



GAS/SLURRY FLOW IN COAL LIQUEFACTION PROCESSES (FLUID DYNAMICS IN 3-PHASE FLOW COLUMNS). QUARTERLY TECHNICAL PROGRESS REPORT, OCTOBER 1, 1979-31 DECEMBER 1979

AIR PRODUCTS AND CHEMICALS, INC. ALLENTOWN, PA

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GAS/SLURRY FLOW IN COAL LIQUEFACTION PROCESSES (Fluid Dynamics in 3-Phase Flow Columns)

Quarterly Technical Progress Report for Period 1 October 1979-31 December 1979

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<u>Summary</u>

This is the first quarterly report under Contract Number DE-ACO1-79ET14801 titled "Gas/Slurry Flow in Coal Liquefaction Processes". This work covers the period 1 October 1979 to 31 December 1979, the initial three months of a two year program. This work is a continuation of studies initiated by Air Products and Chemicals, Inc. on the fluid dynamics of 3-phase flow to support the design of 6000 T/D dissolver in the SRC-I demonstration plant. Design studies on the SRC-I 6000 T/D demonstration plant began in July, 1978. DOE support for 3-phase flow studies by Air Products began 1 July 1979 under the Bridging Task program and terminated 30 September 1979 at the start of the current contract. A background of information developed at Air Products is included in the text.

Gas holdup in gas/liquid and gas/liquid/sclid systems were investigated both in the presence and absence of liquid flow. The variables studied were: particle size (20/30 mesh and less than 100 mesh), solids concentration (0, 5, 11 and 12.7 lbs/ft³), liquid velocity (0 to 0.05 ft/sec) and gas velocity (0.036 to 0.368 ft/sec). Gas holdup is found to be independent of liquid velocity which agrees with most investigators. At low superficial gas velocities (up to 0.10 τ t/sec) the presence of solids did not change the gas holdup. However, at high gas velocities, only the presence of fine particles (less than 100 mesh) decreased holdup. The most severe reduction in holdup (13.9%) occurred at the highest gas velocity (0.368 ft/sec) and the highest solid concentration (11 lb/ft³). Almost all of the observed decrease in holdup due to the presence of solids is very small and the gas holdup data can be reasonably described by the correlation of Yoshida and Akita (1) except at the high concentration (11 lb/ft³) of fine particles (less than 100 mesh).

Liquid dispersion experiments were conducted in ans/liquid and gas/liquid/solid systems. All the three-phase experiments presented in this report were done with 20/30 mesh sand. The variables studied were: liquid velocity (0.01 to 0.05 ft/sec), gas velocity (0 to 0.327 ft/sec) and solids concentration (5 and 20 lbs/ft³). Liquid axial dispersion coefficients were found to be independent of liquid velocity. Increasing gas velocity, however, increased the liquid dispersion coefficient. The presence of solid particles decreased the This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed or represents that its use would not infringe privately owned rights.

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1.0 OBJECTIVE

The overall objective of this project is to study the solids accumulation and suspension of various gas/liquid/solid systems in cold-flow tubular columns aimed at providing data for the coal dissolver design in the SRC-I demonstration plant.

The specific objectives are:

- To check the adequacy of the existing experimental apparatus using a two-phase system (air/water mixture).
- 2. To study the effects of slurry and gas velocities, solid particle size and concentrations, and liquid viscosity and surface tension on the performance of a cold-flow tubular column.
- 3. To develop an effective slurry withdrawal technique from the bottom of a tubular column as a means to control the solid concentration in the column.
- To study the performance of cold-flow tubular column with an improved distributor and in the absence of a distributor.
- 5. To explore the use of multiple distributors in a tubular column.

2.0 INTRODUCTION

A major element of the coal dissolution section of any liquefaction plant is the dissolver. Although a considerable amount of liquefaction will occur in the preheater, a major amount of necessary chemical change will occur in the dissolver, namely sulfur removal, oil and distillate formation and solvent rehydrogenation.

Vertical tubular reactors are employed in all of the major processes currently under consideration for commercial liquefaction of coal. In all of these processes, SRC, EDS and H-Coal, slurry and gas are concurrently fed upward through these vessels. In the EDS and SRC processes, the reactors are essentially empty vessels, whereas for the H-Coal process a bed of ebullating catalyst is maintained in the reactor. The major differences between the EDS, SRC-I and SRC-II processes in dissolver

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operation are the composition of the feed streams and reactants within the dissolver. Other hardware differences such as distributor plates, draft tubes or recycle loops can also cause differences in the behavior of slurries in these vessels. A requirement necessary to any design that will be technically feasible and cost effective is an understanding of the physical behavior of three phase systems in tubular columns.

All of the major processes under development require understanding of backmixed three phase systems. Each process employs at least a portion of its dissolver volume in a backmixed mode. As the design of the 6000 T/D SRC-I plant progresses, the increased vessel size (and other considerations) may dictate the use of reactors in series, which would decrease the overall backmixed characteristic of the commercial plant.

The SRC-I demonstration plant dissolver will represent a considerable scale-up over the Wilsonville and Ft. Lewis dissolvers. The relative sizes of the dissolvers for the two pilot plants mentioned above and the SRC-I demonstration plant are shown below for a residence time of 0.56 hr⁻¹, a gas feed rate of 20 Mscf per ton dried coal, and 38% coal slurry.

Plant	Size <u>Tons/Day</u>	Volume ft ³	Dissolver Diameter	Height <u>ft</u>	Superficial Liquid	Velocities Gas
Wilsonville	6	18.6	12 in	23	.012	0.074
Ft. Lewis (1)	50	106.8	24 in	34	.017	0.10
SRC-I Demo Plant	6000	10454.	11 ft	110	. 06	0.36

The volumes of the reactors are dramatically different as are their diameters. To intelligently make good design decisions, more information is needed on the flow properties of three phase systems in large vessels.

More important from the standpoint of slurry behavior is the difference in gas and liquid superficial velocities. A five-fold velocity difference exists between Wilsonville and the commercial SRC-I design. This difference can have considerable impact on the process because the gas and liquid superficial velocities have a strong effect on (a) gas void volume, (b) actual solids concentration in the dissolver and (c) the

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relative degree of backmixing. As velocity through the dissolver increases, the tendency for solids to remain behind diminishes causing a decrease in the actual concentration of ash particles in the reactor. Those particles that do remain will tend to be larger in size. Since considerable evidence points to a definite catalytic effect of the reactor solids, these larger particles will have decreased surface areas exposed and will likely have diminished catalytic activity. Knowing the particle sizes that can accumulate under commercial flow conditions will give us some indication of size of dissolver solids that should be examined for catalytic activity.

3.0 BACKGROUND

3.1 Gas-Liquid Flow

The behavior of the gas and liquid components in two-phase flow has been studied by numerous investigators (1-28). With the fluid as the continuous phase, the gas flow pattern can be categorized as bubbly flow, slug flow, or churn-turbulent flow. The gas flow pattern is determined primarily by the competing rates of bubble coalescence and breakup, which in turn depend on liquid phase properties such as viscosity, surface tension and density. Bubble coalescence involves the processes of drawing bubbles together with concomitant rupture of the thin film of liquid separating the bubbles. Gas bubbles apparently coalesce by the capture of one bubble in the wake of another rising bubble; this has been extensively studied by Crabtree and Bridgwater (18). Calderbank et al. (24), reported that increasing liquid viscosity enhances the rate of bubble coalescence. Bubble breakup is due to disturbance at the interface. In the early fifties, Taylor (17) performed a theoretical study on the instability of liquid surfaces. This mechanism was later proposed by Clift and Grace (23) to explain bubble breakup.

One of the most important aspects of gas flowing through a tubular reactor is its availability at the reaction site. The transfer of the gas to that site, be it a homogeneous liquid phase transition state or a heterogeneous surface activated state, is ultimately related to the interfacial area between the liquid and gaseous phases. In a two phase gas-liquid system, the interfacial area is related to the volume fraction actually occupied by gas. This volume fraction, which is typically referred to as gas holdup or void fraction, is a value which is extremely important to optimum reactor design.

In a two-phase flow reactor, the gas residence time is governed by the gas holdup at the operating conditions and mass transfer is governed by gas contact at the interfacial phase barrier. Numerous investigators (8, 10, 11, 13, 14) have studied gas holdup with many different systems, covering a wide range of liquid viscosity (0.58-152.0 cp), surface tension (22.3-76.0 dynes/cm), and density (0.79-1.70 gm/cc). Several correlations have been developed to predict the gas void fraction in two-phase flow systems. In general, the gas void fraction is expressed in terms of gas superficial velocity, liquid surface tension, viscosity, and density. A partial list of the existing correlation is given in Table (1).

Generally gas void fraction does not depend on liquid input rate. Gasholdup is usually higher for smaller column diameters because of the wall proximity effect. Ellis and Jones (25) studied the column diameter effect on gas holdup and concluded that, for diameters greater than three inches, gas holdup is independent of column diameter. Directionally, work at Air Products agrees with their result.

Mass transfer in bubble columns has been extensively studied (1, 7, 9, 12, 13, 16, 19, 21, 22, 26). The mass transfer resistance in the gas phase is negligible compared with that in the liquid phase, so that the overall mass transfer coefficient is dominated by that of the liquid phase. In general, the volumetric liquid phase mass transfer coefficient (k_1a) depends on gas input velocity, column diameter, bubble diameter, and liquid properties (diffusivity, surface tension, viscosity, and density). Several correlations have been developed (7, 12, 16, 19, 20, 21, 22, 24). The dependence on liquid velocity and gas properties (density and viscosity) has also been proposed (7, 19, 20) but is not unanimously accepted. Higher gas input rate increases mass transfer through its enhancement of interfacial area and liquid turbulence.

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Another significant aspect of gas-liquid flow is the dispersion (backmixing) of the fluid phase. In studies at Air Products we found that in a gas-liquid flow column an extremely small amount of gas flow can trigger a high degree of dispersion in the liquid phase. Shah, Stiegel and Sharma (15) have made an extensive review of the backmixing in gas-liquid reactors. In general, backmixing in the liquid phase dependson column diameter, gas velocity, and the nature of the gas distributor plate. Most investigators agree that the exial dispersion coefficient is independent of liquid flow rate and liquid phase properties (surface tension, viscosity, and density). Quite a number of correlations have been developed to predict the axial dispersion coefficient in gas-liquid flow reactors. A partial list of the correlations, for gas as the dispersion phase and liquid as the continuous phase, is given in Table (?6).

At low gas velocities, the agreement among these published correlations is poor. For example, from paper calculations for a five-inch diameter column, we have estimated a six-fold difference in the predicted dispersion coefficient at a 0.02 ft/sec superficial gas velocity.

Reference	Low Gas Velocity 0.02 ft/sec)	High Gas Velocity (.33 ft/sec)	
28	.047	. 190	
1	.088	. 204	
2	.022	_ 053	
3 -	. 134	.219	
4	. 129	. 229	
- 6	. 091	. 239	-

Axial Dispersion Coefficients (ft²/sec) from Published Correlations

These correlations give better agreement at high gas velocities. Due to inconsistency, these published correlations fail to provide guidelines to predict the dispersion coefficient, which is an essential value for reactor design.

To anyone initiating studies in this area, experiments in two phase flow should be performed to make certain that experimental techniques are adequate to reproduce existing data. Air Products has performed such studies and has developed standard tech: ques that do reproduce existing data. From these studies a keen understanding of the value of the several correlative models has been gained which will be applied in the proposed program.

3.2 Gas-Liquid-Solid Flow

The addition of solid particles to a gas-liquid flow reactor greatly increases the complexity of the system. Not only the presence of solid itself, but its particle size, its size distribution and its density can affect the behavior significantly. The rate of bubble coalescence increases in the presence of solid particles. Kato et al. (29, 30) visually observed larger coalesed bubbles in a gas-liquid-solid system. However, this enhancement in the rate of bubble coalescence decreases to an insignificant level in a turbulence regime at high gas input velocities. Studies of gas holdup in a three-phase flow reactor (Air Products work to be published) showed a reduction in gas void fraction in the presence of solid particles at low gas velocities. At high gas velocities, the dependence of gas holdup on solid particles disappears. These effects of solid particles on gas holdup are directly in line with the observation of the bubble coalescence rate in the presence of solid particles.

A unique feature of the three-phase flow is the contraction of the fluidized bed (34, 35, 37, 39, 40, 41, 42) with the addition of gas to the system, as was first reported by Turner (34, 35). Empirical expressions have been developed and an iterative method has been employed to solve for the volume fraction of the various phases (37, 39, 41).

Effects of solid particles on liquid backmixing in three-phase flow systems have been studied by several investigators (31, 38). Data of Vail et al. (31) indicate that the presence of solid particles reduces liquid backmixing. The effects of particle size, gas velocity, and liquid velocity are interrelated. An empirical correlation was developed by Kato et al. (29, 30) to predict the dispersion coefficient of the liquid in a slurry. The correlation shows no dependence on particle size and liquid velocity.

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$$E_{ZSL} = \frac{V_{SG} D(1 + 8 Fr_{G}^{0.85})}{13Fr_{G}}$$

 E_{ZSL} = dispersion coefficient of liquid in a slurry (ft²/sec)

Comparison with an earlier correlation (Kato et al. (4)) for liquid dispersion in a gas-liquid system indicates that the liquid phase dispersion coefficient is higher in a slurry than in a clear fluid except when the Froude number is very small. This result is different from the findings of Vail et al.

The dispersion of the solid phase in a gas-liquid-solid system has also been studied by Kato et al. (29). The results lead to two empirical correlations depending on particle size. For small particles the dispersion coefficients for both the liquid and solid phases are identical. For large particles, the empirical condition includes the effect of particle diameter as shown in the following expression.

$$E_{ZS} = \frac{V_{sG} D(1 + 8 Fr_G^{0.85})}{13Fr_G(1 + 0.009 Re_p Fr_G^{-0.8})}$$

 $\begin{aligned} & \operatorname{Re}_{p} = \operatorname{dp} \operatorname{Vp} / \mathcal{Y}_{L} \\ & \operatorname{E}_{ZS} = \operatorname{dispersion} \operatorname{coefficient} \operatorname{of} \operatorname{solid} \operatorname{in} \operatorname{a} \operatorname{slurry} (\operatorname{ft}^{2} / \operatorname{sec}) \\ & \operatorname{dp} = \operatorname{particle} \operatorname{diameter} (\operatorname{ft}) \\ & \operatorname{Vp} = \operatorname{particle} \operatorname{terminal} \operatorname{velocity} \operatorname{in} \operatorname{stagnant} \operatorname{fluid} (\operatorname{ft} / \operatorname{sec}) \\ & \mathcal{Y}_{L} = \operatorname{liquid} \operatorname{kinematic} \operatorname{viscosity} (\operatorname{ft}^{2} / \operatorname{sec}) \end{aligned}$

The suspension of solid particles in a batch column depends on the gas input rate. Roy et al. (36) and Imafuku et al. (32) have studied the critical gas velocities for a complete solid suspension. Critical gas velocity is defined as the minimum gas input rate to fully suspend the solid particles. Imafuku et al. (32) has observed the dependence of the critical gas velocity on the shape of the bottom inlet. A conical bottom is recommended and the position of the gas distributor plate is also important to obtain complete solid suspension. Our experience in three phase flow behavior indicates a lack of pertinent data to define behavior in these systems.

3.3 Solids Accumulation

High solids accumulation has been experienced in the dissolvers at Ft. Lewis and Wilsonville. HRI, when running an SRC study (44), observed solids buildup in their experimental reactor. The most quantitative data on solids accumulation are available from Wilsonville. For over two years their reports have presented extensive data on rates of accumulation, quantities, particle sizes and chemical composition of accumulated materials.

Data for four runs at Wilsonville, each on a different coal, are presented in Table (2) to show the levels of solids that can accumulate. The nighest level of accumulation was in Run 100 with Amax Belle Ayr coal from which 1300 pounds of reactor solids were discharged. This corresponds to a solids concentration of 72 lb/ft^3 . On a volume basis these solids occupy about 40% of the total dissolver space. It is not known whether these solids are completely suspended at this concentration. Obviously, such information is critical for designing a commercial vessel that has a diameter on the order of eleven feet, as in the case of the SRC-I demonstration plant.

In addition to the data from Wilsonville on the composition of these solids, an extensive evaluation of reactor solids was carried out at Pennsylvania State University (45). That study led to an understanding of the mechanism of solids formation. The relative growth of particles was related to the calcium content in the feed coal. As calcium content increased, particle growth and the quantity of accumulated solids also increased.

Considerable data have originated from Wilsonville on the particle size distributions of these recovered solids that accumulated when feeding plant grind coal (95% through 200 mesh). The solids recovered from the reactor show considerable growth as demonstrated by the amounts of material that accumulate on 25 and 50 mesh screens.

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The particle size distribution for the solids collected after Run 68, Table (2) was as follows:

<u>Mesh Size</u>	<u>wt %</u>
25	5
25-50	7
.50-100	5
100-200	50
200-325	24
325	9 .

Obviously the larger particles would not have been in the feed coal. Many other examples in the Wilsonville reports shown even higher concentrations of large particles in the reactor solids.

Since mid-1976 considerable attention has been given to controlling the solids concentration in the reactor at Wilsonville. Addition of reactor solids at the start of individual runs has been regularly practiced to bring the dissolver up to a rapid line-out. More recently a solids withdrawal system was added to maintain a steady solids concentration within the reactor. With the capability to both add and withdraw solids, valuable data will undoubtedly be obtained when operating in this mode. This experience has and will continue to provide excellent information for the commercial plant design. However, additional data at higher flows and with different geometries will be needed to use such a system effectively on a larger scale.

Accumulation of solids at commercial design conditions will almost certainly occur. Exxon's results on 8 mesh feed coal showed the necessity of a solids removal system (46). At the recent DOE Project Review Meeting on Preheater Design held at Oak Ridge on 21 March 1979, C. Ackerman, P & M Mining Co., reported 1/16-1/8 inch rock accumulation in the Ft. Lewis dissolver after only 3-1/2 days operation with 1/8" coal feed. From data presented in Section 1.1 on the Ft. Lewis reactor design and data in Table 1.1 from Run 68 at Wilsonville, the relative

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superficial gas velocities are very similar, namely, 0.11 and 0.09 ft/sec, respectively. At these conditions, solids that accumulate when using plant grind coal have particle sizes less than 100 mesh. Certainly much higher levels of accumulation would be expected with the larger coal feed size planned for the commercial plants.

The relevance of the above data at superficial gas velocities of 0.1 ft/sec is apparent when the alternate option of running the 6000 T/D dissolver at 0.18 ft/sec (parallel dissolvers) is a distinct possibility. Knowing the behavior of solids in a column at these flow velocities is good engineering practice.

Very little data on the retention of solids in dissolvers are available from other sources. Ft. Lewis data have been largely limited to the time that a considerable plug was discovered in their dissolver (48). A systematic study on the behavior of solids in their system has not been performed.

In the H-Coal process a considerable effort has apparently been applied to understanding the flow rates that allow retention of solid catalysts within their reactor. However, the catalyst particles used in the H-Coal process are larger than the ash particles anticipated to accumulate in the SRC dissolver. The H-Coal process will likely use a 1/16" extrudate for their commercial design.

Particle growth within the dissolver is apparently the cause of the large accumulation of solids having particle sizes greater than those present in the feed coal. The program at Pennsylvania State University has shown that the particles can be explained by a shell growth effect.

In order to get an initial understanding of the problem of solid accumulation, critical gas velocities were determined at Air Products for different particle sizes in three liquid media using a 5" diameter column. Critical gas velocity is defined as the superficial gas velocity beyond which the solid particles will be in complete suspension.

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The results of the critical gas velocity study are summarized in Table (5). The particle size effect on solids suspension or critical gas velocity is significant; the critical gas velocity is substantially increased with increasing particle size. When the gas velocity dropped below the critical value, sand particles were observed to settle to the bottom with the gas channeling through the settled bed. The amount of settled solids decreased with increasing gas velocity. As the critical gas velocity was reached the momentum transferred from the gas phase was large enough to suspend all the sand particles.

Also shown in Table (5) was the sieve opening (microns) for each particle range. Assuming spherical particles having diameters equal to the sieve opening, the settling velocities of sand particles calculated based upon particle drag in an infinite water medium would be as shown in Table (4). It is reasonable to assume that the largest particle, namely the maximum sieve opening in each group, governs the critical gas velocity. It is then interesting to note that the critical gas velocity in each group (Table (5)) is approximately twice the settling velocity of the largest particle of the corresponding group (with the exception of Group II). Extrapolating these results, the Group VI particles (20-30 mesh) are predicted to have a critical gas velocity in the neighborhood of 0.7 ft/sec. In any event, the results of this critical gas velocity study strongly suggest the problem of settled solids when large particles were used.

The physical properties of liquid has a significant impact on the solid suspension as shown in Table (5). When the mixture of ethanol and water was used, the critical gas velocities for Groups II and V were reduced by approximately 30 percent of those measured in pure water. On examining the physical properties of these two solutions (Table (3)), two possible causes for this reduction are apparent, namely the surface tension and viscosity. The experiment with pure ethanol eliminates the possibility of surface tension effect however. The surface tension of the mixed solution was experimentally determined to be 37.43 dynes/cm which was more than a factor of two lower than that of pure water. If liquid surface tension was responsible for the observed reduction of

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the critical gas velocity in the mixture, then pure ethanol, which has a face tension (22.75 dynes/cm) lower than that of the mixture, should show a similar reduction. Surprisingly, our results showed that the critical gas velocity for the 80-120 mesh solids in pure ethanol was indistinguishable from that observed in pure water, as shown in Table (5). Hence, liquid surface tension is not responsible for the reduction effect observed in the mixture, thereby leaving the liquid viscosity as the sole explanation.

The solid settling velocity was lower in the mixed solution than in pure water because the viscosity between these two solutions differed by a factor of three. Going through the exercise of particle drag calculation, the settling velocity of a 30 mesh (595 microns) particle in the mixed solution and the pure ethanol were found to be 0.13 ft/sec and 0.24 ft/sec, respectively. The insignificant difference between pure ethanol and pure water clearly explains the indistinguishable critical gas velocities measured in those two solutions. On the other hand, the two fold difference in the settling velocities of the 30 mesh particle in mixed solution and pure water explain the reduction in critical gas velocity that was observed. This also agreed with the findings on particle size effect which showed that critical gas velocity increased with increasing particle settling velocity. As a rough rule of thumb the ratio of the critical gas velocity to the particle settling velocity is about 2 in the absence of liquid flow.

Table (6) presents the operating conditions for the SRC-I pilot and demonstration plants. Results presented in Table (5) clearly show that under demonstration plant conditions, solids smaller than 30-45 mesh would not be expected to accumulate, while both Wilsonville and Ft. Lewis would accumulate solids. The actual distribution of mesh sizes is indeterminate since larger particles displace smaller ones and any size larger than 80-100 mesh may accumulate at Wilsonville. That this is clearly so at Wilsonville has been reported.

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3.4 Data Needs for Dissolver Design

The design goal for the commercial dissolvers is to ensure that solids passing through these reactors remain in suspension in sufficient quantity and distribution to give the necessary catalytic effect. These solids and ash particles can accumulate over an extended operating period. The recent data from Ft. Lewis on rock accumulation in the dissolver when feeding 1/8 inch coal is evidence of the problem that can occur commercially. From a design standpoint this result is quite alarming since Tarrer and coworkers have shown that coal fed to the process unit would immediately dissolve into liquid leaving extremely small particle ash suspended in the reactor.

- Determine the necessary superficial gas and slurry velocities to suspend these particles at turndown velocities.
- Determine the solids sizes necessary to give adequate solids accumulation within the reactor at commercial linear liquid and gas velocities.
- Determine adequate slurry correlations to predict accumulations in the commercial reactors, especially for start-up mode and equilibrium operation after solids removal.
- Determine dispersion (backmixing) as a function of fluid and gas velocities, solids concentration, density and particle size.
- Determine in a large system the effect of surface tension on gas void fraction and solids suspension.
- Determine factors that influence adequate solids removal.
- Determine the influence of distributor plates on fluid properties as a function of position relative to the plate.

- ^o Determine the solids removal efficiency with regards to plate design and location.
- Examine the solids removal as a function of velocity through the draw-off line and its=affect on dissolver behavior.

With the start of Air Products' "Phase O" design of a 6000 T/D SRC-I demonstration plant, the necessity for larger scale cold-flow simulator studies were realized. It was decided that a glass column twenty-five feet tall with a 12-inch internal diameter would have the required material strength and would afford maximum visual access to the behavior of the fluid within the column. This design also corresponds exactly to the SRC-I dissolver in use at Wilsonville.

4.0 TECHNICAL PROGRESS

The overall objective of this project is to study solids accumulation, liquid dispersion, gas-liquid mass transfer, and gas holdup in gas-liquid and gas-liquid-solid systems in cold-flow tubular columns. Under this contract, work will be conducted in a 5-inch diameter by 5-foot tall Plexiglas column and a 12-inch diameter by 25-foot tall glass column available at the contractor site. The configuration and operational details of both columns are described in the report.

This report contains a large number of figures and tables which give the experimental results for this reporting period. In order to maintain clarity and continuity of the text, these figures and tables are added to the end of the report.

4.1 Task 1. - Experimental Planning

Seven tasks are planned for this 2-year DOE-APCI contract. The specific objectives and duration of each task are discussed in the Experimental Plan attached in the appendix. Specific experimental plans for the entire program are covered in the plan.

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4.2 Task 2. - Two and Three Phase Flow In Vertical Columns

The objectives of this task are:

- o to study gas holdup and liquid dispersion in two and three phase systems
 - ° to study gas-liquid mass transfer
 - to study solid dispersion

All of the experimental work during this quarter was related to this task. The experimental program proceeded at an expected rate for October and November. During December, several experiments were repeated to ensure data reproducibility. In our program we had not anticipated the necessity nor the problems associated with sand recovery. Sand recovery is necessary for the following reasons: determination of sand inventory in the column and the effort necessary to sift sufficient quantities of specific sized Tractions for our experiments. These time losses could possibly be recovered by operating both columns simultaneously in some experiments for the next few months.

5.0 EXPERIMENTAL SECTION

5.1 Cold Flow Model Equipment

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

5.1.1 5-Inch Diameter Column

A schematic of the 5-inch x 5-foot 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 typicar 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 inch 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 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 cc syringe was also attached to the column at the bottom in order to inject tracer into the feed stream for dispersion studies.

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5.1.2 12-Inch Diameter Column

A schematic of the 12-inch 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-inch diameter column. The column was comprised of five sections of 5-foot glass tubing interconnected with machined metal flanges. The column rested on a one-inch thick stainless steel plate which was supported on scaffolding which 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 inlex 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 cc tube was attached to the column at the bottom for injecting tracer during

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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 (half-inch openings) to protect personnel from flying glass in case of rupture.

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5.2 Experimental Procedures

The experimental procedures described below are essentially the same for both the 5" and 12" columns. The only difference in operating procedures between the two columns will be the quantity of tracer injected into the columns for liquid dispersion experiments. Since all the experiments in this quarter were conducted in the 12" column, the procedures are described for the 12" column. The ranges of the variables were chosen to include the operating conditions for the SRC-I pilot and demonstration plants.

5.2.1 Gas Holdup

Gas holdup was measured in the 12-inch diameter column in both the absence and the presence of fluid flow. Effects of solid particles on gas holdup were also investigated in both modes of fluid motion.

In the absence of liquid flow, the experiments were performed by completely filling the column with liquid and then passing air through the liquid at specified rates. Excess liquid exited the column at the top through a side opening. A waiting period of 5 minutes was allowed to ensure that steady state was achieved. The bottom valve was then closed to shut off the gas input. The final liquid level was measured, and the difference between the initial and the final levels represented the gas holdup at that particular gas flow rate. A total of 12 gas flow rates, ranging from 0.036 ft/sec to 0.368 ft/sec, were studied. With fluid flow, the liquid and gas passed into the column through a centrally located opening at the bottom. Excess liquid exited the column through a side opening at the top. After 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 above.

Both 20-30 mesh (841-595 microns) and 100 mesh minus (less than 149 microns) sand particles were used to investigate the effect of solids on gas holdup. Either 98 lb (equivalent to a solid concentration of 5 lb/ft³ or 8 wt%) or 250 lb (equivalent to 12.7 lb/ft³ or 18 wt%) of each type of sand were initially placed into the column. The experiments proceeded as described above for both the presence and absence of liquid flow. In the case of liquid flow, some sand particles were carried throughout the column by the fluid phase, and were collected to account for an average solid concentration during the run.

5.2.2 Liquid Dispersion

Liquid phase dispersion in water/air and sand/water/air systems was investigated in a 12-inch diameter column using a tracer detection method. Sodium chloride was used as the tracer and a conductivity probe mounted at the exit line of the column monitored the ion conductivity of the solution continuously. Liquid flow rates were measured periodically to ensure a steady-state flow. An ultrasonic deppler flow meter was also used to check for fluctuations in the liquid flow rate. Gas flow rate was monitored using a flow meter which was pre-calibrated with a dry-test meter.

In the typical experimental procedure liquid and gas was passed through the column at the desired rates. The signal from the conductivity probe was monitored using a recorder. After establishing a steady base line on the recorder, a pulse of 400 cc concentrated NaCl solution (0.243 gm/cc) was injected into the column at the bottom. Typical injection time was between 2 and 5 seconds. The conductivity of the exiting 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 whereas water flow continued for 5-10 minutes to ensure the column had been purged of residual salt before starting the next experiment.

When solid particles were used, a slightly different procedure was followed. To determine the effects of solids on liquid dispersion, a constant concentration of solids should 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 In order to perform just one slurry pumping experiment, ວວໄນຫກ. more than one ton of sand would be required to prepare a slurry feed that contained 20 lb/ft³ 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 to maintain the desired solids concentration in the column that eliminated the massive amount of material. required. The column was partially filled with water first. 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 that no sand could exit the column during this slow filling process. Slight amounts of sand left the column during the time to achieve steady state and to complete the experiment however. The inventory of the solid concentration in the column-during the experiment was maintained by monitoring the amount of sand that exited the column.

Conductivity versus time plots 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. This axial dispersion model was based on the analogy between mixing in the actual flow and a diffusional process. A computer model was 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.

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5.0 RESULTS AND DISCUSSION

Prior to this contract, considerable work had been done at Air Products in a 5-inch diameter column to measure gas holdup and liquid axial dispersion coefficients. Experimental uncertainties were estimated during the course of that investigation. These uncertainties were used as guidelines in discussing the results presented in this report. Experimental precision for the 12" column will be estimated and presented in a subsequent report.

6.1 Gas Holdup

Particle size, solid concentration, settled versus suspended solids and gas rate were found to affect gas void fraction. Two different particle sizes were used in these studies, namely 20-30 mesh and less than 100 mesh. Under the experimental conditions studied, the smaller particles (less than 100 mesh) were in complete suspension whereas settled solids were present in experiments involving the 20-30 mesh particles.

Solid concentrations represent average values for the column. With a completely suspended system the solids concentration along the column varies in a gradual manner. When settled solids are present, the specific solids concentration at the bottom of the column can be quite high while at the top the specific concentration in some cases can approach zero.

Realizing these characteristics of the experimental system, data relating the effect of solid particles on gas holdup were obtained. Measurements were made both in the absence and presence of liquid flow.

6.1.1 No Liquid Flow

The effect of solid particles on gas holdup in the absence of fluid flow is shown in Figure (3). Particle size and solid concentration were the two variables used in this study at several different gas rates. These data with solids are compared in Figure (3) with results from the two-phase flow studies conducted earlier. At low superficial gas velocities (up to 0.10 ft/sec) the presence of solids did not change the gas holdup appreciably. However, at high gas velocities the presence of solids decreased gas holdup.

Gas holdup was also affected by solids concentration under certain conditions. In the presence of small particles, namely the minus 100 mesh sand, gas holdup decreased, compared to the 2-phase system, as gas velocity and solids concentration increased. A 13.9% reduction was measured for a solids concentration of 11 lb/ft³ at a superficial gas velocity of 0.368 ft/sec.

The larger particle size solids decreased gas holdup only at the highest gas velocity at high solids concentration (12.7 lb/ft^3). The magnitude was less than for the smaller size material. At the lower concentration no change was observed.

The gas holdup was influenced by particle suspension in this flow regime. The 20-30 mesh sand in this work was largely settled at the bottom of the 12-inch diameter column. They showed less reduction in gas holdup than the 100 mesh minus suspended particles. It was possible that the suspended particle enhanced the coalescence of gas bubbles more effectively than the settled solids, thereby resulting in a larger reduction in gas holdup. Therefore, the effect of increasing solid concentration could be related to an increase in bubble coalescence. In any event, with the exception of the fine particles at high concentration the presence of solids did not have a significant effect on gas holdup. As shown in Figure (3) the results (with and without solids) are reasonably described by the correlation of Yoshida and Akita (26) presented in Table 1.

Since large accumulation of fine particles is not likely to occur under the high flow rates in the actual liquefaction process, the correlation of Yoshida and Akita should have significant accuracy for estimating gas holdup.

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6.1.2 Liquid Flow

Liquid velocity had no effect on gas holdup in gas-liquid two-phase flow as demonstrated in Table (7). One order of magnitude variation in liquid velocities showed no change in gas holdup at each of the four gas flow rates which were also varied over one order of magnitude. Also included in Table (7) are the values obtained in the absence of fluid flow; these values again showed no difference from those measured at different liquid velocities.

The effect of fluid flow on gas holdup are also investigated for the three-phase flow system at different particle sizes and solid concentrations. The gas holdup results for the small particle sand/water/nitrogen system at two solids concentrations, namely 5 lb/ft^3 (8 wt%) and 11 lb/ft³ (16 wt%), are summarized in Tables (8) and (9), respectively. Within experimental accuracy, liquid velocity had no effect on gas holdup in the presence of fine particles. A slight effect of fluid flow in the presence of larger particle settled solid was observed as shown in Tables (10) and (11). At low gas velocities, little change was observed over an order of magnitude variation in liquid velocity. A small consistent decrease in gas holdup with increasing liquid velocity was measured at high gas velocities for both 5 1b/ft³ and 12.7 lb/ft³ of 20-30 mesh sand particles. The cause of this reduction in gas holdup may be the combination of fluidization of the settled solid bed and the high gas turbulance which facilitated gas channeling through the solid bed. Several more gas holdup measurements at substantially higher gas and liquid velocities are needed to confirm this reduction in gas holdup. Nevertheless, liquid velocity had no effect on gas holdup either in the absence of solid, in the presence of fine particles at all flow conditions, or in the presence of settled solid at low gas velocity regime.

6.2 Liquid Dispersion

Liquid dispersion experiments were performed in the 12-inch diameter column for gas/liquid and solid/gas/liquid systems. Table (12) lists the ranges of variables studied during this reporting period.

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The fits of all the experimental tracer curves with the theoretical curves from axial dispersion model are displayed in Figures (5) to In these curves, the circles represent the experimental tracer (34). curve while the solid line represents the theoretical curve predicted by the axial dispersion model. The matching of experimental and theoretical curves was found to be sensitive to column height. Since the dispersion number ($\frac{D}{uL}$, a dimensionless quantity) is inversely proportional to the height of the column, determination of the dispersion number by the best fit technique became more difficult with increasing column height. The curve matching technique was extremely sensitive to any tailing in the curve. Since some fits were less perfect than others due to tailing problems, a consistent method of selecting the dispersion numbers was developed. Dispersion numbers were always determined by fitting the peak time (peak position) of the tracer curve with the dispersion model because peak time was not affected by tailing in the experimental curve. Tailing only changed the height of the As long as the mean residence time could be measured accurately, curve. peak position was the best way to match dispersion numbers.

Fluctuations in liquid flow could cause errors in the determination of mean residence time if averages of several discrete measurements were used. Liquid flow measurements were modified during this quarter to improve accuracy. In addition, mean residence times, which were calculated using the experimental tracer curve, were compared with the values calculated using average flow rates. Whenever a large (greater than 10%) discrepancy existed between the two numbers, the residence time calculated using the experimental tracer curve was used to generate the tracer curves. This procedure also resulted in improvement of data analysis.

Table (13) lists the axial liquid dispersion coefficients and the operating conditions for all the experiments conducted in this quarter. The axial dispersion coefficients were not reported for those experiments in the absence of gas flow. The tracer curves in the absence of gas flow (Figures 14, 25 and 30) approached plug flow regime. The curves rose steeply; the peak times were very close to a dimensionless time of unity. However, tailing problems existed in the experimental tracer curves and could not be handled by the axial dispersion model.

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The effect of liquid velocity on axial liquid dispersion coefficient is shown in Table (14) for the three solid concentrations (0, 5 and 20 lb/ft^3). The data were taken at a constant gas velocity of 0.327 ft/sec while the liquid velocity ranged from 0.009 to 0.059 ft/sec. Table (14) clearly shows that in the absence of solids, liquid velocity had no effect on axial liquid dispersion coefficient. Similar results were observed at the other gas velocities (0.05 and 0.194 ft/sec) for experiments with no solids. At low concentration of solids (5 lb/ft^3), the axial dispersion coefficient was independent of liquid velocity. However, at the high concentration of solids, liquid velocity seemed to alter the dispersion coefficient. Also, at this high solid concentration, large discrepancies existed between predicted and theoretical curves. The particle size of the solids used in these experiments was large (20/30 mesh). These particles were not in complete suspension since a portion of the solids remained settled at the bottom of the column. The settled solids at high_concentration could possibly be the cause for the observed discrepancies. Results from runs using smaller particle sizes are necessary to arrive at reasonable conclusions about the effect of liquid velocity on axial dispersion coefficient at high solid concentrations.

The effect of gas velocity on liquid phase dispersion is shown in Table (15) and Figure (4) at various solid concentrations. Gas velocity had a definite influence on axial liquid dispersion coefficient. Dispersion coefficient increased with increasing gas velocity as shown in Table (15). However, the rate of increase seemed to depend on the solid concentration as shown in Figure (4). The rate of increase decreased with increasing solid concentration. These results will be discussed in detail in the next quarterly report after results from runs using smaller particle sizes become available.

Most correlations available in the literature assumed that liquid flow rate had no influence on liquid dispersion coefficient which was correlated as functions of gas flow rate and column diameter. A partial list of the correlations is shown in Table (16). The comparison of the liquid phase dispersion coefficients obtained from the 5-inch diameter column at three different gas velocities with the predicted values from these

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correlations are shown in Table 17. This work was done prior to the initiation of the current contract. At high gas velocity, with the exception of the value predicted from Cova's correlation (2), the correlation showed reasonable agreement with our experimental value. The dispersion coefficient measured at a superficial gas velocity of 0.09 ft/sec agreed excellently with Tower and Ackerman's correlation (9) as shown in Table (17). However, this value was 30% lower than the other predicted values. At low gas velocities, the agreement among the published correlations is poor. The prediction from these six correlation. In general, our experimentally determined dispersion coefficients are less than the predicted values.

Comparisons of the experimental liquid dispersion coefficients, obtained from the 12-inch diameter column at various gas velocities and solid concentrations, with the predicted values from the correlations are shown in Table (18). One should note that none of these correlations were developed to account for the presence of solids. Baird and Rice's correlation was quite good at low velocities but failed to predict at high gas velocities. These results indicated that a better correlation was needed to account for the effect of solid particles on dispersion coefficient. Also, an investigation of whether the presence of settled solids affects liquid dispersion coefficient differently than suspended solids should be conducted. Results from experiments using smaller particles might be able to answer some of these questions.

7.0 FUTURE WORK

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In the next quarter, liquid dispersion experiments will be completed for particle sizes smaller than 20/30 mesh. Modifications to the $12^{\prime\prime}$ column will be made to enable us to withdraw samples at various heights of the column. Solid dispersion experiments will be initiated in the 5" column to guide us in designing meaningful experiments in the $12^{\prime\prime}$ column.

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In subsequent quarters, experiments will be conducted to study the effect of distributors. Planning will be done to safely initiate experiments with organic liquids to study the effect of physical properties of the liquid on holdup, liquid dispersion and solids accumulation.
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TABLE (1)

Correlations for Predicting Gas Holdup

° G. A. Hughmark (16)

$$g = \frac{V_{sG}}{U_s} \left[\left(\frac{62.4}{C_{\perp}} \right) \left(\frac{72}{C_{\perp}} \right) \right]^{1/3}$$

- ^a Akita and Yoshida (26) $\frac{\mathcal{E}_{g}}{(1 - \mathcal{E}_{g})^{4}} = C_{1}^{-} (N_{Bo})^{1/8} (N_{Ga})^{1/12} (N_{Fr})$
- Nikita and Kikukawa (3)

$$\epsilon_{g} = 0.505 V_{sG}^{0.47} (72/\sigma)^{2/3} (1/\mu)^{0.05}$$

Where \mathcal{E}_{g} = gas void fraction V_{sG} = gas superficial velocity (ft/sec) U_{s} = slip velocity (ft/sec) \mathcal{C}_{L} = liquid density (lb/ft³) \mathcal{T} = liquid surface tension (dynes/cm) $N_{g_{0}}$ = gD² $\mathcal{C}_{L}/\mathcal{T}$ (Bond number) N_{Ga} = gD³ \mathcal{D}_{L}^{2} (Galileo number) $N_{Fr} = V_{sG}/\sqrt{gD}$ (Froude number) $C_{1} = 0.20$ for nonpolar solutions, 0.25 for polar solutions D = diameter of column (ft) \mathcal{V}_{L} = liquid kinematic viscosity (ft²/sec) g = gravitational acceleration (32.2 ft/sec²) \mathcal{M} = liquid viscosity (cp)

Coal	Run Length (Days)	LHSV in Diss <u>o</u> lver (hr)	Superficial (ft. Slurry	Velocities /sec) Gaș	Solids in Beactor (lb/ft)	% Ash in "Solids
Kentucky 9 & 14	68	1.7	0.011	.09	33	83
Monterey	95 [°]	1.7	0.011	. 05	22	70
Indiana V	129	0.9	0.006	. 03	53	61
Amax Bell Ayr	100	1.1	0.007	. 06	72	71

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TABLE (2)

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Wilsonville Solids Accumulation

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TABLE (3)

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Physical Properties of Various Liquids Used In The Critical Gas Velocity Experiments

	Physical Properties			
Liquid Identity	Surface Tension (dynes/cm)	Viscosity (cp.)	Density (gm/cm ³)	
Water	72.8	1.0	1.000	
30 wt% ethanol- 70 wt% water	37.43	3.2	0.9556	
Ethanol	22.75	1.2	0. 7893	

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Mesh Number	Diameter, <u>Mm</u>	Settling Velocity, Ft/Sec
80	177	0.0567
50	250	0.0971
45	354	0.1535
30	595	0.2592
20	841	0.3707

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Particle Settiing Velocity in Infinite Water Medium

TABLE	(5)
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Effects of Particle Size and Liquid Properties On Critical Gas Velocities

	Particle Size		Critical Gas Velocities (ft/sec) at Various Liquid Phases		
Group Number	Mesh	∫L m	Water	Ethanol-Water*	Ethanol
I	140 minus	less than 105	0.013		
II	80-120	177-125	0.137-0.165	0.10-0.11	0.137-0.165
III	60- 80	250 - 177	0.193-0.217		
IV	45- 60	345-250	0.298-0.362		
V	30- 45	595-354	0.482-0.503	0.344	
VI	20- 30	841-595	No full suspension detected up to 0.57 ft/sec gas velocity.	 :	

*30 wt% ethanol - 70 wt% water mixture

	<u>Wilsonville</u>	<u>Ft. Lewis</u>	Demonstration	
Liquid Velocity (ft/sec)	0.006	0.012	0.05	
Gas Velocity (ft/sec)	0.040	0.080	0.35	
Liquid Viscosity (cp)	1.0	1.0	1.0	

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TABLE (6)

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Operating Conditions of SRC-I Pilot and Demonstration Plants

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Superficial Gas Velocity (ft/sec)	Superficial Fluid Velocity (ft/sec)	Gas Void Fraction $(\boldsymbol{\epsilon}_{g})$
	ZERO	0.026
	0.0059	0.027
0.036	0.0239	0.027
	0.0356	0 .029
	0.0575	0.028
	ZERO	0.071 ^(a)
	0.0059	0.075
0.084	0.0230	0.074
	0.0358	0.072
	0.0575	0.072
	ZERO	0.132
	0.0052	0.134
0.194	0.0251	0.131
	0.0386	0.129
	0.0554	0.127
	ZERO	0.173
	0.0080	0.176
0.368	0.0219	0.170
	0.0380	0.171
	0.0542	0.168

TA	BL	E	C	7)
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Effect of Fluid Flow on Gas Holdup in the Absence of Solids

(a) interpolated value

TABLE (8)

(8	8 wt%) 100 Mesh Minus Sand	
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Superficial Gas Velocity	Superficial Flui <u>d</u> Velocity	Gas Void Fraction
(ft/sec)	(ft/sec)	$(\boldsymbol{\epsilon}_{g})$
	ZERO	0.025
	0.0062	0.024
0.036	0.0212	0.025
	0.0369	0.025
	· 0.0548	0.026
	ZERO	0.070 ^(a)
	0.0039	0.070
0.084	0.0276	0.069
	0.0444	0.068
	0.0729	0.067
	ZERO	0.128
	0_0051	0.122
0.194	0.0291	0.122
	0.0444	0.122
	0.0861	0.120
	ZERO	0.165
	0.0108	0.162
0.368	0.0373	0.164
	0.0536	0.162
	0.0713	0.162

Effect of Fluid Flow on Gas Holdup in the Presence of 5 lb/ft³

(a) interpolated value

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TABLE (9)

Effect of Fluid Flow on Gas Holdup in the Presence of 11 1b/ft ³				
<u></u>	weby 100 mesh minus said			
Superficial Gas Velocity (ft/sec)	Superficial Fluid Velocity (ft/sec)	Gas Void Fraction ((g)		
	ZERO	0.027		
	0.0033	0.023		
0.036	0.0192	0.023		
	0.0326	0.025		
<u></u>	0.0515	0.025		
	ZERO	0.068 ^(a)		
	0.0045	0.059		
0.084	0.0197	0.060		
	0.0340	0.062		
	0.0509	0.064		
	ZERO	0.116		
	0.0040	0.106		
0.194	0.0195	0.105		
	6.0341	0.107		
	0.0519	0.108		
	ZERO	0.149		
	0.0055	0.142		
0.368	0.0195	0.144		
-	0.0329	0.148		
	0.0513	0.152		

(a) interpolated value

.

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TABLE (10)

Effect of Fluid Flow	(8 wt%) 20-30 Mesh Sand	sence un 5 Ib/IL
Superficial Gas Velocity (ft/sec)	Superficial Fluid Velocity (ft/sec)	Gas Void Fraction (Cn)
		<u> </u>
	ZERO	0.029
	0.0053	0.026
0.036	0.0203	0.024
	0.0354	0.024
	0.0564	0.025
	ZERO	0.046
	0.0055	0.045
0.056	0.0210.	0.042
	0.0355	0.042
	0.0552	0.043
	ZERO	0.071 ^(a)
	0.0056	0.069
0.087	0.0216	0.067
· .	0.0348	0.068
•	0.0543	0.066
	ZERO	0.132
:	0.0076	0.126
0.194	0.0208	0.122
	0.0358	0.120
4	0.0563	0.119
······································	ZERO	0.174
	0.0073	0.167
0.368	0.0215	0.162
	0.0355	0.164
	0.0527	0.160

Effect of Fluid Flow on Gas Holdun in the Presence of 5 lb/ft³

(a) interpolated value

-39-

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Effect of Fluid Flow on Gas Holdup in the Presence of 12.7 1b/ft³ (18 wt%) 29-30 Mesh Sand Gas Void Fraction Superficial Superficial Gas Velocity Fluid Velocity (**€**g) (ft/sec) (ft/sec) ZERO 0.026 0.0043 0.025 0.036 0.0197 0.025 0.0343 0.023 0.0559 0.024 0.066^(a) ZERO 0.0050 0.066 0.084 0.0212 0.061 0.0348 0.060 0.0540 0.060 ZERO 0.121 -_____ 0.0058 0.118 0.194 0.0215 0.106 0.0347 0.105 0.0542 0.105 ZERO 0.162 0.0068 0.157 0.358 0.0201 0. 146 ::::··· 0.0361 0.148 0.0513 0.145

TABLE (11)

(a) interpolated value

TABLE (12)

LIQUID DISPERSION - TWO AND THREE PHASE FLOW

VARIABLES STUDIED

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LIQUID VELOCITY	-	0.01-0.05 ft/sec
GAS VELOCITY	-	0.0-0.327 ft/sec
PARTICLE SIZE	-	20/30 MESH
SOLIDS CONCENTRATION	- -	5 and 20 1b/ft ³

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TABLE (13)• EXPERIMENTAL DATA

						Axial
	Liquid	Gas	Particle	Solids		Dispersion
	Velocity	Velocity	Size	Concentration	Dispension	Coefficients
Run Number	ft/sec	ft/sec	Mesh	lb/ft ³	Number	ft ² /sec
XIII - 1	0.0489	0.327		0.0	0.50	0.617
- 2	0.0505	0.194		0.0	0.39	0.497
- 3	0.0410	0.194		0.0	0.47	0.486
- 4	0.0302	0.194		0.0	0.65	0.495
- 5	0.0199	0.194		0.0	1.00	0.502
- 6	0.009 9	0.194		0.0	2.00	0.500
- 7	0.0506	0.097		0.0	0.38	0.485
- 8	0.0499	0.050		0. 0	0.32	0.403
. ~ 9	0.0048	0.050		0.0	3.2	0.405
- 10	0.0503	0.0		0.0	Very Low	Very Low
- 11	0.0097	0.327	·	0.0	2.5	0.612
- 12	0.0190	0.327	20/30	5.0	1.00	0.479
- 13	0.0400	0.327	20/30	5.0	0.47	0.474
- 14	0.0598	0.327	20/30	5.0	0.32	0.482
- 15	0.0092	0.327	20/30	5.0	2.20	0.511
- 16	0.0295	J. 327	20/30	5.0	0.65	0.483
XIV - 1	0.0536	0.050	20/30	5.0	0.22	0 .297
- 2	0.0541	0.097	20/30	5.0	0.25	0.341
- 3	0.0535	0.194	20/30	5.0	0.27	0.364
- 4	0.0533	0.327	20/30	5.0	0.35	0.471
- 5	0.0534	0.0	20/3 0	5.0	Very Low	Very Low
XV - 1	0 .0548	0.050	20/30	20.0	0.22	0.304
- 2	0.0532	0.097	20/30	20.0	0.22	0.295
- 3	0.0538	0.194	20/30	20.0	0.27	0.366
- 4	0.0497	0.327	20/30	20.0	0.30	0.376
- 5	0.0537	0.0	20/30	20.0	Very Low	Very Low
- 6	0.0404	0.327	20/30	20 .0	0.35	0.356
- 7	0.0285	0.327	20/30	20.0	0.79	0.503
- 8	0.0255	0.327	20/30	20. 0	0.60	0.385
- 9	0.0130	0.327	20/30	20 .0	0.60	0.196

TABLE (14)

EFFECT OF LIQUID VELOCITY ON AXIAL LIQUID DISPERSION COEFFICIENT

Gas Velocity = 0.327 ft/sec Particle Size = 20/30 mesh

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Liquid Velocity, ft/sec <u>+</u> 0.001	Axial Lie	quid Dispersion [®] Coefficient, Solid Concentration, lb/ft ³ 5.0	ft ² /sec 20.0
0.009	0.612	0.571	
0.013			0.196
0.025			0.385
0.029		0.483	0.503
0.040		0.474	0.356
0.049	0.617		0.376
0.053		0.471	
0.059		0.482	

TABLE (15)

EFFECT OF GAS VELOCITY ON AXIAL LIQUID DISPERSION COEFFICIENT

Liquid Velocity = 0.052 ⁺ 0.003 ft/sec Particle Size = 20/30 mesh

Gas Velocity, ft/sec	Axial Liqui So	d Dispersion Coefficient, lid Concentration, lb/ft ³	ft ² /sec
	0.0	5.0	20.0
0.05	0.405	0.297	0.304
0.097	0.485	0.341	0. 29 5
0.194	0.495	0.364	0.366
0.327	0.614	0.471	0.376

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TABLE (16)

CORRELATIONS FOR PREDICTING AXIAL DISPERSION COEFFICIENTS

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Kato and Nishiwaki⁽⁴⁾

$$E_{ZL} = \frac{V_{GD} (1 + 6.5 \text{ Fr}_{G}^{0.8})}{13 \text{ Fr}_{G}}$$

Towel and Ackerman⁽²⁸⁾

$$E_{ZL} = 1.23 \text{ p}^{1.5} \text{ v}_{G}^{0.5}$$

Cova⁽²⁾

$$E_{ZL} = 0.344 V_G^{0.32} 2^{0.07}$$

Deckwer, et al⁽¹⁾

$$E_{ZL} = 0.678 \text{ D}^{1.4} \text{ V}_{G}^{0.3}$$

Hikita and Kikukawa⁽³⁾

$$E_{ZL} = (0.15 + 0.69 V_G^{0.77}) 0^{1.25} (1/\mu_1)^{0.12}$$

Baird and Rice⁽⁶⁾

$$E_{ZL} = 0.35 D^{4/3} (V_{G}g)^{1/3}$$

Ying⁽⁴⁹⁾

$$E_{ZL} = 0.27 D V_{G} \left(\frac{Dg}{V_{G}^{2}}\right)^{0.32}$$

Where
$$Fr_G = Froude Number = V_G^2/gD$$

 $E_{ZL} = axial dispersion_coefficient, (ft^2/sec)$
 $V_G = gas superficial velocity (ft/sec)$
 $D = column diameter (ft)$
 $\sum_{l=1}^{d} 2 = liquid density (gm/cm^3)$
 $\mu_L = viscosity of liquid (cp.)$
 $g = acceleration of gravity (ft/sec^2)$

				TABLE (1)	7)			
	Compar	ison of Axial	Dispersio	n Coefficien	ts in Liquid P	hase for the	5" Column	
		Predicte	Experimental Axial Dispersion Coefficient (ft ² /sec)					
las Velocity (ft/sec)	Kato & Nishiwaki	Towel & Ackerman	Cova	Deckwer	Hikita & Kikukawa	Baird & Rice	C _S = Zero	C _S = 16 wt% P _S = 30/45 mesh
0.02	0.129	0.047	0.022	0.088	0.134	0.091	0.0663	0.0442
0.09	0.157	0.155	0.035	0.138	0.158	0,155	0.0909	0.0614
0. 03	0.229	0.190	0.053	0.204	0.219	0.239	0.1842	0.0810

TABLE	(17)
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			Predict	ted Axial	d Axial Dispersion Coefficient Experimental Axia (ft ² /sec) (ft ² /sec)						Axial ficient)
G 	as Velocity (ft/sec)	Kato & Nishiwaki	Towel & Ackerman	Cova	Deckwer	Hikita & Kikukawa	Baird & Rice	Ying	C _S =ZERO	$C_{\rm s}=5$ 1b/ft ³	$C_{c}=20$ lb/ft ³
	0.0500	0.0000	0.2750	0.1653	0.2760	0,2187	0.4096	0.2789	0.405	0.297	0.304
47	0.0970	0.0002	0.3831	0.2044	0.3367	0,2645	0.5097	0.3541	0.485	0.341	0.295
•	0.1940	0.0007	0.5418	0.2551	0.4145	0.3452	0.6407	0.4545	0.495	0,364	0.366
	0.3270	0.0024	0.7034	0.3015	0.4848	0,4418	0.7611	0.5484	0.614	0.471	0.376

TABLE (18)

Comparison of Axial Dispersion Coefficients in Liquid Phase for the 12" Column

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C_S = Solids Concentration Particle Size = 20/30 Mesh











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Figure (4)





Figure (6)

Dimensionless Time, O





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Figure (10)



Figure (11)

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Figure (12)

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Tracer Concentration (Ce = C/C_0)

Dimensionless Time, O


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Dimensionless Time, O

Tracer Concentration ($C_0 = C/C_0$)











Tracer Concentration ($C_0 = C/C_0$)



Figure (20)



Figure (21)





Figure (23)

Tracer Concentration (Ce = C/Co)





Figure (25)



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Figure (26)

Figure (27)



Tracer Concentration ($Ce = C/C_0$)





Figure (29)

Dimensionless Time, Θ

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Tracer Concentration (Ce = C/C_0)







Tracer Concentration ($C_0 = \frac{C/C_0}{C_0}$)



Figure (32)

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Figure (34)

Tracer Concentration (Ce)

Appendix 1

EXPERIMENTAL PLAN

ON

GAS/SLURRY FLOW IN COAL LIQUEFACTION PROCESSES

TO

UNITED STATES DEPARTMENT OF ENERGY

NOVEMBER 16, 1979

BY

DAVID H. S. YING AND EDWIN N. GIVENS AIR PRODUCTS AND CHEMICALS, INC. ALLENTOWN, PA 18105

UNDER CONTRACT NO. DE-AC01-79ET14801

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This detailed experimental program schedule is prepared and submitted in comliance with the work statement under "Task 1. Experimental Planning" in the DOE-APCI contract titled "Gas/Slurry Flow in Coal Liquefaction Processes".

The overall objective of this project is to study the solids accumulation, liquid dispersion, and gas holdup in both gas-liquid and gas-liquid-solid systems in cold-flow tubular columns, aimed at providing data for the coal dissolver design in the SRC-I demonstration plant. The specific objective and duration of each task are the same as specified in the work statement. The sequence of the experimental plan is slightly rearranged however. The rearranged sequence is based upon the most recent experimental results which reinforces the fact that Task 2 forms the data base for the entire program. Therefore it is important that Task 2-be completed prior to the start of other tasks as snown in the attached program schedule. A detailed experimental plan for fiscal year 1980 is also prepared.

The contract work will be carried out in existing apparatus available at the contractor site, including two cold-flow vertical tubular columns as follows:

Diameter (in)	<u>Height (ft)</u>	Volume (gal)	
	-		
5	5	5.1	
12	25	147	

The distributor plate in the 12-inch diameter column is of identical design to that at Wilsonville, and will be used throughout this contract work except for Task 3.

Task 2 Two and Three Phase Flow in Vertical Columns

Task 2.1 Two Phase Flow

The objective of this subtask is to check the adequacy and reproducibility of the experimental apparatus and the technique used. An air-water mixture will

be used throughout this subtask. The ranges of variables to be investigated are summarized as follows:

- Liquid flow rate 0-0.10 ft/sec
- Gas superficial velocity 0.05-1.0 ft/sec

Void fraction, liquid dispersion, and gas-liquid mass transfer will be studied in this subtask. The detailed experimental plan for each subject is presented below.

- <u>Void fraction</u> will be measured in the 12-inch column under the following conditions:
 - In absence of liquid flow at 12 different gas velocities.
 (A few runs will also be performed at a different gas inlet pressure to assure the accuracy of the equipment.)
 - In liquid flow at four different liquid velocities at each of four different gas velocities.

Therefore a total of 28-35 runs will be performed for the void fraction measurement in this subtask.

Liquid dispersion in the tubular column will be investigated in the 12-inch diameter column using tracer technique. Experimental data will be obtained for five different gas velocities at one liquid flow rate and for five different liquid velocities at one gas velocity. The effect of liquid flow rate will be confirmed by obtaining data at two different liquid velocities at another gas velocity. The data will be presented in the form of normalized tracer curve and its corresponding dispersion coefficient. A total of 12 runs will be performed for liquid dispersion study in the 12-inch diameter column. The technique of tracer injection and the appropriate location of the ion detector on the column will be investigated separately to provide consistent results.

Gas-liquid mass transfer study will be performed in both the 5-inch diameter and 12-inch diameter columns. Several experimental techniques will be tested in the 5-inch diameter column and one will be selected for this task. Two such methods that will be investigated will be oxygen absorption in water and oxygen absorption in sodium sulfite solution. Both batch and continuous flow experiments will be performed; a minimum of 4 gas velocities and 4 liquid velocities will be investigated. The overall mass transfer coefficients (k_La) will be reported. The total number of runs in the 5-inch diameter column will be 16-20 including those in the technique screening tests. A limited number of runs for mass transfer measurement will also be carried out in the 12-inch diameter column. In addition, organic liquid may be employed for mass transfer measurement in the 5-inch diameter reviewing with DOE TPO.

The experimentally derived data including void fractions, mass transfer coefficients, and liquid dispersion coefficients will be correlated by the published and in-house correlations to determine the adequacy of apparatus and experimental technique. The schedule of all the runs performed in this subtask is shown in the attached detailed experimental plan for FY '80.

Task 2.2 Three Phase Flow

The objective of this subtask is to simulate solids suspension and accumulation in coal dissolver via the use of an air/water/sand mixture in cold-flow vertical tubular columns. The ranges of variables to be investigated are summarized as follows:

		Slurry	flow rate	0-0.10 ft/sec
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• Superficial gas flow rate 0.05-0.6 ft/sec

Sand particles taken from the following narrow ranges:

20-30 mesh 30-40 mesh 40-60 mesh 60-80 mesh 80-100 mesh 100-120 mesh greater than 140 mesh

Sand concentration 5-25 lb/ft³ of dissolver volume

For many of the narrow particle size expriments, at least one ton of sieved material shall be prepared by the contractor using sifting equipment which is available at contractor site. Void fraction, liquid dispersion, and solid phase dispersion will be studied in this subtask.

Void fraction will be measured in the 12-inch diameter column. The variables are gas velocity, liquid velocity, solid particle size, and solid concentration. Two particle sizes will be investigated. For each particle size a total of 44 runs will be performed at different combinations of solid concentration and gas and liquid velocities as summarized below:



Therefore a total of 88 runs will be performed. The two extremes of the particle size ranges shown earlier (20-30 mesh and greater than 140 mesh) are the appropriate choices to detect any particle size effect on void fraction measurement. Other particle sizes will be studied following consultation with DOE TPO.

Liquid Dispersion will be measured in the 12-inch diameter column at different combinations of particle size, solid concentration and gas and liquid velocity as shown below:



Two particle sizes will be studied; the total number of runs will be 36.

Solid phase dispersion experiments will be carried out in the 5-inch and l2-inch columns, with the 5-inch column used to develop a satisfactory technique and the l2-inch column used to obtain performance data. The operating variables to be studied will include gas and liquid velocities, solid concentration and particle size. Results from the 5-inch column will be reviewed. Based on these results, an experimental plan will be prepared for the l2-inch column. Also included in this subtask will be the sampling of slurry from five (5) locations along the height of the l2-inch column and the subsequent solid content analysis of these samples during a few selected runs. Solid accumulation at various chosen flow conditions and the solid dispersion coefficients will be reported in this subtask. Sampling technique will be developed in both 5-inch and l2-inch diameter columns to provide adequate results. Based on the results of this subtask (void fraction, liquid phase and solid phase dispersion coefficients), a model will be developed to correlate the solids accumulation and suspension of the air/water/sand mixture in a tubular column. Additional runs will be carried out to validate this model. The performance schedule is hown in the attached detailed experimental plan for FY '80.

Task 3 Effect of Distributor on Flow

The objective of this task is to study the entrance effects on the performance of the 12-inch column. Under this task, the contractor shall design, fabricate, and install a new distributor in the 12-inch column which will be aimed at providing better solid suspension than with the distributor used under Task 2. The contractor shall also study the performance of the cold-flow tubular column in the absence of a distributor.

Task 3.1 New Distributor (12-inch Column)

Task 3.1.1 Two Phase Flow

Based upon the results of Task 2, a new distributor will be designed to improve dissolver performance. The objective, experimental plan, and the data workup procedure of this subtask are the same as those of Task 2.1 in which the standard Wilsonville design distributor was used. Results from these two subtasks will be compared. Repeating the complete set of experiments under Task 2.1 is not highly desireable. The exact number of runs will be determined following consultation with DGE TPO.

Task 3.1.2 Three Phase Flow

The objective, experimental plan, and the data workup procedure of this subtask are the same as those of Task 2.2 using the standard Wilsonville design distributor. In case the new distributor does not yield improved solid suspension over that used under Task 2.2, a second new distributor will be fabricated, installed and tested.

Task 3.2 Absence of Distributor (12-inch Column)

The schedule for this task is also shown in the detailed experimental plan.

Task 3.2.1 Two Phase Flow

The objective, experimental plan, and data workup procedure of this subtask are the same as those of Task 2.1. However, the number of runs will be reduced due to the constraint of column physical strength that results from removal of the distributor.

Task 3.2.2 Three Phase Flow

The objective, experimental plan, and data workup procedure of this subtask are the same as those of Task 2.2. Again the number of runs will be reduced as in Task 3.2.1 above.

Task 4 Schids Removal Study

The objective of this task is to test the effectiveness of slurry withdrawal from the column bottom as a means to control solids accumulation. Experiments will be carried out in the 5-inch column to develop a satisfactory technique for the slurry withdrawal in continuous mode, and then in the 12-inch column to obtain performance data.

The experimental plan with the 12-inch column runs will have only one variable, i.e., the slurry withdrawal rate from the bottom. The withdrawal rate will be studied over a range from 1-20% of the feed slurry rate. The other operating conditions including gas and liquid velocities, solid concentration, and particle size will be determined based on the results of Task 2, and will remain constant throughout this task. The experimental plan with the 12-inch column will also include limited number of runs in which the slurry is withdrawn under the intermittent rather than the continuous mode, and the results from these two modes of operation will be compared.

Upon the completion of the work as listed above, a meeting will be held with DOE TPO to determine if this task should be extended.

Task 5 Organic Fluid Phases

The objective of this task is to study the effects of liquid surface tension and viscosity on the performance of a cold-flow tubular column.

Prior to the initiation of experimental work, the contractor shall enclose the existing 12-inch column to provide safe handling of volatile and flammable organic liquids.

A list of candidate organic liquids which have lower surface tension compared to water will be prepared by the contractor, and submitted to DOE TPO for review approximately one month before the initiation of this task. From this list, two organic liquids will be chosen in the 5-inch column study using an air/organic fluid/sand mixture as the feed. One of the above two organic fluids or a third one will be chosen in the 12-inch column study. The experimental plan and data workup procedure of this task will be the same as those of Task 2.2.

Results from this task will be compared with those from the aqueous system, and will be fitted by the correlations developed from Task 2. If necessary other models will be developed to fit the experimental data from this task.

Task 6 Behavior of Multiple Distributors

The objective of this task is to explore the use of a second distributor to achieve a higher plug flow characteristic in a cold-flow tubular column. Under this task, the contractor shall fabricate and install an additional distributor in the 12-inch column. This second distributor will be identical to the inlet distributor, and its position relative to the inlet distributor will be adjustable. A limited number of runs will be carried out with an air/water mixture for the shake-down of the 2-distributor column. Performance data will be obtained in the 12-inch column using an air/water/sand mixture. The only variable in the experimental plan will be the distance between the second and inlet distributors, and its range will be 5-20 feet. The other operating conditions including gas and liquid velocities, solid concentration, and solid particle size will remain constant throughout this task, and will be

chosen based on the results of Task 2.2. The types of experimental data to be obtained under this task and the subsequent data workup procedures will be the same as those of Task 2.2.

Upon completion of the work as listed above, a meeting will be held with DOE TPO to determine if this task should be extended. Additional work to be considered will include the performance of 2-distributor column as a function of slurry and gas velocities, and the slurry withdrawal from two individual sections in this column.

Task 7 Final Report Writing

Under this task a final report will be prepared including all research results and containing recommendations for further evaluation of dissolver performance. An evaluation of the potential for employing the techniques described here for preheater and heat exchanger flow studies will also be included.

Deliverable Items

The deliverables will be in the form of data, data correlations, and conclusions relating the performance of cold-flow tubular columns conducted under this contract, and will be delivered according to the schedule listed in the Reporting Requirement Checklist. The deliverable data include gas void fraction, degree of liquid backmixing, liquid dispersion coefficients, gas-liquid mass transfer coefficients, solid accumulation, and solid phase dispersion coefficients.

DETAILED EXPERIMENTAL PLAN FOR FY 1980

October	Task 1 Task 2.1 Task 2.2	Detailed Experimental Planning Void Fraction Void Fraction
November	Task 2.2 Task 2.1 Task 2.2	Void Fraction Liquid Phase Dispersion Liquid Phase Dispersion
December	Task 2.2 Task 2.2	Liquid Phase Dispersion Solid Phase Dispersion
January	Task 2.2	Solid Phase Dispersion
February	Task 2.2 Task 3.1.1 Task 2.1	Solid Phase Dispersion New Distributor Plate Design Gas-liquid Hass Transfer
March	Task 2.1 Task 3.1.1	Gas-Liquid Hass Transfer New Distributor Plate Design
April	Task 3.2.1 Task 3.2.2 Task 3.2.1	Void Fraction Void Fraction Liquid Phase Dispersion
May	Task 3.2.2 Task 3.2.1	Liquid Phase Dispersion Gas-liquid Nass Transfer
June	Task 3.2.2 Task 3.1.1 Task 3.1.2	Solid Phase Dispersion Void Fraction Void Fraction
July	Task 3.1.1 Task 3.1.2	Liquid Phase Dispersion Liquid Phase Dispersion
August	Task 3.1.1	Gas-liquid Mass Transfer
September	Task 3.1.1	Sold Phase Dispersion Study

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