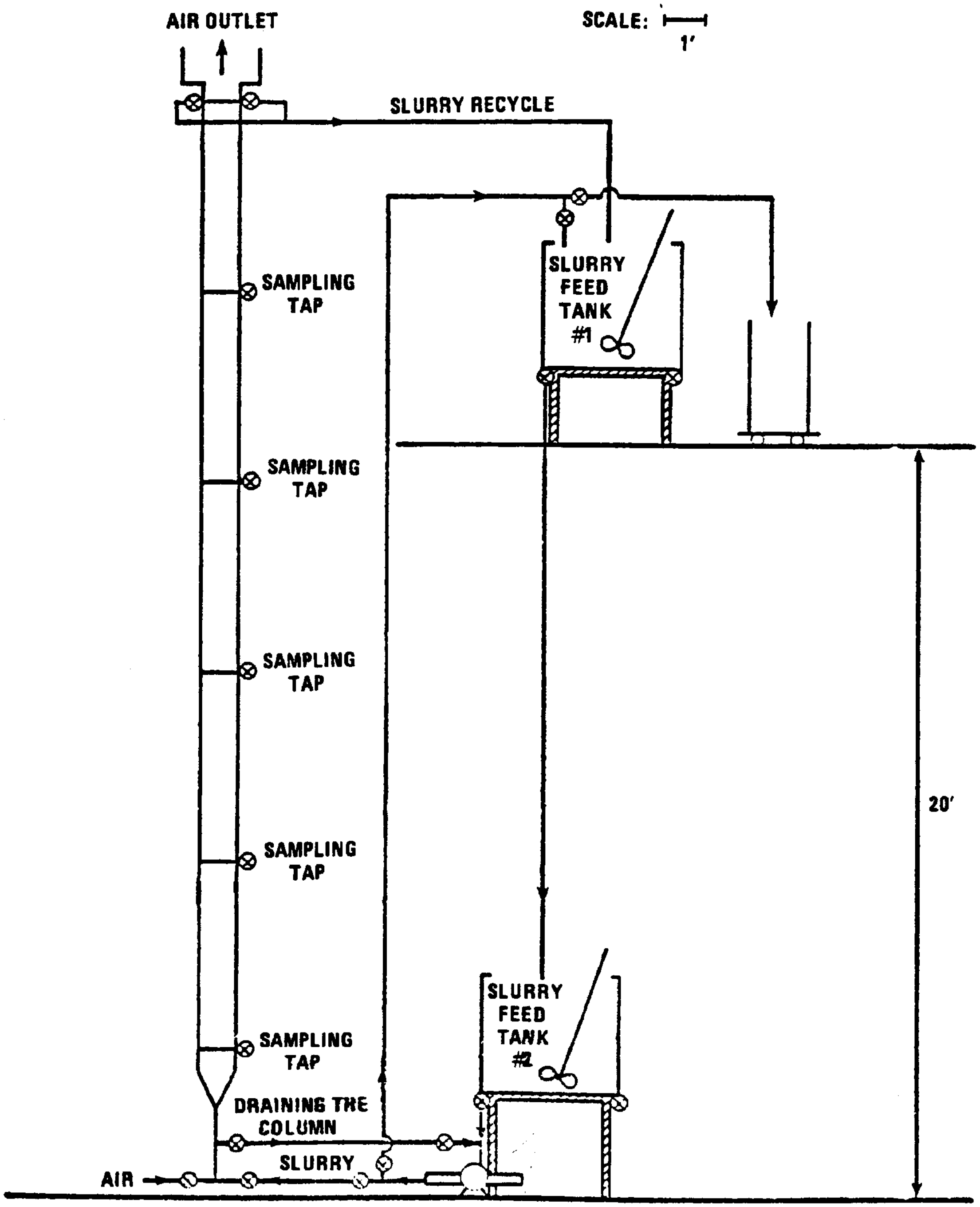
A schematic of the 12-in. diameter column is shown in Figure 2. The flow paths of slurry and gas were identical in both columns although larger slurry inventory and equipment were required for the 12-in.-diameter column. The column comprised of five sections of 5-ft glass tubing interconnected with machined metal flanges. The column rested on scaffolding that completely surrounded the column. Guide bars at the top kept the column in true vertical alignment by preventing the column from tilting. The supporting structure had additional horizontal bars to serve as a ladder to service the column and sample the column through the ports in each interconnecting flange.

The inlet to the column was a conically shaped glass cone having a 60° angle with the horizontal. The cone was suspended from the support plate and could be removed for insertion of a distributor plate without having to dismantle the entire column. A 400 cm^3 tube was attached to the column at the bottom for injecting tracer during dispersion studies. The top of the column was enlarged to disengage the gas from the liquid phase. The column was completely wrapped with metal screen (0.5-in. openings) to protect personnel from flying glass in case of rupture.

Two different distributors were examined in the 12-in. diameter column:

Figure 2

Schematic Of 12-Inch Diameter Glass Column



- Distributor #1

This distributor which closely resembles that in Wilsonville's dissolver, has seven 9/16-in. openings with a 3-3/4-in. center-to-center spacing. A 3-1/2-in. long, with 9/16-in. internal diameter (i.d.) riser tube was welded to each opening (see Figure 3). Two 1/4-in. by 1-in. rectangular slots face each other at the end of each riser tube. The bottom of the distributor faces the inlet cone of the column. On the top side of the distributor, a 1-1/14-in. by 3-1/2-in. long tube protrudes through each hole in the plate. Each tube was covered by a cap, leaving a 5/32-in. spacing between the tube and the cap. The gas/slurry mixture flows through the riser tube and enters into the upper tube. This mixture continues to climb through the upper tube and then runs down along the spacing between the upper tube and the cap. The material finally flows out of the cap through a 1/4-in. gap between the cap and the distributor plate. The complete assembly is shown in Figure 4.

- Distributor #2

This plate has nineteen 9/16-in. openings with a 2-in. center-to-center spacing, as illustrated in Figure 5. Each hole has a riser tube identical to that in distributor No. 1, but a bubble cap and upper tube are not attached to the other side of the plate. The distributor is shown in Figure 6.

In the study of organic fluids, the entire 12-in.-diameter column was enclosed so that volatile flammable liquids could be handled safely. A schematic drawing of the enclosure and its associated safety features are depicted in Figure 7.

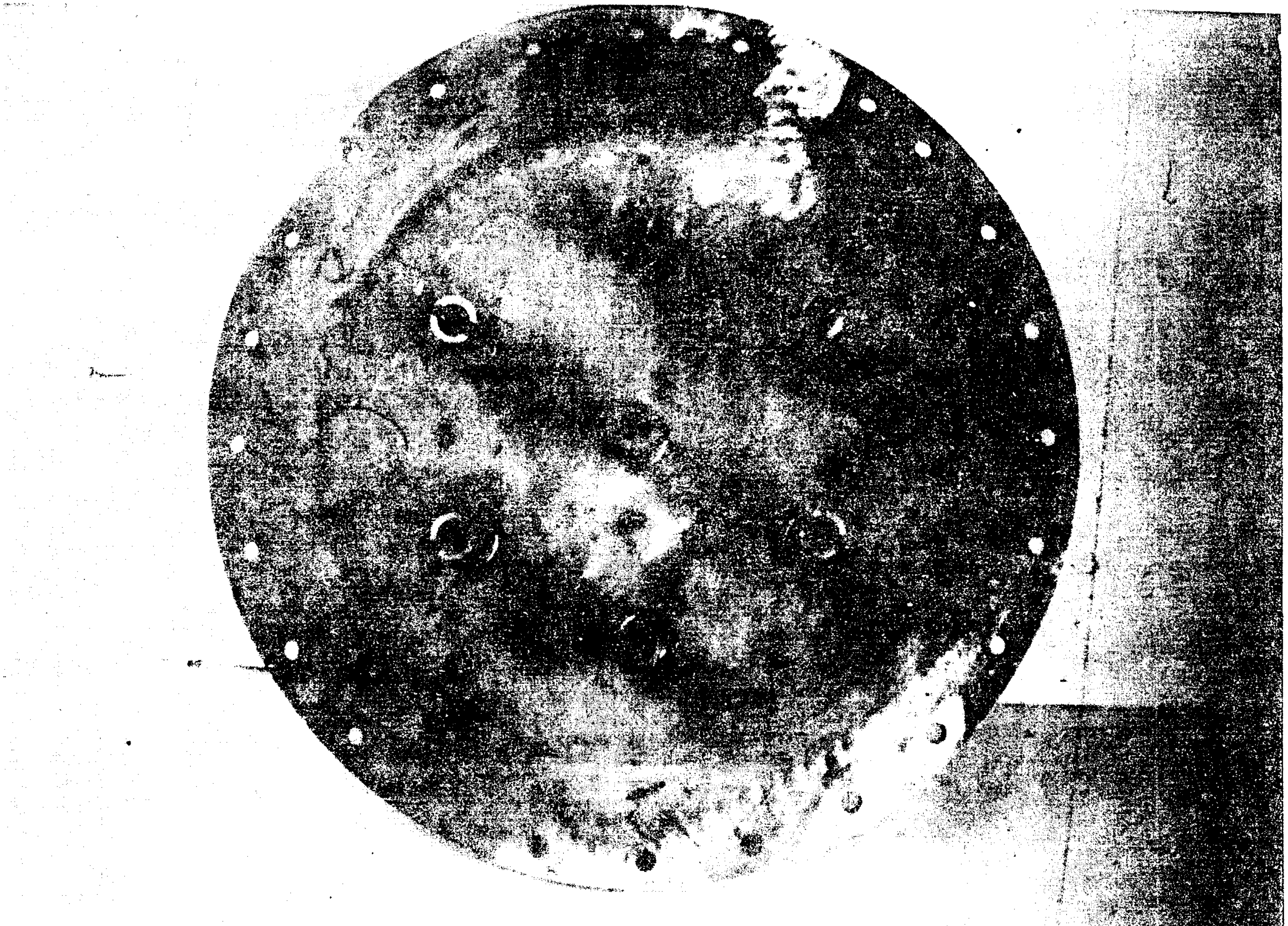


Figure 3

Distributor Number 1

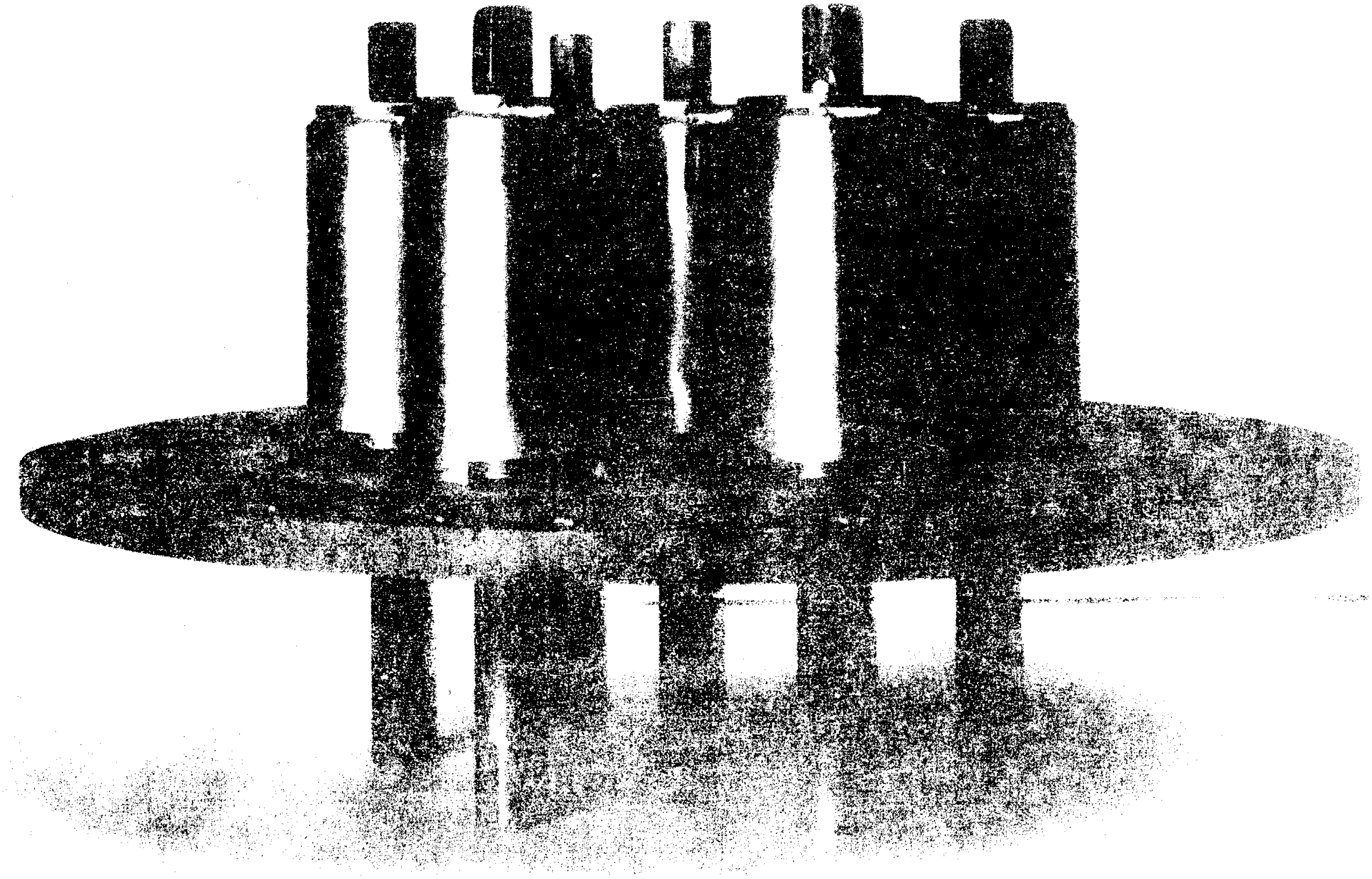


Figure 1

Distributor Number 1

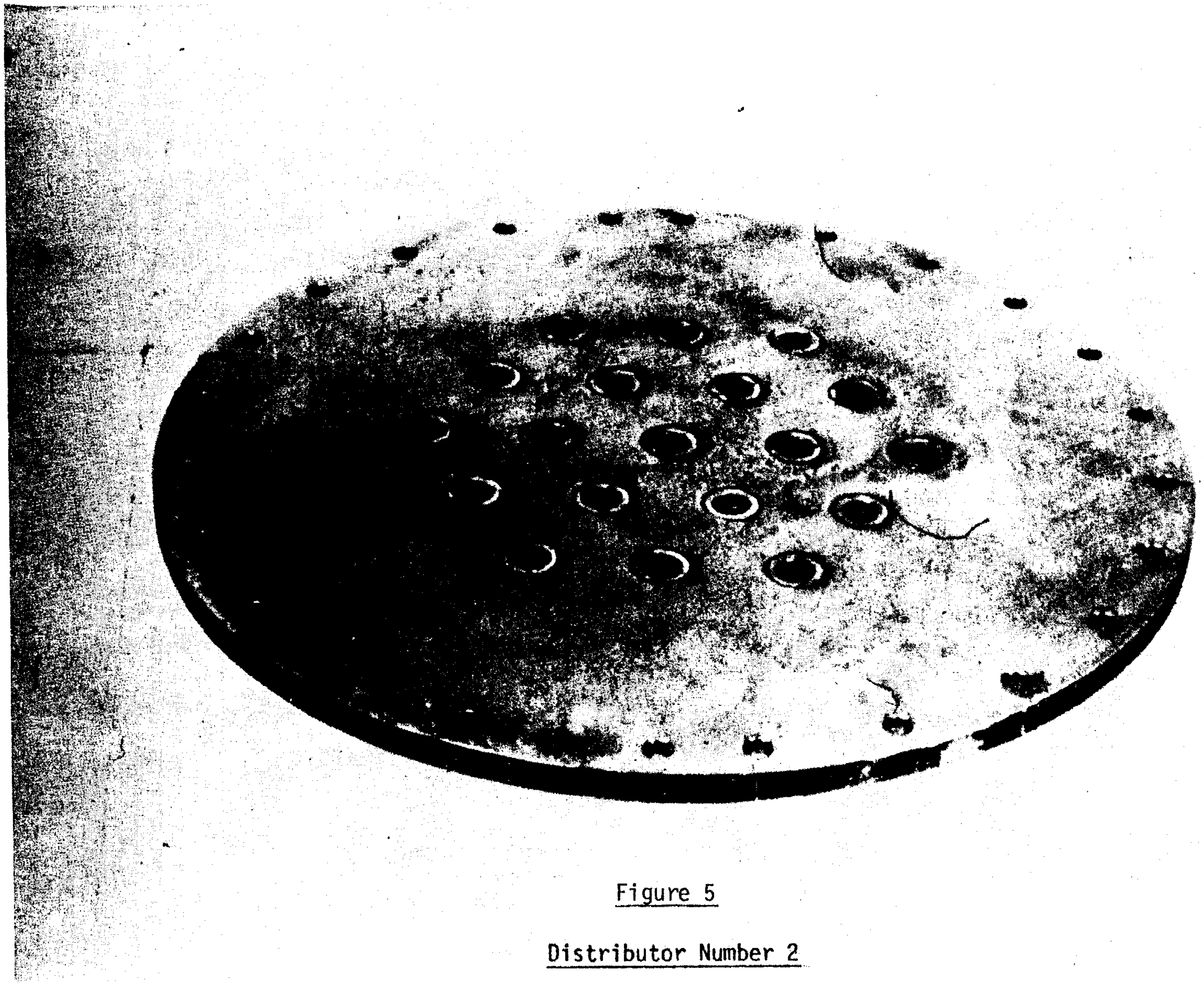


Figure 5

Distributor Number 2

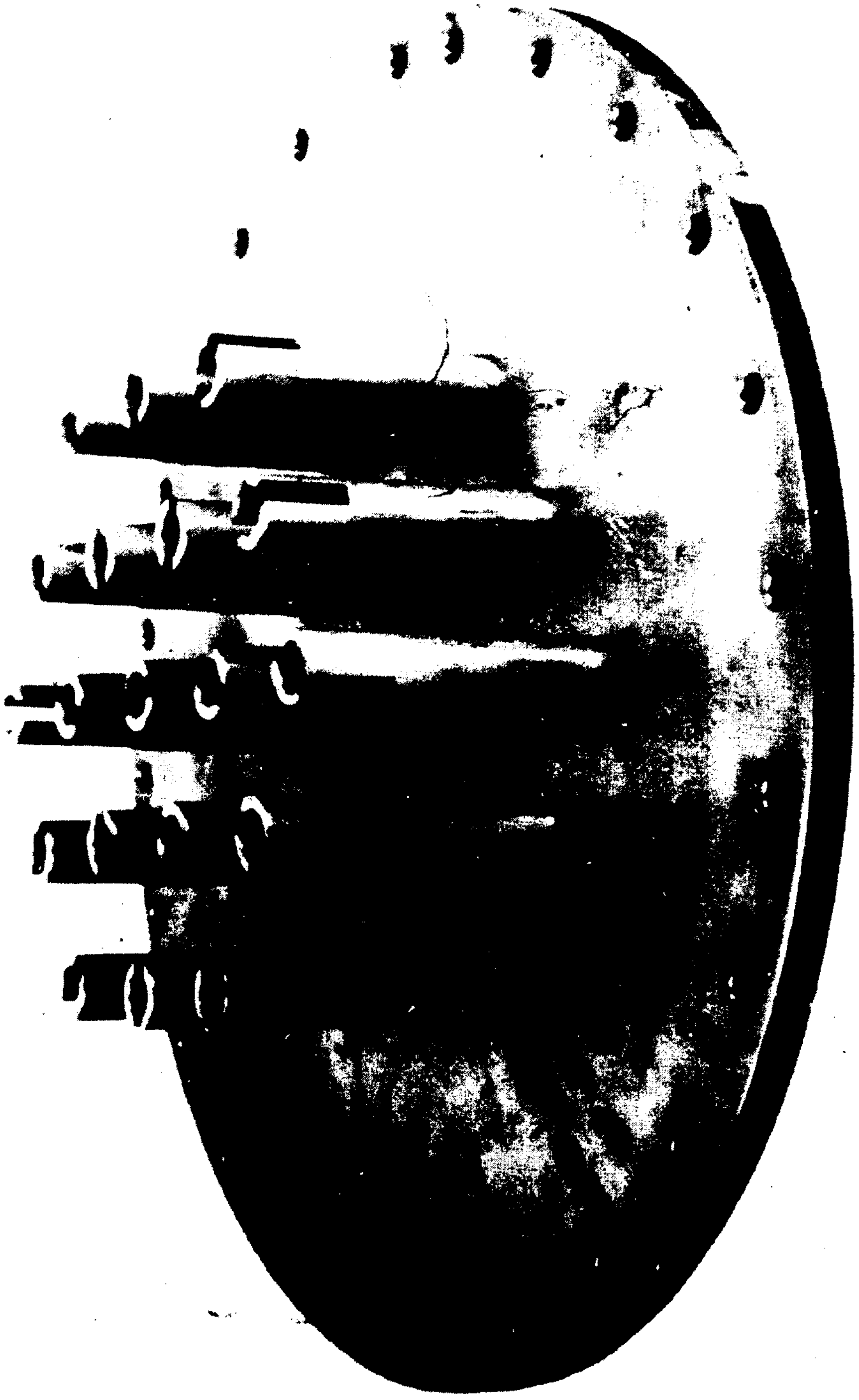
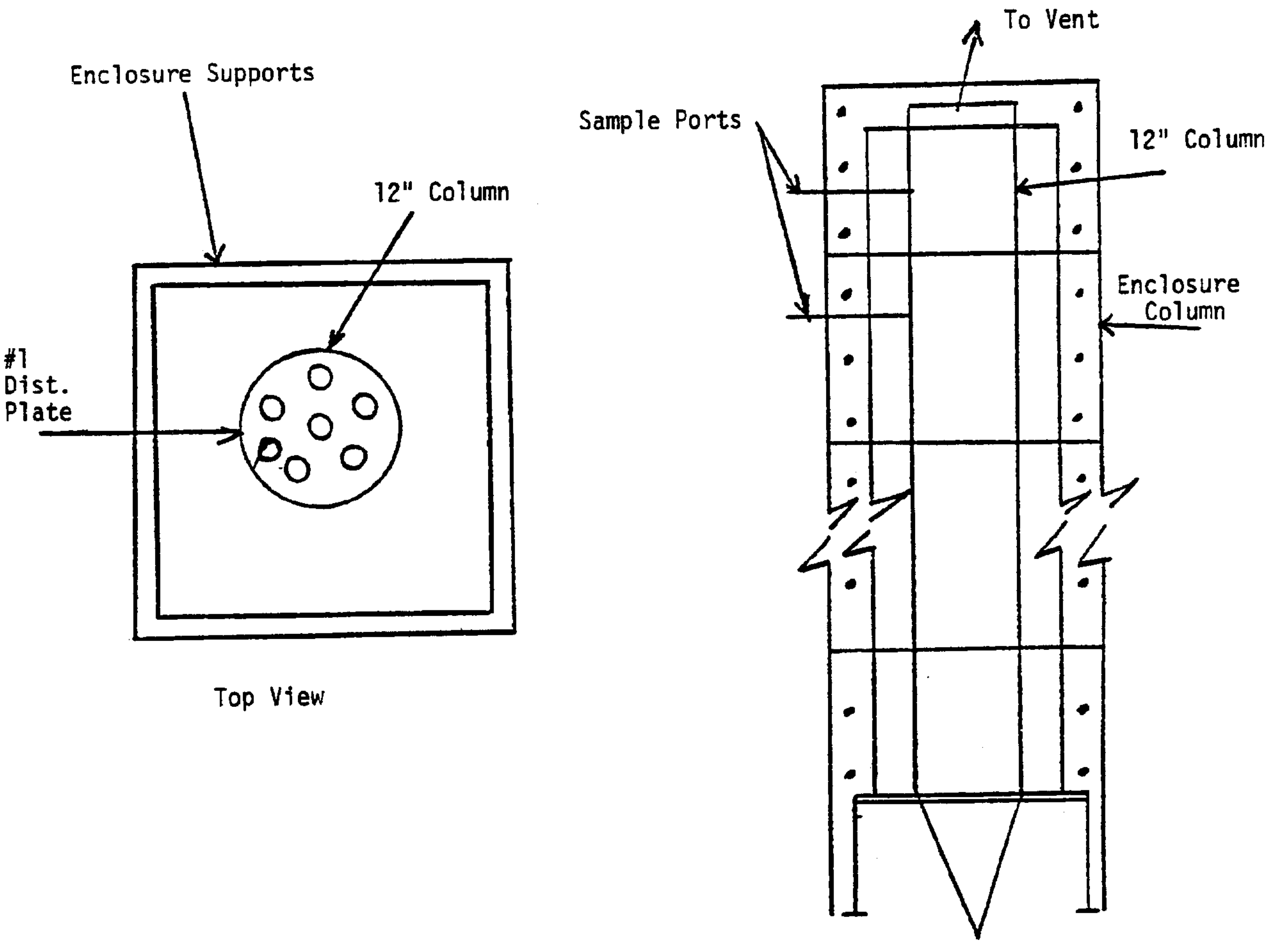


Figure 6

Distributor Number 2

Figure 7

Schematic Diagram of the 12-Inch Diameter Column Enclosure



Top View

Bottom View

The enclosure completely encompassing the 12-in. diameter column is made of 1/2-in. thick Plexiglas and 1/8-in. thick aluminum sheets bolted to an aluminum angle-iron framework that stands independently on the floor. Horizontal bars were used to connect the enclosure to the surrounding scaffolding, ensuring a rigid structure. The lower 8 feet of the column is completely leak proof; and the strength of this section was checked hydraulically, to guarantee that it could hold the entire contents of the glass column in case of breakage. A set of damper blades was installed as a top cover for the enclosure and a large blower was installed to provide adequately vent for nitrogen and any entrained volatile fumes.

4.3 Experimental Procedures

The procedures described below are essentially the same for both the 5- and 12-in. diameter columns, the only difference being the quantity of tracer injected into the columns for liquid dispersion experiments. The variable ranges were chosen to include the operating conditions for the SRC-I Pilot and Demonstration Plants. Operation systems included: air/water, air/water/sand, nitrogen/tetralin, nitrogen/tetralin/sand, nitrogen/glycol-water, and nitrogen/glycol-water/sand. Operating conditions were as follows: Liquid superficial velocity, 0-0.05 ft/sec; gas superficial velocity, 0.05-0.50 ft/sec; solid particle size, 20-140 minus mesh; solid concentration, 0-40 lb/ft^3.

4.3.1 Gas Holdup

Gas holdup and the effects of solid particles on holdup were measured both without and with fluid flow. In the absence of liquid flow, the column was completely filled with liquid and then gas was passed through the liquid at specified rates. Excess liquid exited the column at the top through a side opening. After waiting a period of 5 minutes to ensure that a steady state was achieved, the bottom valve was closed to shut off the gas input. The final liquid level was measured; and the difference between the initial and final levels represented the gas holdup at that particular gas-flow rate.

With fluid flow, the liquid and gas entered the column through a centrally located opening at the bottom. Excess liquid exited the column through a side opening at the top. After a steady state was reached, the liquid level was measured. Then a common valve at the bottom was closed, stopping both liquid and gas flow simultaneously. The gas void fraction was measured as described previously.

Two different solid concentrations were used to study the effect of solids on gas holdup. For experiments using large particles, such as 20/30 mesh sand, a known amount of sand was initially placed in the column and the experiments were conducted as described above. The sand particles that were carried out of the column, if any, were collected to account for an average solids concentration during the run. For experiments using fine particles, such as -140 mesh sand, a slurry containing sand and water was pumped into the column at a specific flow rate. The experimental procedure for determining holdup was the same as described above.

In order to qualitatively understand the bubble sizes in the column, we photographed the column. The experimental procedure was similar to the one used to measure gas holdup. Both liquid and gas were allowed to flow through the column at predetermined flow rates. After a steady state was established, a common valve at the bottom of the column was closed, stopping both liquid and gas flow simultaneously. The aerated liquid level dropped as the gas phase left the system. This change of aerated liquid level as a function of time was recorded photographically at 1-sec intervals. From the rate of level drop, the relative bubble size could be determined qualitatively.

4.3.2 Liquid Dispersion

Liquid-phase dispersion was investigated using a tracer detection method. Sodium chloride was used as the tracer, and a conductivity probe mounted at the exit line of the column continuously monitored the ion conductivity of the solution. Liquid flow rates were measured periodically to ensure a steady-state flow. An ultrasonic doppler flow meter was also used to check for fluctuations in the liquid-flow rate. Gas-flow rate was monitored using a flow meter that was precalibrated with a dry-test meter.

In the typical experimental procedure, liquid and gas were passed through the column at the desired rates. A recorder monitored signal from the conductivity probe. After a steady base line was established on the recorder, a pulse of 400-cm^3 concentrated NaCl solution (0.243 g/cm^3) was injected into the bottom of the 12-in.-diameter column. Typical injection time was between 2 and 5 sec. The conductivity of the existing solution was recorded as a function of time. The experiment was terminated when the recorder signal reached the preset base line; at this point, gas flow was stopped, although water flow is used for 5-10 min to ensure that the column had been purged of residual salt before the next experiment started.

A slightly different procedure was followed with solid particles. To determine the effects of solids on liquid dispersion, a constant concentration of solids must be maintained in the column while other parameters such as gas and liquid flow rates are varied. For practical reasons, slurry was not pumped into the column. In order to perform just one slurry pumping experiment, more than 1 ton of sand would be required to prepare a slurry feed containing 20 lb/ft^3 of sand at a slurry feed rate of 17.5 gpm (the highest rate used in these experiments). Handling that volume of sand is extremely difficult and time-consuming. Instead, a different procedure was used that maintained the desired solids concentration and eliminated the need for massive amounts of material sand. First, the column was partially filled with water. A predetermined amount of sand (20/30 mesh) was added to the column.

Then the column was completely filled with water at a low flow rate. The 20/30-mesh sand particles were large enough to prevent their exiting the column during this slow filling process. However, minor amounts of sand did leave the column during the time that a steady state was being achieved. During the experiment, solid concentration in the column was maintained by monitoring the amount of sand that exited the column.

Plots of conductivity vs. time obtained from these experiments were transformed to dimensionless curves, and tracer curves were prepared. The tracer curves were then fitted with theoretical curves predicted from an axial dispersion model, which was based on the analogy between mixing in the actual flow and a diffusional process. A computer model was also developed to generate a family of axial dispersion curves having different Peclet numbers. The dispersion coefficient in the liquid phase was calculated from the Peclet number of the best matching curve.

A different method was used to measure the liquid dispersion coefficient in a nitrogen/tetralin system. The liquid dispersion experiments were performed in batch mode (no liquid flow). Typically nitrogen was bubbled at a desired rate through the column filled with tetralin. A concentrated blue dye solution was injected at the bottom of the 12-in.-diameter column. The blue dye solution was selected as trace for tetralin dispersion because (1) it is completely miscible with tetralin (2), it exhibits a maximum absorbance at a wavelength significantly different from that of tetralin, and (3) only a small quantity is needed to give good measurement sensitivity. Samples were taken 20 ft above the injection point every 20 sec. The amount of sample drawn from the column was kept very small to avoid disturbing the equilibrium level. The concentration of dye in the sample was determined from its adsorbance as measured by a colorimeter.

In the nitrogen/tetralin/sand system, the solid particles in the sample were allowed to settle before adsorbance measurement. Then the adsorbance vs. time plots were transformed into dimensionless curves, from which the liquid dispersion coefficients were determined.

4.3.3 Solid Dispersion

Solid dispersion experiments were conducted in both 5-in. and 12-in.-diameter columns using batch as well as continous operating modes. In batch operation, gas was bubbled through the column, which was filled with liquid and a known weight of solid particles. During a 30-min bubbling period, steady-state conditions were established for each gas velocity. Then slurry samples were withdrawn from sampling ports at various column heights and the concentration of solids was measured.

During continuous operation, the water/sand slurry flowed continuously through the column. Samples were withdrawn from all ports at periodic intervals over 4-6 hr until a steady-state condition was achieved. Two different types of continuous experiments were conducted using the 5-in.-diameter column. In the first method, a water/sand slurry of known concentration was fed to the column on a once-through basis. This method, however, required a minimum of two feed tanks in series and an enormous amount of effort and material to keep up with the once-through operation. The second method involved recycling the column exit stream back to the feed tank; a slurry of known concentration was prepared in a feed tank, and the exit from the 5-in.-diameter column was returned to the feed tank, thus creating a closed loop.

In experiments with the 12-in.-diameter column, two feed tanks were used, and the exit from the column was returned to the first tank, which in turn was fed to the second tank in series. The advantages of this method are: a) less sand is required per experiment; (b) less labor is needed to prepare a fresh batch of slurry every 10 min.; and (c) there is less possibility of human error than when multiple batches are prepared in a short time. However, the solids accumulation measured from the recycle mode differs significantly from that measured for the once-through operation because the particle-size distribution of the feed material is different. In the recycle mode, the larger particles of the feed initially prepared in the tank are depleted because they are retained in the column. This leads to a slightly higher estimate of solid accumulation based on the steady-state feed concentration and size distribution. This difference decreased with a decreasing particle-size range in the feed, however. Therefore, using a narrow size range could reduce the error caused by the recycle mode operation. One set of operating conditions was duplicated in both methods to check the experimental reproducibility and sample relationship to each other. This result will be presented in a later part of this report (discussion of solid dispersion).

4.3.4 Solid Removal

Solids removal experiments were similar to solids distribution experiments, with minor modifications. Slurry and gas were allowed to flow through the column in a recycle loop for several hours to achieve steady state. Samples were taken along the column to obtain profiles of steady-state solids concentration. Then the lowest sample port in the column (just above the distributor plate) was opened, and the slurry was allowed to flow into a storage tank while the slurry and gas flow into the column was maintained. Simultaneously, recycle operation was changed to once-through operation. However, in the once-through mode, the length of the experiment was limited by the slurry inventory in the holding tanks. Most of the experiments were terminated 15 min after the lowest sampling port was opened. In this short time, multiple sets of samples were obtained along the axis of the column in order to measure changes in the solids distribution profile.

4.3.5 Particle/Particle Interaction

A special set of experiments was undertaken to investigate the effects of settled particles and particle/particle interaction on solids accumulation in the 12-in.-diameter column. Three consecutive experiments were performed using various particle sizes: -140 mesh, 60/80 mesh, and 20/30 mesh. A slurry containing -140 mesh particles and water was prepared in the feed tank. This fine-particle slurry recycled through the column at a liquid velocity of 0.05 ft/sec, with gas flowing at a velocity of 0.33 ft/sec. A steady-state solid concentration profile was obtained by taking samples along the column. At the end of the run, a known quantity of 60/80 mesh sand was added to the feed tanks, and the new slurry was recycled through the column at the same operating conditions. Samples were withdrawn from the sampling ports until a new equilibrium state was reached. In these samples, in addition to determining the total solids concentration, the concentration of different particle sizes was also determined. At the end of the experiment, a known amount of 20/30 mesh particles was added to the column to simulate the presence of settled particles. The slurry was allowed to flow through the column at identical operating conditions. Again concentration of different-sized particles and the total solids concentration were measured for each sample withdrawn from the column. At the end of the experiment, the column was drained and the sand retained in the column was weighed.

4.3.6 Gas/Liquid Mass Transfer

Gas/liquid mass-transfer studies were also conducted in the 5-in. and 12-in.-diameter columns, in a batch operation (no liquid flow). The mass-transfer rate was measured as the rate of oxygen dissolving in water. Polarographic probes (stationary and submersible) continuously monitored the concentration of dissolved oxygen in the solution as a function of time. The experimental procedure described below was used with for both columns.

The oxygen content of water in the 5-in.-diameter column was measured with stationary probes installed at various column heights. For the 12-in.-diameter column, the submersible probes were used because the column structure prohibits installing stationary probes. Also, several measurements were made using both types of probes in the 5-in.-diameter column at various heights. Negligible differences in the measurements ensured the validity of using the submersible probes.

At the beginning, nitrogen was allowed to flow through the column filled with water to remove oxygen. After the oxygen concentration in the dissolver leveled out at zero for several minutes, the nitrogen flow was stopped and air was introduced at a predetermined rate. Changes in the dissolver oxygen concentration as a function of time were then recorded at three different positions for the 5-in. diameter column and at two different positions for the 12-in. column. The rate of oxygen uptake was used to calculate the gas/liquid mass-transfer coefficient using a completely backmixed model.