Energy

F
O
S
S
I
L

245
6/2/84
MRC (2)

DR # 0105-8

DOE/PC/30026-T5
(DE84010230)

STUDY OF EBULLATED BED FLUID DYNAMICS

Final Progress Report for the Period September 1980—July 1983

By
R. J. Schaefer
D. N. Rundell
J. K. Shou

July 1983
Date Published

Work Performed Under Contract No. AC22-80PC30026

Amoco Oil Company
Naperville, Illinois

Technical Information Center
Office of Scientific and Technical Information
United States Department of Energy

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of 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. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of 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. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Printed Copy A16
Microfiche A01

Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: *Energy Research Abstracts (ERA)*; *Government Reports Announcements and Index (GRA and I)*; *Scientific and Technical Abstract Reports (STAR)*; and publication NTIS-PR-360 available from NTIS at the above address.

STUDY OF EBULLATED BED FLUID DYNAMICS

FINAL PROGRESS REPORT
SEPTEMBER, 1980, TO JULY, 1983

R. J. SCHAEFER, D. N. RUNDELL, J. K. SHOU

DATE PUBLISHED: JULY, 1983

CONTRACT DE-AC22-80PC30026

AMOCO OIL COMPANY
RESEARCH AND DEVELOPMENT DEPARTMENT
P. O. BOX 400
NAPERVILLE, ILLINOIS 60566

ERRATA

1) TABLE OF CONTENTS

Appendix B starts on page 225 (not page 235)

Appendix C starts on page 235 (not page 245)

Appendix D starts on page 260 (not page 258)

Appendix E starts on page 282 (not page 280)

Appendix F starts on page 286 (not page 284)

Appendix G starts on page 303 (not page 301)

2) Page 25

Equation 10 should read:

$$S_i = \sqrt{\ln \left(\frac{\sigma_i^2}{T_m^2} + 1 \right)}, \text{ where } i = 1 \text{ and } 2$$

3) Page 25

Equation 11 should read:

$$C = C_0 + \frac{Q}{T \cdot s \cdot \sqrt{2\pi}} \exp \left[- \frac{(\ln T - \ln T_m)^2}{2s^2} \right]$$

STUDY OF EBULLATED BED FLUID DYNAMICS

FINAL PROGRESS REPORT
SEPTEMBER, 1980, TO JULY, 1983

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	
I) INTRODUCTION	4
II) OBJECTIVES AND SCOPE OF PRESENT WORK	6
Task 1: Model Validation	6
Task 2: Bubble Dynamics Studies	6
Task 3: Gas/Liquid Mixing	7
Gas Mixing	7
Liquid Mixing	7
Catalyst Mixing	7
Task 4: Model Implementation	7
III) AMOCO COLD-FLOW EXPERIMENTS: BED EXPANSION AND PHASE HOLDUPS	7
Equipment and Procedures	8
Experimental Conditions and Data Collection	9
Data Analysis and Results	9
Phase Holdup and Other Hydrodynamic Parameters	9
Catalyst Bed Expansion	11
IV) AMOCO COLD-FLOW EXPERIMENTS: RADIAL UNIFORMITY OF PHASE HOLDUPS	12
Review of Prior Work	12
Equipment and Procedure	13
Experimental Conditions and Data Collection	13
Data Analysis	14
Results and Discussion	14
V) AMOCO COLD-FLOW EXPERIMENTS: AXIAL SLURRY DISPERSION	15
Background	15
Review of Liquid Dispersion in Multi-Phase Fluidization	16
Gas/Liquid Systems	16
Gas/Slurry Systems	17
Gas/Slurry/Solid System	18
Reacting Systems	18
Tracer Selection	19
Bench-Scale Experiments and Results	20

(Continued)

TABLE OF CONTENTS

	<u>Page</u>
Du Pont Stadis-450	20
Mobay Blue ZV Dye	21
Thiophene	21
Pilot Plant Testing of Thiophene as a Tracer	21
Equipment	22
Pilot Plant Results and Discussion	23
Data Collection	23
Data Analysis	24
Conclusions	26
VI) BUBBLE DYNAMICS	26
Experimental System	27
Description of the Apparatus	27
Fluids and Solids	28
Bed Expansion and Pressure-Drop Measurements	28
Bed Expansion Measurements	28
Pressure-Drop Measurements	28
Light Beam Probe and Bubble Breakup Model	29
Description of the Light Beam Probe	29
Results of the Light Beam Technique	29
Equilibrium Bubble Size Model	29
Bubble Breakup and the Critical Particle Size	30
Holography	30
Description of Holographic Technique	30
Holographic Analysis System	31
Results	31
Resistivity Probe	31
Summary	32
Recommendations	32
VII) HYDROCARBON RESEARCH, INCORPORATED, PDU EXPERIMENTS	33
Samples and Data Collection	33
PDU Operating Conditions	33
Liquid Samples	33
PDU Reactor Effluent Composition	33
Catalyst Samples	33
Gamma-Ray Scan	34
Density and Viscosity of PDU Reactor Liquid Slurry Samples	35
Data Transformations	37
Gas Velocity	37
Slurry Velocity	37
Catalyst Bed Expansions	38
Phase Volume Fractions	39
Mass Absorption Coefficients for the PDU Fluid	40
Dynamics Tests	

(Continued)

TABLE OF CONTENTS

	<u>Page</u>
Volume Fractions	40
Discussion	41
Comparison with Cold-Flow Pilot Plant Tests	42
VIII) MATHEMATICAL MODEL	43
Review of the Bhatia-Epstein Model	44
Model Parameters	45
Cold-Flow Tests	46
Application to HRI PDU-10 Tests	48
Comparison of PDU and Cold-Flow Pilot Plant Model Results	50
IX) MODEL APPLICATION	50
X) CONCLUSIONS	51
ACKNOWLEDGMENTS	52
REFERENCES	53
NOMENCLATURE	58
TABLES	60-113
FIGURES	114-206
APPENDIX A: CATALYST BED EXPANSIONS AND PHASE HOLDUPS FOR RUNS 218-224	207
APPENDIX B: LIQUID DISPERSION DATA ANALYSIS	235
APPENDIX C: VISCOSITY AND DENSITY OF H-COAL PDU LIQUID SLURRY SAMPLES: BATTELLE INSTITUTE FINAL REPORT	245
APPENDIX D: VISCOSITY AND DENSITY OF H-COAL PDU LIQUID SLURRY SAMPLES: OAK RIDGE NATIONAL LABORATORIES FINAL REPORT	258
APPENDIX E: CALCULATION: FORCE TO SUSPEND A CATALYST PARTICLE	280
APPENDIX F: I/O DATA FOR PDU-10 FLUID DYNAMIC TESTS	284
APPENDIX G: PREDICTION OF RICHARDSON-ZAKI PARAMETERS FOR PDU EXPERIMENTAL MODELING	301

ABSTRACT

The fluid dynamics occurring in HRI's H-coal process development unit (PDU) coal liquefaction reactor during Run PDU-10 were measured and compared with Amoco Oil cold-flow fluidization results. It was found that catalyst bed expansions and gas holdups are higher in the PDU than those observed in the cold-flow tests for slurries having the same nominal viscosity. Comparison of PDU results with cold-flow results shows that the bulk of the operating reactor gas flow lies in the ideal bubbly regime. It also appears that the gas bubbles in these PDU tests are rising quite slowly. Only two of the operating points in our test program on the PDU were found to lie in the churn turbulent regime. Existence of churn turbulent behavior during these two experiments is consistent with trends observed in earlier cold-flow experiments.

Two- and three-phase fluidization experiments were carried out in Amoco's cold-flow fluid dynamics unit. The data base now includes fluidization results for coal char/kerosene slurry concentrations of 4.0, 9.8, and 20.7 vol% in addition to the 15.5 and 17.8 vol% data from our earlier work. Both HDS-2A and Amocat-1A catalysts were used in the tests. Bed expansion is primarily a function of slurry velocity, with gas velocity having only a weak effect. Bed contractions have been observed in some cases at sufficiently high gas velocity. Gas and liquid holdups were found to be uniform across the cross-section of the Amoco cold-flow fluid dynamics pilot plant.

A viscometer was adapted for measurement of the viscosity of coal slurries at high temperature and pressure. Three techniques were developed for the study of the bubble dynamics occurring in three-phase fluidized beds: 1) A laser light beam probe for measuring the behavior of bubbles greater than 120 microns; 2) A laser holographic technique for determining the size, shape, and position of bubbles in the bed; and 3) A resistivity probe for determining bubble frequencies and a qualitative measure of the flow regime. The bubble size studies showed that the design of the inlet distributor played an important role in the breakup of bubbles entering the fluidized bed. It was shown that smaller bubbles can be generated by this particular inlet distributor than could be produced by the breakup of bubbles by solid particles within the bed.

Based on experiments carried out in the Amoco cold-flow unit, a significant degree of backmixing was found to occur in the H-Coal system. Dispersion coefficients and Peclet numbers were found to lie in the ranges 70 to 130 cm²/sec and 3.6 to 9.9, respectively.

I) INTRODUCTION

The H-Coal process, developed by Hydrocarbon Research, Incorporated (HRI), involves the direct catalytic hydroliquefaction of coal to low-sulfur boiler fuel or synthetic crude oil. The process has been demonstrated in a 3 T/D process development unit (PDU) at HRI's Trenton, New Jersey, facilities and in a 600 T/D pilot plant at Catlettsburg, Kentucky.

The HRI H-Coal process involves the use of an ebullated-bed reactor, as shown in Figure 1. The catalyst in the reactor is fluidized by the flow of slurry and hydrogen gas passing up through the reactor. The slurry stream entering the reactor consists of fresh coal slurry feed and a recycle slurry stream. The gas stream consists of recycled hydrogen and fresh make-up hydrogen.

The design, operation, and control of the reactor requires a detailed understanding of the fluid dynamics of this complex reacting system. Factors affecting the performance of the reactor and information needed for design include the following:

- A) Catalyst bed expansion.
- B) Void fraction (phase holdup) of the gas, slurry, and catalyst phases and their variation along the reactor length.
- C) Bubble dynamics, including wake effects.
- D) Mixing parameters: Axial and radial mass dispersion of the gas, slurry, and catalyst phases, and axial and radial dispersion of heat.
- E) Transport parameters: Gas/liquid and liquid/solid mass transfer coefficients and gas/liquid interfacial area.
- F) Thermodynamic parameters: Vapor/liquid thermodynamic data, specific heats, thermal conductivities.
- G) Reaction data: Kinetics, conversion, product yields, and product qualities.

Our previous research on the fluid dynamics of the H-Coal system (1) addressed several of the above items (A, B, and part of C and D). A cold-flow pilot plant was built which is dimensionally identical with the H-Coal PDU reactor shown in Figure 1. Cold-flow fluidization experiments were carried out using kerosene, mineral oil, and several coal char/kerosene slurries. The gases included nitrogen and helium. A standard (1/16" x 3/16") catalyst, Cyanamid HDS-2A, was used in the majority of the experiments. Direct sampling of the slurry at various points throughout the system showed that the concentration of the slurry was uniform throughout the system and thus could be treated as a single phase.

Bed expansion data and phase holdup data were collected for a variety of gas and liquid (slurry) flow rates. Although the majority of the data were collected using pure fluids of the appropriate viscosity, some data were also collected using coal slurries. Analysis determined that the Richardson-Zaki correlation describes the effect of slurry flow rate on catalyst bed expansion. Bed expansion is primarily a function of liquid (slurry) velocity, with gas velocity having a secondary effect. When a lower-viscosity pure fluid such as kerosene is the fluid phase, an increase in gas velocity always results in an increase in bed expansion. However, when a higher-viscosity fluid is used such as mineral oil or a coal char/kerosene slurry, a slight contraction of the fluidized catalyst bed can occur above a sufficiently high gas velocity.

The experimental data were used to develop a modified Bhatia-Epstein model which can predict bed expansion and phase holdups for the various phases as a function of gas and liquid (slurry) superficial velocities. It was shown that the bed could be described by the gas bubbles, wakes following the bubbles, and the slurry/catalyst particulate phase, as shown in Figure 2. Correlations were derived describing bubble rise velocity, wake volume, and solids holdup in the wake as a function of operating conditions.

The nature of the bubble dynamics for the three-phase system was studied using the Darton-Harrison drift flux model. Analysis showed that ideal bubbly flow occurs in the ebullated bed over much of the operating range when pure kerosene is used as the liquid phase. Above a certain velocity, however, the gas flow will change from small bubbles (ideal bubbly) to large bubbles (churn turbulent). The latter type of flow results in an unsteady flow behavior in the bed. When a high-viscosity fluid such as mineral oil or a coal char/kerosene slurry is the liquid phase, the gas flows in the form of large bubbles over much of the gas velocity range. Mass transfer effects and chemical reactions depend strongly on bubble size distribution and residence time; and therefore, knowledge of the type of gas flow occurring in the system is very important.

Radioactive gas tracer studies were also carried out to investigate the nature of the bubble dynamics and determine the extent of gas dispersion in the H-Coal system. It was determined that the flow of gas through the catalyst bed is intermediate between plug flow and backmixed. It was also concluded that at sufficient gas velocity, large bubbles having a higher rise velocity are flowing up the center of the column with a flow of smaller bubbles down along the wall.

Reaction data (Item G), including conversion, product yields, and product qualities, have been obtained by HRI (2) during operation of the 3 T/D PDU under a variety of operating conditions. Micro-autoclave data have also been obtained by HRI as a supplement to the PDU data and as an operational aid.

Although the above results provided a better understanding of the fluid dynamics in the three-phase fluidized H-Coal system, several major and important areas remained unexplored and required investigation. This, then, is the basis for our current work, and the results are discussed in this report.

II) OBJECTIVES AND SCOPE OF PRESENT WORK

Task 1: Model Validation

A major question in fluidization research is how well cold-flow experiments conducted at ambient temperature and pressure simulate the fluid dynamics occurring in a reacting system under reaction conditions. With regard to our studies, a corollary question involves how well the modified Bhatia-Epstein model based on cold-flow data predicts bed expansions and phase holdups for the H-Coal system. An experimental program was conducted in this current study on the HRI PDU during Test PDU-10 to obtain the necessary fluidization data. The objective was to compare the fluid dynamics measured in the actual H-Coal reactor with those measured in the cold-flow pilot plant experiments. The analysis included a critical evaluation of our modified Bhatia-Epstein model which had been developed based on cold-flow data.

Density, viscosity, and surface tension of the liquid phase play important roles in three-phase fluidization. Samples of the slurry were withdrawn from the PDU reactor by HRI during the PDU-10 experiments for analysis. As far as we know, there is no readily available technique for measurement of the surface tension of coal slurries at high temperature and pressure. Measurement of the density is relatively straightforward. The viscosities of the slurry samples were determined by Battelle Research Institute and Oak Ridge National Laboratory in their viscometers at reaction temperature and pressure. Battelle had successfully used the same technique previously for viscosity measurement of Synthoil products. An objective of our current work was the adaption by Battelle of their viscometer to the measurement of H-Coal reactor samples at reaction temperature and pressure.

Although not funded by this contract, four H-Coal reactor slurry samples were sent to Oak Ridge for viscosity measurement in their pipeline viscometer. The purpose was to make a comparison of the viscosity values from two independent techniques.

An additional objective of Task 1 was to obtain additional bed expansion and phase holdup data for coal char/kerosene slurries having several concentrations. Although the earlier slurry data were valuable, they were limited in scope. This additional fluidization information will enlarge the slurry data base and provide additional confidence to our modeling efforts.

Task 2: Bubble Dynamics Studies

It was clear from our earlier work as well as results in the literature for other experimental systems that a basic understanding of the bubble dynamics in the system is essential. The objective of this task was to determine gas holdup, bubble size distribution, and respective bubble velocities for a given set of operating conditions. A major part of this task to be carried out by Northwestern University researchers was to develop techniques suitable for studying such phenomena in this complex three-phase fluidized bed system. The technical plan included development of a resistivity probe, a narrow-beam laser probe, a laser holographic technique,

and a laser-Doppler anemometric technique. (It turned out that only the laser-Doppler anemometry could not be developed, because the bubbles were smaller than anticipated due to the nature of the fluids chosen in this part of the cold-flow study. This precluded study of the liquid flow patterns using this technique.)

Task 3: Gas/Liquid Mixing

Gas Mixing.--A common assumption in many modeling studies, including our modified Bhatia-Epstein model, involves radial uniformity in the system. Results from the radioactive tracer studies in our earlier work raise some question whether such an assumption is valid in the H-Coal reactor. It was found earlier that for at least certain operating conditions there are larger bubbles having large rise velocities traveling up the center of the column, with downflow of smaller bubbles along the wall. Therefore, an objective of this study was to determine the gas holdup as a function of radial position in the cold-flow column. Because bubble coalescence--i.e., churn turbulent behavior--explains the gas tracer results, coal slurries were chosen as the fluid of interest in this task.

Liquid Mixing.--The liquid residence time distribution is also required to understand the mixing/dispersion phenomena occurring in this three-phase system. Because our earlier results showed that the concentration of coal char fines in the slurry was uniform throughout the system, the slurry can be treated as a pseudo-homogeneous phase. The objective of this task was to determine the amount of slurry mixing/dispersion occurring in the H-Coal system. Our cold-flow pilot plant was used in this phase of the study.

Catalyst Mixing.--As part of a separate DOE contract, Sandia National Laboratories charged Co-60 tagged catalyst to the HRI PDU during PDU-10 to determine the extent of catalyst mixing in the H-Coal reactor. Catalyst withdrawals were made daily, and the level of radioactivity was determined. Sandia found that the reactor is acting approximately as a backmixed system. A maximum in radioactivity level of the withdrawn catalyst was found to occur three days after addition.

Task 4: Model Implementation

The results from the current studies were incorporated into our modified Bhatia-Epstein model. The model is available upon request to users in easy-to-understand format in the form of computer cards or tape.

III) AMOCO COLD-FLOW EXPERIMENTS: BED EXPANSION AND PHASE HOLDUPS

Cold-flow experiments were performed to measure the fluid dynamics properties of the three-phase ebullated bed (catalyst bed expansion and component phase holdups). The goal of these experiments was to study the response of these variables to changes in operating conditions and to provide data to develop parameters for the Bhatia-Epstein model. In order to validate and improve on this model, which was developed under the previous contract (1), these

cold-flow fluidization experiments were carried out to provide a wider range of fluid dynamic data. Kerosene and slurries of 4.0, 9.8, and 20.7 vol% coal char fines in kerosene as the continuous fluid phase were studied with the H-Coal (Cyanamid HDS-2A, 1/16" ID x 3/16" long) catalyst. A 15.4 vol% slurry was also used with the newly developed Amocat-1A catalyst particles. In both cases, nitrogen was used as the gas phase.

Equipment and Procedures

A schematic diagram of the H-Coal cold-flow fluid dynamics pilot plant is shown in Figure 3. The process flow of this pilot plant simulates that of the process development unit (PDU) built by HRI. Detailed descriptions of the equipment have been presented in a previous report (1). The reactor column, gamma-ray equipment, and some changes of piping are briefly described here.

The reactor is constructed from four 6" ID x 5' long glass sections, which are connected via flanges to five metal spool pieces. These spool pieces have connections for sample taps, pressure taps, and thermowells to monitor the system. Samples of the slurry are withdrawn through the spool pieces to establish the concentration of coal char fines along the reactor column.

A computer-controlled sled carries a Cs-137 gamma-ray source and a detector vertically along the reactor. The sled was modified to allow parallel chords at any given elevation to be penetrated by the Cs-137 gamma rays. Gamma-ray scan data obtained during the experiments are used to calculate bed expansions and phase holdups.

Minor piping modifications (shown by dashed-line connection in Figure 3) were made to improve the pilot plant's operability. During Run 221, it was necessary to keep liquid flowing through the recycle cup downcomer line (see Figure 1) to the slurry recycle pump to prevent blockage by the coal fines. Mixing of this stream and a parallel stream from Separator D-3 through the slurry feed pump into the unit was hampered by interaction between the two pumps. The recycle pump, a positive-displacement pump, was overcoming the head of the centrifugal slurry feed pump. Some experiments were performed by draining the separator and recycling all the slurry feed through the recycle cup. Gas entrainment in this recycle line, however, hampered data collection at higher gas velocities. Subsequent runs were conducted using the modified piping system, shown by the dashed line, in which both recycled slurry streams are now drawn from Feed Tank D-1.

For those tests that started with a new batch of catalyst, the catalyst particles were allowed to soak in the kerosene for a period of two days. The coal char fines concentration in the kerosene was then adjusted to the desired value. The gamma-ray scanning technique was employed to find the bed height and volume fractions occupied by the various phases for each run.

A zero gamma-ray scan was then carried out to find the bed height (H_0) at zero gas and liquid flow. For each subsequent set of operating conditions,

the gamma-ray elevator was used to scan the reactor length. These data, obtained by the Modcomp II computer, were stored for subsequent analysis and plotting. An example of gamma-ray scans of various gas velocities is shown in Figure 4. From plots of this type, a bed height was established for each test. Based on this bed height, the initial catalyst charge, and the gamma-ray measurements along the reactor, the holdup of each individual phase was determined. When coal char fines are present, additional information is needed. This was established by measuring the fines distribution along the reactor by direct sampling through the spool pieces. Details of the calculation procedures to establish the holdup of each phase can be found in an earlier report (1).

Experimental Conditions and Data Collection

Table I summarizes the operating conditions and other information for all experimental cold-flow runs conducted during this program. All of these tests were performed at ambient temperature and pressure. Gas, catalyst, and slurry volume fractions were determined at various gas and slurry velocities within the respective gas and slurry flow rate ranges listed in Table I.

Run 218, which involved kerosene, was performed to measure unit performance and provide a base line for the subsequent runs. After the cold-flow pilot plant was configured as shown in Figure 3, fluid dynamics tests were conducted with various coal char/kerosene slurries. Slurry samples were taken at each spool piece during Runs 221-224 under flow conditions to check the coal char concentration in the kerosene.

Bench-scale experiments were also performed to measure the apparent density of the coal char fines, the soaked particle density of the catalysts, and the average dimensions of the catalyst particles. These results are summarized in Table II. Runs 219 and 220 were exploratory runs and are not presented in Tables I and II.

Data Analysis and Results

Appendix A contains experimentally determined catalyst bed expansions and phase holdup results for both the catalyst bed itself (dense phase) and the region above the bed (dilute phase).

Phase Holdup and Other Hydrodynamic Parameters.--The coal char/kerosene slurry is considered here as a pseudo-homogeneous liquid. This is supported by the uniformity of the coal char fines concentration as measured throughout the pilot plant system. For a two-phase liquid/solid system, the liquid (or slurry) holdup can be correlated with operating conditions and the physical properties of the system by the application of the Richardson-Zaki correlation:

$$\epsilon_{sl}^n = U_1/U_t$$

where $\epsilon_{sl} = \epsilon_l + \epsilon_f$.

Slurry holdup, ϵ_{sl} , can be calculated from the gamma-ray data or from catalyst bed expansion $(1 - \epsilon_c)$. When plotting ϵ_{sl} versus U_l on log-log paper (see Figure 5), the Richardson-Zaki parameters n and U_t can be determined from the slope and the intercept of the straight line that best fits the data. Table II summarizes the Richardson-Zaki parameters determined for each of the experiments. For purposes of comparison, the fines concentrations and corresponding viscosities are also presented in Table II.

Three-phase gas/liquid/solid fluidization data may be correlated using the drift flux approach of Darton and Harrison (3), where the drift flux, V_{CD} , is defined as

$$V_{CD} = U_g (1 - \epsilon_g) - U_l \epsilon_g (1 - \epsilon_g) / (\epsilon_l + \epsilon_f) \quad (1)$$

The drift flux physically represents the volumetric flux of gas relative to a surface moving at the average gas plus liquid velocity. A schematic diagram of V_{CD} versus ϵ_g is shown in Figure 6 to illustrate characteristics of different flow regimes defined by the V_{CD} and ϵ_g relationships. Two distinct flow regimes are identified and described by Darton and Harrison (3): 1) The ideal bubbly regime, in which the bubbles are relatively small and rise uniformly with little interaction; and 2) The churn turbulent regime, in which bubble coalescence is dominant; the bubble size is larger, and the flow is unsteady. There exists also a transition region between the ideal bubbly and fully developed churn turbulent flow regimes.

Data from Run 218 were analyzed according to Equation 1, resulting in a linear relationship between V_{CD} and ϵ_g , as seen in Figure 7. Because Run 218 used straight kerosene, the bubble size and the bed behavior could be observed visually at the glass column wall. Within the range of superficial gas and liquid velocities investigated in Run 218, ideal bubbly behavior was observed at the wall. The plot in Figure 7 substantiates the fact that the flow is ideal bubbly, as defined by the Darton-Harrison drift flux analysis. These results for Run 218 will define the ideal bubbly operating line for all subsequent cold-flow results involving slurries.

Drift flux plots for Runs 221 through 224 are presented in Figures 8-11. The solid line represents the ideal bubbly flow regime as defined by Run 218 experimental data. The dashed lines represent bed behavior in a transition regime between the ideal bubbly and fully developed churn turbulent states. A comparison of the drift flux results for Runs 221, 222, and 223 indicates that increasing the viscosity of the slurry phase tends to enhance the churn turbulent behavior of the bed at comparable operating conditions. This is consistent with the theory that a higher liquid viscosity promotes bubble coalescence, resulting in larger bubbles, which are characteristic of the churn turbulent flow regime. Higher slurry velocity has a common effect of reducing the magnitude of V_{CD} . Therefore, increasing slurry velocity will stabilize the bed behavior. A comparison of drift flux results for Runs 222 and 224 (Figure 9 versus Figure 11) indicates that drift flux correlations for the Amocat-1A (Run 224) catalyst are comparable to those for HDS-2A (Run 222).

Gas holdup is one of the most important variables characterizing the fluid dynamics of three-phase fluidization and is sensitive to the properties of the liquid phase, particularly the viscosity. The gas holdup in the catalyst bed, plotted versus the superficial gas velocity in Figure 12, increases with increasing velocity in all cases at low velocities. There is a velocity in each case, however, above which the gas holdup becomes constant. Alternatively stated, the average bubble rise velocity is increasing at the same rate that the gas superficial velocity is increased. Note in this latter region that the gas holdup decreases as the concentration of fines in the slurry is increased. This indicates that the higher concentration of fines, and thus higher slurry viscosity, promotes the coalescence of gas bubbles into larger bubbles having larger rise velocities. The end result is a decrease in the gas holdup.

Catalyst Bed Expansion.--Knowledge of catalyst bed expansion effects as a function of operating conditions is required for proper design and operation of the reactor. As the superficial slurry velocity is increased, the bed expansion increases, as shown in Figures 13 and 14 for 0.0 and 0.10 ft/sec superficial gas velocity, respectively. Addition of a small amount of fines does not affect the bed expansions to any great extent. Addition of larger amounts of fines does affect the bed expansions significantly due to the increased viscosity of the resulting slurries.

Bed expansions as a function of superficial gas velocity are presented in Figures 15 and 16. Note that the bed expansion is a much weaker function of gas velocity (Figures 15 and 16) than of slurry velocity (Figures 13 and 14). The results also show that there is apparently a slight bed contraction in some cases at sufficiently high gas velocity. Bed expansions of about 60% are observed in Figure 15 for a nominal H-Coal reactor slurry velocity of 0.10 ft/sec. This will be compared later with bed expansions measured on the actual H-Coal reactor in Section VII.

Bed contractions at higher gas velocities have been observed before (1), and a theory has been advanced (4,5) which has phenomenological appeal. The wake trailing a rising bubble contains pure liquid or slurry, which is traveling at the velocity of the rising bubble. This slurry in the wake is not available in the slurry proper for fluidization of the catalyst particles. Thus, the effective velocity of the slurry regarding fluidization of the catalyst is less, and thus the bed contracts. The bed contractions observed here are slight but real. Although bed contraction effects will not influence reactor design to any great extent, these effects are of considerable importance in the fundamental understanding of three-phase fluidization.

A qualitative difference between HDS-2A and Amocat-1A was observed in the nature of the catalyst bed/dilute phase interface. At certain combinations of liquid and gas flow rates, the Amocat-1A catalyst formed a very diffuse interface. By contrast, the interfaces measured with HDS-2A catalyst were distinct. Examples of sharp and diffuse interfaces are illustrated in Figures 73 and 74, respectively. Catalyst bed expansions for Run 224 are shown in Figure 16, along with the line delineating sharp and diffuse catalyst bed interfaces.

IV) AMOCO COLD-FLOW EXPERIMENTS: RADIAL UNIFORMITY OF PHASE HOLDUPS

It has been assumed in many studies that gas holdup is uniform across the reactor diameter. It is reasonable to make this assumption when the flow regime can be described as bubbly. In the churn turbulent flow regime, however, gas holdup becomes non-uniform, due either to large eddies or bubble coalescence or both. In the transition zone between the bubbly and the churn turbulent flow regimes, the degree of non-uniformity of gas holdups varies, depending on the physical properties of the continuous phase and on the velocities of both phases.

It was assumed in the previous contract work (1) that the phase holdups were constant across the diameter of the reactor. Some very limited experimental data indicated that this was a reasonable assumption. In order to verify this assumption, gamma-ray scanning experiments were carried out in this current contract at various parallel chords across the column.

Review of Prior Work

In spite of the wide variety and the vast amount of literature in the field of gas/liquid and gas/liquid/solid fluidization, few publications deal with the matter of whether the phase holdups vary in the radial direction. Only a few papers have dealt specifically with this question for two- and three-phase systems.

Hills (6) studied radial non-uniformity of gas holdup and liquid velocities in a 13.8 cm diameter column, two-phase, air-water system operated in a batch fluidization mode. Three different gas distributors were tested in order to evaluate their effects on radial gas holdup distribution. The results showed that for gas distributors having a large number of small holes (0.4 mm) evenly distributed on the plate, the gas holdups are radially uniform at gas velocities below 3.0 cm/sec. At higher gas velocities, there is an increasingly sharp central maximum in gas holdups. Results obtained with a seven-large-hole (1.6 mm) gas distributor showed the central maximum in the gas holdup at all gas velocities.

Recently, Linnweber and Blass (8) extended Hill's work to three-phase fluidization and measured both the gas and solid holdups as a function of radial position. Gas holdup profiles were measured for the air-water-plastic slurry and the air-water-glass slurry systems. The gas holdup profiles were essentially the same as those measured in the two-phase air-water system. The absolute values of gas holdup, however, were slightly less than those measured in the air-water system, due mainly to the addition of the solid phase. These authors also showed that at higher gas rates ($U_g \geq 3$ cm/sec), gas holdups increase toward the center of the column. The solid holdup profiles were relatively more flat in comparison with the gas holdup profiles, and they did not show obvious radial non-uniformity up to the gas velocity of 3.0 cm/sec. The results also showed that the gas and solid holdups are compensating each other--i.e., while gas holdups tend to increase toward the center of the column, the solid holdups decrease correspondingly toward the center of the column. As a result of this solid-gas compensating effect, liquid holdups are expected to be radially uniform at velocities less than 3.0 cm/sec.

Tsung (7) studied the radial gas holdup distribution in a gas-liquid bubble column while the system was operating in a steady-state flow condition. Nitrogen was used as the gas phase, and the liquid phase was a mixture of 66% dipropylene glycol monomethyl ether and 34% diphenyl ether. A single bubble cap distributor was used to disperse both the gas and liquid flows. The qualitative results obtained with a resistivity probe showed that the gas holdups vary with radial position, gas velocity, and liquid velocity. Gas holdups obtained from experiments conducted at low gas and liquid velocities ($U_g \leq 0.44$, $U_l \leq 1.34$ cm/sec) showed parabolic distributions with the maximum values at or near the center of the column. It was noted that the gas holdup distribution curve becomes flatter at a higher liquid flow rate. Nevertheless, the results are of limited value to our study because of the low gas and liquid velocities used in his study, and also because the probe could not be calibrated.

The limited data in our earlier contract work (1) are the only prior data available of direct relevance to the three-phase H-Coal system. Gamma-ray scans through parallel chords of the cross-section of the column indicated that the gas-phase holdups were essentially constant.

Equipment and Procedure

The Amoco H-Coal cold-flow pilot plant was modified so that gamma-ray scanning could be performed at various radial positions. This involved remounting the gamma-ray source and the detector on a traversing support structure and realigning the gamma-ray instrument. A detailed description of the pilot plant, the gamma-ray equipment, and the operating procedure was presented in a previous report (1).

Experimental Conditions and Data Collection

The gamma-ray scan experiments were carried out on the Amoco cold-flow pilot plant using a 25 wt% coal char fines in kerosene slurry system. The physical properties of the system are listed in Table II. The nitrogen gas flow rates were 0.08 and 0.10 ft/sec, while 0.10 and 0.125 ft/sec were selected for the slurry flow rates. Catalyst bed height data were taken during each test.

Gamma-ray scans were conducted at 30, 50, 70, 90, and 110" above the bubble cup distributor. At each elevation, gamma-ray scanning data were taken manually at seven chord positions across the reactor column. As shown in Tables IV and V, the chord position is indexed by R_i/R_o , where R_i is the corresponding radius of gamma-scan position with respect to the column center and R_o is the radius of the column. Negative R_i/R_o values designate chord positions to the left of the column center, and a zero index value designates the column center position.

A zero gamma-ray scan of the reactor filled with kerosene only was also carried out at zero gas and liquid flow. The data acquisition procedure for the zero gamma-ray scan was the same as that for the 25 wt% coal char in kerosene system. These data are necessary in order to calculate the various phase holdups.

Data Analysis

The average of five gamma-ray scans is used in the phase holdup calculations.

The volume fraction of the catalyst is calculated from measurement of the bed height and from the mass of catalyst in the reactor:

$$\epsilon_c = \frac{M}{\rho_{cAH}} \quad (2)$$

The volume fraction of liquid can then be calculated using gamma-ray scan data:

$$\epsilon_l = \frac{(\ln \frac{I_l}{I_m})/d - \mu_c \rho_c \epsilon_c + \mu_l \rho_l}{\mu_l \rho_l + \frac{\mu_f \omega_f \rho_l}{(100 - \omega_f)}} \quad (3)$$

The volume fraction of coal char fines is calculated from the known concentration of fines:

$$\epsilon_f = \frac{\omega_f \rho_l \epsilon_l}{(100 - \omega_f) \rho_f} \quad (4)$$

The volume fraction of gas is then calculated from the difference:

$$\epsilon_g = 1 - \epsilon_c - \epsilon_l - \epsilon_f \quad (5)$$

In the dilute phase above the catalyst bed, ϵ_c is set to 0.0, and the same equations are solved.

Results and Discussion

The average gamma-ray scan data presented in Table III and Table IV are average values of five scans taken at each radial position. Phase holdups calculated from these scan data are presented in Table V. The averaged gamma-ray scan data were found to have less than 4% standard deviation, which translates to a maximum of about 5% standard deviation in the calculated liquid holdups. The gas holdups, calculated from the other phase holdups, do not vary significantly across the reactor diameter except for those data at $R_1/R_0 = 0.75$. The phase holdups at this radial position are

affected by the presence of a 1" diameter slurry recycle line. The phase holdup data at this radial position are not used in this evaluation. Therefore, the gas and liquid holdups shown in Table V are essentially constant across the reactor diameter within experimental error. The results show that the gas and liquid holdups in both the dense and dilute phases do not vary significantly across the reactor diameter at the gas and slurry velocity ranges tested.

The radial uniformity of gas and liquid holdups does not necessarily imply that the bubble sizes across the reactor are constant. Based on gas tracer tests carried out in the earlier contract (1), it was concluded that large bubbles having a high rise velocity were traveling up the center of the column while smaller bubbles were traveling down at the walls. Although these earlier gas tracer results and the current radial scanning results appear to contradict each other, some interesting conclusions can be drawn from the results.

The gamma-ray scans, which cut horizontally across parallel chords of the column at a given elevation, cannot discriminate between upflowing and down-flowing bubbles. The gamma-ray scan sees an average density. Even though the bubble size distribution varies across parallel chords of the column, the average gas and slurry volume fractions are essentially constant.

Table V also shows that the gas and slurry holdups do not vary axially within the bed or dilute phase. The gas holdup is about 10% higher in the dilute phase than in the bed. These results have large implications for our understanding of the heat and mass transfer processes as well as chemical reactions taking place in the reactor, all of which depend strongly on the bubble size distribution.

V) AMOCO COLD-FLOW EXPERIMENTS: AXIAL SLURRY DISPERSION

Background

Gas- and liquid-phase mixing have been studied extensively in gas/liquid fluidized systems but to a much lesser extent in gas/liquid/solid systems. Several review articles [Wen and Fan (9); Shah, et al. (10); Ostergaard (11); and Bischoff (12)] have summarized the mixing characteristics of various types of multi-phase reactors. It is clear from these literature surveys that axial mixing within the different phases has not been adequately studied for the gas/liquid/solid fluidized system. No published data exist for liquid-phase and gas-phase mixing in a three-phase fluidized system where the liquid phase contains suspended fine particles such as the unreacted coal and mineral particles in the H-Coal ebullated bed reactor. Backmixing, recirculation, and other transport mechanisms such as bubble coalescence complicate the nature of gas-, liquid-, and solid-phase motion.

Recently, Bickel and Thomas have studied solid-phase (catalyst) mixing in the H-Coal PDU reactor during a 28-day steady-state operation using Kentucky high-sulfur coal (13). The catalyst residence time distribution was determined

by tracking the radioactivity of daily withdrawn ^{60}Co -tagged American Cyanamid HDS-1442A CoMo catalyst samples from the PDU reactor. The Peclet number was calculated to be 0.45 based on an axial flow dispersion model. This indicates that catalyst mixing in the H-Coal PDU is expected to be well mixed after an initial period.

Amoco's contract tasks include characterizing the gas- and liquid-phase dispersion using the cold-flow unit. Gas-phase mixing experiments were completed in 1980 under the first H-Coal contract (1) using argon-41 as a radioactive tracer. A circulation model consisting of completely mixed tanks in series with a recycle flow was proposed (1). This model is in agreement with visual observation of the cold-flow unit, where small gas bubbles were seen flowing down along the outer walls. Liquid-phase mixing experiments were carried out under the current contract. Experimental results and a one-parameter dispersion model are presented here.

Review of Liquid Dispersion in Multi-Phase Fluidization

Axial liquid dispersion data in the literature for gas/liquid, gas/slurry, and gas/slurry/solid fluidization are summarized and compared. The effects of various independent system variables (gas velocity, liquid velocity, particle size, column diameter, and fluid properties) on the extent of liquid dispersion are discussed in this section.

Gas/Liquid Systems.--The subject of axial mixing characteristics in a vertical bubble fluidization column has been extensively studied and was recently reviewed by Joshi (14), Bischoff (12), and Shah, et al. (10). In general, the liquid dispersion coefficient (D_L) can be correlated with a characteristic velocity and the column diameter (d_t), assuming the column has a large length-to-diameter ratio. A majority of the investigators agree that whereas the dispersion coefficient is a strong function of the gas velocity, it is essentially independent of the liquid flow rate. Therefore, the following model is often used to correlate results:

$$D_L = a u_g^b d_t^c$$

Table VI summarizes the liquid dispersion coefficient correlations derived by various investigators.

In an attempt to develop a unified model for axial mixing in a multi-phase fluidized reactor, Joshi (14) concluded that the liquid circulation velocity could be used to correlate all experimental dispersion data. The liquid circulation velocity is based on the multiple circulation cells theory in a bubble column and can be calculated from the energy balance. Although it uses liquid circulation velocity, Joshi's unified model (see Table VI) is inherently related to the gas bubble rise velocity. For all practical purposes, gas velocity is by far the most popular characteristic velocity used in liquid dispersion correlations.

Effect of Liquid Properties: The effect of liquid viscosity on the axial dispersion coefficient is not completely resolved in the literature. Cova

(23) and Alexander and Shah (24) reported that the axial dispersion coefficient is independent of the viscosity and surface tension of the liquid. Pilhofer, et al. (26) and Hikita and Kikukawa (19), on the other hand, found that the axial dispersion coefficient is dependent upon liquid viscosity (see Table VI). Their conclusion was derived, however, from a much larger liquid viscosity range (20-200 cp). Cova and Alexander's conclusion was derived from experimental results obtained in a smaller liquid viscosity range (1-15 cp).

The effect of liquid density on the axial dispersion coefficient was studied by Cova (23) using small-diameter columns (less than 1.8" ID). The results indicate that an increase in liquid density increased the dispersion coefficient.

Gas/Slurry Systems.--Liquid-phase dispersion in the gas/slurry system has been studied to a lesser extent, and consequently, fewer liquid-phase dispersion coefficient correlations have been derived. Published liquid dispersion models are summarized in Table VI.

An interesting and important effect of introducing fine suspended solid particles to the gas/liquid system is the enhancement of bubble coalescence which would be expected to enhance the axial liquid dispersion. The fine solid particles suspended in the liquid constitute a pseudo-homogeneous fluid of higher density and viscosity than the pure liquid. The increased viscosity is known to promote bubble coalescence (1).

The nature of the pseudo-homogeneous fluid was illustrated by Kato, et al. (34) in evaluating the magnitudes of liquid- and solid-phase dispersion in a three-phase fluidized column. Kato, et al., studied liquid and solid dispersion using various types of fines ranging from 53 to 250 microns and with particle densities ranging from 1.0 to 9.0 g/cm³. Experiments were carried out with different diameter columns and with fines concentrations up to 20%. The significant conclusions of these studies was that the longitudinal dispersion coefficient for the fines in the gas/slurry fluidized bed is the same as that for the gas/liquid. These results imply that the fines in the liquid phase are homogeneously mixed. Vasalos, et al., (1) also found that the concentration of their coal slurry was uniform throughout their three-phase system.

Axial mixing in the bulk of the bed is contributed mainly by wake formation and shedding. These wakes, carrying appreciable solids, contribute to the solid mixing. Therefore, for fine solids ($d_p < 0.1$ mm), there is little difference between axial dispersion coefficient for the solid and the liquid phases.

Effect of Liquid Velocity: Whereas in the gas/liquid system the liquid dispersion coefficient is dependent only upon the gas velocity, the axial liquid dispersion coefficient in gas/slurry fluidization is also dependent upon the liquid (or slurry) velocity. Experimental data in the literature (14,34,35,37) indicate that the liquid dispersion coefficient increases with increasing gas velocity but decreases with increasing liquid (or slurry) velocity. The magnitude of the effect of liquid (or slurry) velocity on

axial liquid dispersion depends on the gas velocity and the solid particle size. A valid approach for evaluating the liquid velocity effect is to compare the Darton and Harrison (3) drift flux values for determining bed behavior (bubbly or turbulent) as a function of liquid (or slurry) velocity. Data obtained from the present work show that a higher liquid velocity suppresses the churn turbulent behavior of the system, resulting in the reduction of liquid mixing. Liquid dispersion coefficient models proposed by other investigators (see Table VI) further quantify the magnitude of the liquid velocity effect.

Gas/Slurry/Solid System.--The effect of adding fluidized coarse, solid particles (such as catalysts) to a gas/liquid or gas/slurry system is quite different from that of adding fine, solid particles to a gas/liquid system. In the gas/slurry/solid case, the liquid dispersion coefficient decreases relative to a gas/liquid system under the same flow conditions, due to bubble disintegration by the large solid particles. The disintegration causes a reduction in bubble size and rise velocity, resulting in less axial dispersion.

Effects of independent variables (gas and slurry velocities and the diameter of the column) upon liquid dispersion are similar to those discussed in the gas/liquid and gas/slurry systems.

Effect of Solid Particle Size: Analysis of liquid dispersion coefficients reported by various investigators (30,31,32,39), including data obtained from this work, indicates that liquid dispersion coefficients decrease as the particle diameter is increased. Ostergaard (31) measured bubble sizes in a three-phase system and reported that bubble sizes in beds composed of 1 mm particle size were one order of magnitude larger than bubble sizes in beds composed of 6 mm particles. Ostergaard's dispersion coefficient data appear much higher than those reported by others (see Figure 17). The use of the larger diameter column may have contributed to this high result. Discrepancies appearing at the low gas velocities may be due to the differences in distributors used in these studies.

Reacting Systems.--Numerous data exist in the literature, and many models are available for predicting the dispersion coefficient for non-reacting two-phase and three-phase systems. However, application of these data and models to predict real system performance is still inadequate. For instance, Panvelker, et al., (38) reported that the liquid dispersion coefficient predicted by the application of the Deckwer, et al., (16) correlation was more than ten times higher than that which was actually measured from a solvent refined coal (SRC) dissolver. It is clear that further study of liquid (slurry) phase mixing is needed with systems more closely simulating both the physical properties and operating conditions of industrial systems.

A recent study of the effect of vertical alignment of the column on liquid axial dispersion indicated that axial mixing can be reduced significantly if the column is well aligned (40). Although the study was conducted in a liquid/liquid spray column, it may have significance for the H-Coal system

and other three-phase systems. The effect of reactor internals also needs further investigation.

Tracer Selection

A suitable liquid tracer is necessary to measure the extent of dispersion in the liquid phase. A number of tracers and techniques have been used to obtain residence time distribution (RTD) functions for chemical reactors. In this particular case, the purpose is to obtain an RTD for the slurry phase of the gas/slurry/solid system under non-reacting conditions.

The criteria for a suitable tracer include 1) little or no absorption by the coal char fines and the catalyst for a short period of time (30-60 minutes); 2) good miscibility in kerosene; 3) accurate determination of the tracer concentration in the liquid; and 4) good chemical and physical stability. These criteria were used to determine the choice of a tracer for our liquid dispersion experiments.

Examples of experimental techniques prior to 1980 are reviewed in several books and review articles. Three-phase experiments are discussed in Shah's text (45). A table of commonly used tracers is included.

Wen and Fan (9) divided liquid-phase experiments into categories by tracer method. Four classes were indexed: conductivity salts, color and UV dyes, radioactive tracers, and titrations. Forty-five papers are included, eight of them dealing with fluidized beds.

Shah, Stiegel, and Sharma (10) give a more extensive review of experimental work, indexed by reaction system. Twenty-eight examples of liquid-phase tracers from the literature are given, with three citations for work in three-phase fluidized beds, but no additional tracer techniques are presented. Other review articles covering the earlier development of tracer methods and mixing models include Ostergaard (5) and Bischoff (12).

Most of the liquid dispersion studies reported in the literature have used clear liquids and non-porous solid particles. Air, water, and glass balls are the most popular components used by the investigators. For these systems, conductivity salts, UV and visible dyes, and radioactive chemicals are equally effective as tracers. These tracers and techniques, however, are generally not suitable for our three-phase system involving a coal char fines/kerosene slurry phase. This is primarily due to the physical properties of the slurry. The coal char/kerosene slurry is essentially non-conductive and opaque. Furthermore, the coal char fines in the slurry are porous particles and strongly absorb certain chemicals. Potential liquid tracers in each category were evaluated in order to select the best qualified liquid tracer for this study.

While conductivity salts are normally used in the air/water systems, additives are available that can increase the conductivity of normally non-conductive organic liquids. These additives are miscible organic salts and are detectable

in the ppm range by using a conductivity probe. The disadvantage of the conductive salt is that an invasive conductivity probe in the fluid may cause flow disruptions. A possible complication of conductivity measurement also exists due to gas bubbles adhering to the probe.

Organic soluble dyes are also available from a number of sources and have been used in studying mixing of clear liquids. UV and visible dyes offer the advantage that the detector is located outside the reactor and will not disturb the flow. The disadvantage is that the dye method requires the use of a transparent system. This is incompatible with coal char fines used in the H-Coal fluid system.

Use of radioactive tracers is an attractive experimental technique, because the tracer concentration can be measured non-invasively. Unfortunately, use of radioactive tracers generates large amounts of low-level radioactive waste for disposal. Therefore, radioactive tracers were excluded from consideration for this task.

Three potential tracers and their detection methods were selected for further experimental evaluation:

<u>No.</u>	<u>Tracer for Liquid Mixing</u>	<u>Tracer Detector or Analytical Method</u>
1	Du Pont Stadis-450 (An Oil-Base Conductivity Improver)	Conductivity Cell
2	Mobay Chemical Blue ZV (An Oil-Base Blue Dye)	Visible Light Absorbance
3	Thiophene	Sulfur Analysis by X-ray Fluorescence

Bench-Scale Experiments and Results

Du Pont Stadis-450.--Stadis-450 is a jet fuel additive currently marketed to reduce static hazards by making the hydrocarbon fluid electrically conductive. According to the manufacturer, the resulting conductivity of the mixed liquid is a function of the additive concentration. Therefore, in each of the tests, a known amount of the additive was introduced into a mechanically stirred vessel which contained kerosene or a kerosene/coal char slurry. A meter measured the resulting electrical conductivity, which was recorded as a function of mixing time. Du Pont Stadis-450 absorption by HDS-2A catalyst was also tested separately. In these experiments, catalyst particles were first soaked in kerosene before the absorption rate tests were carried out.

Results from these tests showed that over a limited range, Stadis-450 concentration and electrical conductivity were related. However, when kerosene slurried with fully soaked coal char was tested, the Stadis-450 was strongly absorbed by the char. A rapid decrease in conductivity was observed after the additive was injected into the solution. The rate of absorption of the

additive seems to be affected by its concentration in the slurry. Below 1,500 ppm, Stadis-450 is consistently absorbed at a high rate. Above 1,500 ppm, it is still being absorbed at a high rate for the first five minutes. This is then followed by a slower absorption.

The phenomenon of absorption by HDS-2A catalyst is quite different. The conductivity increased initially for almost 20 minutes, indicating little or no absorption by catalyst particles in this period. The catalyst particles absorb Du Pont Stadis-450 additive at an insignificant rate compared with that of the coal char fines.

Due to the consistently fast absorption of the additive by the coal char fines, Du Pont Stadis-450 and the conductivity detection method were judged to be not suitable for the liquid mixing experiments.

Mobay Blue ZV Dye.--A saturated solution of this dye was used as a tracer in visible light absorptivity tests. The experimental procedure for these tests was similar to that used for the conductivity experiments. Mobay Chemical Blue ZV dye was added to kerosene or kerosene/coal char slurries to produce a known concentration. Spectro-photometric absorption was then used to correlate signal attenuation with concentration. A linear correlation between dye concentration and its light absorption was established satisfactorily with pure kerosene. This method failed, however, to give a definite correlation between dye concentration and absorptivity with the slurry system. Since the inclusion of the coal char fines is necessary to model the H-Coal reactor, this method was also discarded.

Thiophene.--Thiophene was investigated as a candidate liquid tracer, because it is miscible with kerosene, and because it can be accurately detected by gas chromatography or by x-ray fluorescence. Thiophene tests were also run by mixing a known amount of thiophene with well-stirred kerosene/coal char and kerosene/coal char/catalyst slurries. The mixtures were separated by filtration, and the liquid was analyzed for sulfur content by x-ray fluorescence. Thiophene was absorbed at a very slow rate by coal char fines. As shown in Figure 18, there was practically no absorption in the first 90 minutes. Additional absorption tests were carried out up to 180 minutes' mixing time, and results indicated insignificant absorption.

Thiophene absorption by the HDS-2A catalyst particles was also investigated. No appreciable absorption was evident up to 180 minutes' mixing time. Hence, thiophene meets the most important criteria for a liquid tracer--i.e., little or no absorption by coal char fines and catalyst. In addition, thiophene is readily miscible with kerosene and is chemically stable in the solution. Therefore, thiophene has successfully met all four criteria required for a good liquid tracer.

Pilot Plant Testing of Thiophene as a Tracer

Based on the encouraging bench-scale results from using thiophene as a tracer material, scoping experiments were carried out using the H-Coal cold-flow

pilot plant. The objectives of these tests were to gain experience in obtaining the tracer C-curve (concentration versus time curve) at different positions in the fluidized bed and to gain some insight into how best to design the equipment for the tracer injector and the continuous-flow sampling device.

Results of pilot plant liquid mixing tests are presented in Figure 19. The input pulse curve (lower curve) and the resulting downstream C-curve (upper curve) were successfully obtained when 2 cc of the thiophene was injected into the reactor at the slurry inlet port. The extent of liquid dispersion can be estimated based on the data derived from these C-curves. Based on these pilot plant test results, it was concluded that thiophene is a suitable liquid tracer and was therefore selected for the liquid mixing experiments.

Equipment

Liquid-phase dispersion experiments were carried out in the Amoco H-Coal cold-flow pilot plant by injecting a pulse of thiophene at the slurry inlet port and measuring thiophene concentration as a function of time at two locations in the dense-phase region and at two locations in the dilute-phase region. The pilot plant has been modified to include the additional equipment and instrumentation necessary for liquid-phase dispersion tests. The following sections give a description of equipment and experimental procedure used to obtain liquid dispersion test data.

A detailed description of the Amoco H-Coal cold-flow pilot plant and its operation was presented in a previous report (1). A schematic diagram of the pilot plant is shown in Figure 20. A description follows for the equipment added to the pilot plant in order to perform liquid-phase mixing experiments.

- 1) Continuous-flow slurry sampling device: Located just below the slurry sampling probe outlet, this automatic sampling device holds twenty-five 20-cc glass vials evenly distributed around the circumference of a vial tray. The tray rotates at 0.5 RPM, giving a 4.8-second sampling time per vial. Figures 21 and 22 show the detailed mechanical design of the continuous-flow slurry sampling system.

Four identical sets of the continuous-flow sampling device were built. They were installed at the four spool pieces on the fluidization column. The lower two sets receive samples at elevations from within the fluidized bed (dense-phase region), whereas the upper two sets receive samples at elevations from within the dilute phase.

- 2) Constant-volume tracer injector: Constructed of stainless steel material, the injector is a 2-cc, precision-bore barrel with an O-ring sealed plunger. A 200-cc liquid tracer reservoir is connected to the injector's inlet port to provide an adequate supply of liquid tracer. The injector is installed at the slurry inlet line leading to the calming section of the fluidized column. A drawing of the tracer injector assembly is shown in Figure 23.

- 3) Reactor slurry sampling probe: Inserted into the reactor through each spool piece, the sampling probe is equipped with an adjustable tube which can be positioned at any radial position. A drawing of the sample probe assembly is shown in Figure 24.
- 4) Automatic continuous-flow sampling system: The system consists of a programmable controller which controls the rotational movements of the four continuous-flow slurry sampling devices. Each device consists of a synchronous motor and a limit switch. The switch serves a dual function in that it provides a starting position for the vial tray and also stops the vial tray movement after one complete revolution. Each device is provided with a start pushbutton and a starting position indicator on the instrumentation panel board located in the control room. There is also an overall start button which is mounted on the pilot plant structure which controls the initiation of automatic sampling sequence.

The controller logic is such that a sampling sequence cannot be started unless all four vial trays are at the starting position. Once the sampling sequence has been started, the automation controls the time delays between the start and stop of all vial trays. Based on the planned experimental slurry feed rate range (1-5 cm/sec) and on the assumption that the average linear slurry velocity will not exceed 10 cm/sec, the proper time delays for Tray Nos. 2, 3, and 4 can be calculated by dividing the distance between two sampling ports by the maximum linear slurry velocity. It was determined that a 15-second delay for the second tray and subsequent 10-second delays for the third and fourth trays are appropriate.

Pilot Plant Results and Discussion

The experimental plan consisted of four series of tests. Data collected from these tests have been processed according to the liquid mixing data analysis program (Appendix B). Liquid mixing parameters--Peclet number, and the dispersion coefficient--are presented in this section. Correlation of the Peclet number as a function of process variables was developed.

Data Collection.--The first series of tests, consisting of Runs 490-6 through -8 and Run 490-21, was designed to investigate the effect of gas flow rate on the liquid dispersion coefficient (or Peclet number). The nitrogen flow was varied from 0 to 4.57 cm/sec (0-0.15 ft/sec), while the slurry flow rate was controlled at 3.05 cm/sec (0.10 ft/sec). Slurry samples were continuously drawn from the reactor at the reactor wall for 4.8 sec/sample. In most cases 25 samples were taken per tray.

The second series of tests, consisting of Runs 490-10 through -16, was designed to investigate the variation of liquid dispersion across the reactor diameter. These tests were carried out under both the nominal H-Coal gas and liquid flow conditions and under zero gas flow conditions.

The third series of tests, consisting of Runs 490-17 through -20 and Run 490-23, was carried out to investigate the effect of gas flow rate on liquid dispersion at the center of the reactor.

The last series, consisting of Run 490-9 and Run 490-22, was intended to evaluate the effect of increasing slurry velocity on liquid dispersion.

Samples from Trays III and IV, located above the fluidized bed in the dilute phase, were also collected and analyzed in order to determine the dilute-phase liquid dispersion coefficient. Unfortunately, good-quality RTD curves could not be obtained, due to the large amount of liquid mixing occurring in this phase. The experimental concentration/time distribution data (C-curves) have been presented in quarterly progress reports (43,44). Figures 25 and 26 contain an example of a C-curve plot and a logarithmic probability plot of cumulative area under the C-curve, respectively. The mean (first moment) and the standard deviation (second moment) of a normal distribution curve which plots as a straight line on probability paper are derived from the fiftieth and eighty-fourth percentile points.

Data Analysis.--Various correlations were tested involving gas and slurry velocities and radial position. In general,

$$Pe = A + [B + D (U_g - 0.1) + E (R)] \exp (-C U_g) \quad (5)$$

1) Liquid Dispersion Coefficient: Experimental data were processed according to the liquid dispersion data analysis program (Appendix B). Liquid mixing parameters--Peclet number and dispersion coefficient--are presented in Table VII.

2) Liquid Dispersion Correlations: The liquid dispersion results were used to develop a correlation between the liquid dispersion coefficient (or the Peclet number) and the process variables such as superficial gas and slurry velocities. Least squares curve-fitting methods were used to evaluate a variety of correlation formulas. Radial position dependency was also assessed.

As seen in Table VIII, the least squares analysis results for Correlation 1 indicates that there is no significant effect of radial position on the Peclet number. Consequently, the term representing the radial position (R) was excluded from other correlations. Statistical results for Correlation 1 and 2 show that the slurry velocity effect is insignificant within the ranges of gas and slurry velocities employed for these liquid dispersion experiments. The conclusion is that the Peclet number is not a function of the radial sampling position but is a strong function of superficial gas velocity, U_g .

Correlations 3 and 4, which both adequately fit the data for the nominal H-Coal slurry velocity (approximately 0.1 ft/sec) in the superficial gas velocity range from 0 to 5 cm/sec (0-0.164 ft/sec), are

$$\text{Correlation 3: } Pe = 5.05 + 42.91 \exp (-35.48 U_g) \quad (6)$$

and

$$\text{Correlation 4: } Pe = 40.26 \exp (-17.83 U_g), \quad U_g \text{ in ft/sec} \quad (7)$$

Correlation 4 implies that $Pe \rightarrow 0$ when $U_g \rightarrow \infty$, while Correlation 3 implies that $Pe \rightarrow 5.05$ when $U_g \rightarrow \infty$. Correlation 3 fits the data better at $U_g = 0$ (see Figure 27) and gives an overall better fit to the experimental data (see Tables IX and X). Figure 27 compares the experimental results with both correlations.

3) Application of the Dispersion Model: Using Correlation 3, the C-curve for the second sampling tray can be reconstructed based on the C-curve data (from Tray I) and the process conditions (U_g and U_1) for that run. The following procedure is employed:

A) Estimate Pe for a given U_g :

$$Pe = 5.05 + 42.91 \exp(-35.48 U_g), U_g \text{ in ft/sec} \quad (8)$$

B) Estimate σ according to the dispersion model:

$$\frac{\Delta \sigma^2}{T_m^2} = \frac{2}{Pe}, \text{ where } T_m = \frac{L}{U_1} \quad (9)$$

C) Calculate the log normal variance s using the conversion formula:

$$S_i = \sqrt{\frac{\sigma_i^2}{\ln^2 + 1}}, \text{ where } i = 1 \text{ and } 2 \quad (10)$$

4) Calculate tracer concentration versus time profile using the following equation:

$$C = C_0 + \frac{Q}{T \cdot s \cdot \sqrt{2\pi}} \exp\left[-\frac{(\ln T - \ln T_m)^2}{2s}\right] \quad (11)$$

where Q = Amount of tracer injected into the reactor.

C_0 = Initial tracer concentration (i.e., Tray I curve).

Run 490-17 data were used as an example to demonstrate the validity of the proposed Correlation 3 and the calculation procedure. The predicted C-curve compares reasonably well with the experimental data, as can be seen in Figure 29.

4) Tracer Material Balance: The amount of thiophene calculated from the tracer C-curve and the amount of thiophene actually injected into the system are compared and shown in Figure 28. While reasonable thiophene material balances were obtained for most of the runs (within $\pm 10\%$ of actual injection amount), several runs showed that the calculated amount of thiophene was lower than that actually injected.

The x-ray fluorescence analytical precision (± 15 wppm) accounts for a part of the thiophene imbalance. The fluctuant tail of the C-curve contributes the other part of the error.

The effect of thiophene imbalance upon the liquid dispersion results is not significant, because the amount of axial mixing is estimated from the relative change of variance of the two RFD curves. Application of the method of moments to estimate the dispersion coefficient and its sensitivity upon parameters such as the mean and variance of the tracer C-curve is discussed in Appendix B.

Conclusions

The amount of slurry-phase axial dispersion occurring in a cold-flow system simulating the HRI PDU reactor was studied. Slurry-phase axial dispersion coefficients were obtained for the fluidized-bed phase of the ebullated bed reactor. Typical values of the slurry dispersion coefficient at nominal H-Coal operating conditions are in the range of 70-130 cm²/sec. Peclet numbers range from 3.6 to 9.9. This represents a significant degree of backmixing in the reactor. A correlation was developed which relates the axial Peclet number to the superficial gas velocity. Application of this correlation in conjunction with the axial dispersion model to predict residence time distribution information is demonstrated.

VI) BUBBLE DYNAMICS

Results from our earlier work (1) showed that the presence of fines in the slurry had a strong influence on the gas flow behavior in the three-phase system. Increasing the gas velocity normally increases the bed expansion slightly when liquid phase is a pure liquid. However, increasing the gas velocity when fines are present in the liquid (i.e., slurry) can, in fact, result in a slight bed contraction. This matter of bed contraction is discussed further in Section III. A very complex process of bubble coalescence and breakup is occurring in a cold-flow fluidization system. The fluidization occurring in a chemically reacting system is even more complex.

Bubbles are coalescing and growing as they rise through the system. The fines in the slurry are promoting this coalescence, due to their presence on the bubble surface through a complex but poorly understood process. Bubble coalescence is also promoted by the higher viscosity caused by the fines in the liquid. Bubbles break up if they grow through coalescence to a size larger than their maximum stable bubble size. The catalyst particles also promote bubble breakup. It is clear that a better understanding of the bubble dynamics occurring in the H-Coal system is essential.

Because of the complexity of the three-phase system being studied, it was anticipated that development of a quantitative technique and subsequent data collection would be difficult. Therefore, development of three techniques for investigating different aspects of the bubble dynamics was carried out in parallel: 1) A light beam probe was developed which involves passing light from a helium-neon laser through the three-phase system and monitoring the transmitted light intensity; 2) A holographic technique was developed to capture three-dimensional images of bubbles rising through the three-phase system; 3) A resistivity probe was developed to measure bubble size and bubble frequency.

Due to the nature of the liquids chosen in this study on bubble dynamics, very small bubbles were observed within the cold-flow system. These bubbles were smaller than originally anticipated when this project was first proposed. This resulted in the inability to use laser-Doppler anemometry, as was originally planned. Also, the development of the holographic technique required extensive efforts and is only now becoming a useful tool. Nonetheless, significant progress has been made in the development of three quantitative techniques, and important results are presented and discussed.

Experimental System

Description of the Apparatus.--The experimental setup consisted of a three-phase fluidized bed operating in a 6" diameter vertical column. A schematic diagram of the apparatus is shown in Figure 30. The vertical column was constructed using four pieces of glass pipe, each section being 2 ft in length. Carbon-steel spool pieces placed between the glass sections were radially drilled and fitted with threaded pipe connectors, thus providing points of access to the interior of the column. A diagram of the metal spool pieces is given in Figure 31.

The inlet distributor section, shown in Figure 32, was modeled after H-Coal pilot plant distributors and was constructed by Amoco Oil. It consisted of a carbon-steel mixing chamber, into which the gas and liquid flowed; a vertical exit pipe; and a slotted bubble cap. The gas/liquid mixture leaves the mixing chamber through a stainless steel screen spot-welded to the inlet of the exit pipe. From this point, the fluid mixture flows upward to the top of the bubble cap, where it is forced down and out through the slots and into the fluidized bed.

As shown in Figure 30, we see that when the gas phase passes through the inlet distributor, it first comes in contact with the liquid phase approximately 6' before entering the distributor section. This is the method of fluid injection used in the H-Coal process, thus allowing gas/liquid mixing to occur before reaching the reactor. Further bubble breakup occurs as the fluid flows through the screen in our cold-flow system and out the slots of the bubble cap.

The gas flow could be routed either into the liquid inlet pipe leading to the inlet distributor or directly into the column via the bottom-most spool piece and a gas sparger. The sparger was a length of 3/8" diameter copper tubing which spanned the diameter of the column. Five 1/16" diameter holes spaced approximately 1" apart served as a means of injecting relatively large bubbles (a few centimeters or so in diameter) into the bottom of the bed.

At the top of the column, the fluid mixture is separated into its gas and liquid phases using a spiral entrainment separator (see Figure 33). The liquid then flows through an overflow cup, down a return line, and into the holding tank, where it is recycled. The nitrogen gas used is simply vented to the atmosphere. A stainless steel screen is spot-welded to the overflow cup to prevent the entrainment of solids out of the column.

Fluids and Solids.--The initial solids used in the column were pyrex glass cylinders. These were nominally 2 mm in diameter and 5 mm long. The fluid used was a mixture of two organic solvents in a proportion such that the refractive index of the fluid matched that of the glass cylinders. The purpose of this was to make optical measurements possible within the bed. Property values of the constituent fluids and the mixture are given in Table XI.

In addition to the Pyrex glass cylinders, spherical glass balls with diameters of 1, 2, 3, and 5 mm were also used. This was done in order to determine the effect of particle size on bubble sizes. Basic data concerning the particles are provided in Table XII. The settled bed heights of the particles were approximately 70 cm.

Bed Expansion and Pressure-Drop Measurements

Bed Expansion Measurements.--Bed expansion measurements were made for various flow conditions ranging from a fixed bed at low liquid flow rates to a fully fluidized state in which the bed expanded by 120%. It was found that the bed expansion changed by such a small amount while varying the gas flow rate that this effect could be neglected. Therefore, in this section, bed expansion is assumed to be a function of liquid flow rate only. The minimum fluidization velocity was found to be 1.60 cm/sec. This represents the condition of incipient fluidization in which the solid particles are just supported by the fluid.

The average solid volume fraction was calculated using measurements of the average bed height and knowing the total volume of solids within the system. Results are shown in Figure 34. Assuming a homogeneous distribution of solids throughout the bed, it was possible to model the flow according to an empirical drift flux model as given by Wallis (51), based on the work done by Richardson and Zaki (49). The following model relates the average liquid flux, U_1 (cm/sec), to the average volume fraction of solids, ϵ_s :

$$U_1 = 17.58 (1 - \epsilon_s)^{2.208} \quad (12)$$

From this, we see that the terminal velocity of the solid particles is 17.58 cm/sec, which is consistent with theoretical predictions based on balancing the gravitational and drag forces acting on a single particle.

Pressure-Drop Measurements.--Void fractions of the gas phase (gas holdup) above and within the fluidized bed were determined from pressure-drop measurements (48). It was found that for low liquid flow rates, the gas holdup increased with both gas flux and liquid flux. Because some bubbles were generated by passage of both liquid and gas through an inlet distributor, it was concluded that the inlet distributor did not produce bubbles of a constant size. In fact, because of these studies, it is now felt that the design of the inlet distributor may be very important to bubble size distributions found within the fluidized bed. Results indicate that the inlet distributor was able to produce much smaller bubbles than those produced by the breakup of large bubbles when colliding with solid particles inside the fluidized bed.

Generally, the gas holdup within the three-phase region was found to be much higher than that above the bed. This can be attributed to increased accumulation of gas bubbles inside the bed, caused by collisions between the bubbles and solid particles. These collisions impede the flow of bubbles through the bed, thus increasing the gas holdup.

Light Beam Probe and Bubble Breakup Model

Description of the Light Beam Probe.--A new optical technique was developed to measure bubble sizes in the two-phase region above the catalyst bed (47). This new method can be used in flows of spherical bubbles with diameters greater than 120 microns. Implementation involves passing a narrow beam of light through the bubbly flow and monitoring the transmitted light intensity. Light from a helium-neon laser enters and exits the column through thin probes, shown in Figures 35 and 36. (The beam length was typically between 3 and 12 mm.) As a bubble passes between the probe tips, it blocks the light beam, as shown in Figure 37. The basic units of data are the rate at which each bubble blocks off the beam and the duration of the blockage. Figure 38 shows typical signals from the light beam probe. By statistically analyzing a set of such data, bubble size and average velocity distributions were determined. A typical bubble size distribution curve is shown in Figure 39.

Results of the Light Beam Technique.--Measurements made with the light beam probe showed the bubble size distribution to be highly dependent upon the size of the solid particles within the fluidized bed but relatively independent of the gas and liquid flow rates, at least for the low gas velocities employed here. The latter is illustrated in Figure 40, where the gas and liquid flow rates were varied without significant change in the Sauter mean bubble diameter. Initially, 2 mm x 5 mm cylindrical particles were used, but these were later replaced with glass spheres ranging in size from 1 to 5 mm in diameter. Average results of bubble size measurements are listed in Table XIII.

Equilibrium Bubble Size Model.--Dimensional analysis was applied to the breakup of bubbles passing through the fluidized bed. Based upon the data, a model was constructed of the equilibrium bubble size distribution in a bed of particles of known size. This model relates the dimensionless mean bubble diameter to the Eotvos number as follows:

$$d_m/d_3 = 1.544 (\sigma/\Delta\rho d_3^2 g) + 0.143$$

$$\text{where } \Delta\rho d_3^2 g / \sigma = \text{Eotvos number.}$$

Gas holdup measurements made both within the three-phase fluidized bed and above the bed in the two-phase gas/liquid region are shown in Figures 34A through 34D. The data show the void fraction within the three-phase region to be higher than that found above the bed in the two-phase region at very low gas rates. In Figure 34A the difference between the two values is approximately equal to the void fraction which accumulates inside the bed. This suggests the bubbles are not allowed to flow freely but are trapped inside the bed. Visual observations of the bubbles tend to confirm this hypothesis. The presence of closely packed solid particles tends to decrease the average bubble velocity due to collisions.

In Figures 34B through 34D we see that increases in both liquid and gas flow rates tend to narrow the difference between the two curves. This suggests the bubbles find less resistance to flow when the bed is expanded, thus behaving more like bubbles flowing through liquid alone. These interesting results complement those presented earlier in Section IV (Table V) where gas

holdup in the dilute phase at higher gas velocities was found to be about 10% higher. A plot of the dimensionless mean bubble diameter versus the inverse of the Eotvos number is shown in Figure 41.

Very little information was found in the literature to test the validity of this model. Lee (46), however, does give a mean bubble diameter of 0.24 cm in a water-fluidized bed of 0.6 cm diameter glass particles. The data point determined from this information is also included in Figure 41. It fits quite well, which is surprising, considering the effects of viscosity have been neglected.

Bubble Breakup and the Critical Particle Size.--The breakup of large bubbles to form an equilibrium bubble size is believed to be dependent upon the solid particle size. Yet, it was observed that the larger particles were far more efficient at breaking up large bubbles than were the small particles. If we assume that when a particle passes through a bubble it strips away a volume of gas approximately equal to its own volume, then to be consistent with our model, there exists a critical particle diameter below which large bubbles will not be broken up efficiently. By equating the particle size with the mean bubble diameter, a critical particle diameter of 1.89 mm is determined. This value is consistent with the data, but it could be argued that reasonably efficient breakup of large bubbles requires a particle size somewhat greater than 2 mm.

A similar analysis can be done for water-fluidized beds by substituting the density and surface tension values for water (listed in Table XIII) into the equilibrium bubble size model. By equating the particle size with the mean bubble diameter, a critical particle diameter of 2.9 mm is determined for water-fluidizing a bed of glass particles. This estimate of a particle size required for efficient breakup of bubbles is in good agreement with literature summarized by Vasalos, et al. (1), from which it appears that the critical particle size in water (assuming a solid density of about 2.5 gm/cm³) is between 3 and 4 mm.

Holography

Description of Holographic Technique.--Holography was used to capture three-dimensional images of bubbles flowing within the dense-phase region. Pulses of light from an Nd:YAG laser were used to illuminate the test section and form holograms for later analysis. The short duration of the pulses allows an instant of time to be captured on a holographic plate. Figure 42 shows the beam paths and optical arrangement required to form an off-axis hologram. When first entering the test section, the laser light passes through a diffusing plate. This is done to create a more uniform backlighting against which the bubbles are viewed, providing better clarity and definition of the bubbles in the hologram.

When constructing a hologram, the room containing the equipment must be totally dark, so that only the illumination from the laser strikes the holographic plate. After inserting a new plate into the plate holder, the laser is pulsed once, causing the sizes and positions of bubbles within the viewing volume to be permanently preserved. The plate is then developed and allowed to dry before reconstruction and analysis.

Holographic Analysis System.--To view the three-dimensional image contained on the holographic plate, the output from a helium-neon continuous-wave laser is used to retrace the path of the reference beam. As shown in Figure 43, a real image is formed in front of the holographic plate. A video camera is then used to focus on a plane inside the three-dimensional image and display this plane on a video monitor. By attaching the camera to a traversing mechanism, it is possible to change the focal plane remotely and to measure the radial position of the bubbles within the viewing volume.

A digital electronic circuit was designed to superimpose a thin horizontal and a thin vertical line on the video monitor's image from the video camera. The position of these lines on the screen can be changed while being monitored by a computer. By properly calibrating the magnification of the images displayed on the screen, it is possible to use this horizontal/vertical line generator to measure distances across the screen. This aids in the determination of bubble sizes from holograms. Figures 44 and 45 detail the circuitry used in the horizontal/vertical line generator.

Results.--The optical bench and the holographic analysis system took several years to design and build, so that the results obtained to date are limited. However, the technique has proved to be useful in determining bubble sizes and local void fractions. This technique offers an advantage over intrusive probes in that it dispels any doubts about the actual size and shape of bubbles inside the viewing volume.

Results from an analysis of one typical hologram are shown in Figures 46, 47, and 48. Although shown as a histogram plot, the actual bubble sizes were measured individually with better than ± 0.01 mm precision. Such results compare favorably with those found via the earlier mentioned light beam probe.

Resistivity Probe

A resistivity probe with a DC power supply was developed and used to measure bubble sizes within the three-phase fluidized bed (7). Unfortunately, due to the nature of the liquid phase, many of the gas bubbles in the three-phase fluidized bed were too small to be detected by the probe. Quantitative results in the three-phase system thus could not be obtained. However, the distribution of the gas phase was studied qualitatively in the two-phase region above the bed. It was found that the bubbles were distributed more uniformly at higher liquid and gas flow rates.

Although the probe was unable to be properly calibrated so as to provide accurate bubble size measurements, it did adequately measure the bubble frequency, defined as the number of bubbles that contact the probe tip per unit time. Such a probe could be employed in an actual H-Coal reactor to detect flow regime changes, presence of large bubbles, and channeling of the gas phase. For the probe to work properly, the resistivity of the mixture must be below 10^6 ohms/cm³. This condition is likely to exist at the operating pressures and temperatures of an actual H-Coal reactor.

A schematic diagram of the resistivity probe and associated electronics is given in Figures 49 and 50. Further details of the probe are given by Tsung (7).

Summary

Several new and powerful techniques for measuring bubble sizes and gas holdups were developed and shown to be useful. The narrow light beam probe is a very practical tool for measuring spherical bubbles greater than 120 microns. Holography is capable of determining the exact size, shape, and position of bubbles within some viewing volume. Although it does not provide absolute measurements, the resistivity probe developed during this study can most likely be used in actual H-Coal reactors to determine bubble frequencies and qualitative measurements of the flow regime.

Several interesting results were found during the course of this investigation. First, the design of the inlet distributor was determined to play an important role in the breakup of bubbles initially entering the fluidized bed. It was observed that smaller bubbles were generated by the inlet distributor than could be produced by the breakup of large bubbles by the solid particles within the bed. Second, the volume fraction of solids within the bed was found to be dependent mainly upon the liquid flux and could be predicted by an empirical drift flux model.

An equilibrium bubble size model was developed based upon data gathered by the light beam probe. This model relates the Sauter mean bubble diameter to the solid particle diameter, the solid/liquid density difference, and the surface tension of the fluid. From this model, a critical particle size was also determined at which efficient breakup occurs.

Recommendations

After completing this study of a three-phase fluidized bed, it is apparent that much more basic research is needed to better characterize the dynamics of such a system. The equilibrium bubble size model should be tested by varying the surface tension and the solid/liquid density difference to see if the model accurately predicts the Sauter mean bubble diameter. Also, the effect of liquid viscosity should be determined, since the model contains no viscosity term.

Because of the added complexity involved, this study did not look into the effect of using a slurry other than liquid alone to fluidize the solid bed. Such a system will probably have a different bubble size distribution due to the presence of these small particles. The effect of slurry particles should then also be studied in future work.

Since the holographic technique took several years to develop and is only now useful as a measuring tool, it is felt that further tests should be done to make use of this specialized technique. For example, because large bubbles are not generally spherical, as are small bubbles, additional studies regarding the shape and size distribution of large bubbles would be of interest. Ultimately, the holographic technique has the potential to provide insight into the nature of the wakes trailing behind the bubbles.

Last, it is felt that the design of the inlet distributor should be studied to determine how the initial bubble size distribution entering the reactor can be controlled for maximum reaction to take place.

VII) HYDROCARBON RESEARCH, INCORPORATED, PDU EXPERIMENTS

A series of fluid dynamic experiments was performed during PDU Run 10 (2) on HRI's process development unit (PDU). The response of the gas/slurry ebullated bed to changes in operating variables such as gas and liquid velocity or liquid viscosity was measured. The experimental plan included tests during the normal duration of the PDU run (relatively constant flow rates but changing product viscosity) and an additional period where the gas and liquid flow rates were varied over a wider range. The ultimate goal of these experiments was to extend the model development performed under a previous contract (1) to apply to the operating PDU reactor.

Samples and Data Collection

The data required for analysis of the fluid dynamics tests involved obtaining a variety of samples and operating conditions.

PDU Operating Conditions.--The primary variables were the liquid flow rates (both slurry mix tank feed rate and internal recycle rate) and the gas flow rates (both fresh hydrogen and recycled hydrogen-rich gas). These variables were provided by HRI (62) and are summarized in Table XIV.

Liquid Samples.--Sixteen reactor liquid product slurry samples were withdrawn from the bottom of the high-pressure separator (Figure 51) during the test program. The sampling procedure used by HRI has been previously documented (2). Table XV itemizes the sampling dates. Battelle Memorial Institute later measured the viscosity and density of these samples. Four additional samples were also withdrawn for Oak Ridge National Laboratory (ORNL) for viscosity and density determination.

Liquid samples from the clean oil tank and slurry mix tank were also obtained on selected test dates. Slurry mix tank liquid characterizations appear in Table XVI.

PDU Reactor Effluent Composition.--Based on flow rates and inspections of test samples taken at various process locations, HRI routinely back-calculates an estimated reactor slurry liquid composition for material leaving the bottom of the O-1 separator. The data, reported as fractions of unreacted coal, ash, resid, and distillate, appear in Table XVII. This information will be correlated and used later for estimating the density and viscosity for tests where no liquid sample was taken.

Catalyst Samples.--One sample was taken on 8/3/80. Another sample was taken by HRI at the completion of the PDU run on 9/24/80. A linear variation of catalyst density between these two dates was assumed. The density increased

only 10% over the entire duration of the run. A summary of catalyst-soaked particle densities used for data analysis appears in Table XVIII.

Gamma-Ray Scan.--A gamma-ray scintillation detector was mounted on the existing PDU reactor traverse scanner. This system, previously developed by HRI, consisted of a sled mounted on vertical rails which ran parallel to the reactor. By adjusting the elevation of the gamma-ray source and detector and noting changes in the count rate, the catalyst bed height may be determined. A second 1" x 1" detector crystal was positioned opposite the center of the beam from the 500 mc Cs-137 source. This installation was designed to retain the existing HRI bed density monitoring equipment and to provide gamma-ray attenuation data in small, well-defined areas of the reactor.

Side and top views of the assembly appear in Figures 52 and 53, respectively. A schematic description of the electronics train appears in Figure 54. The criteria for setting the gain of the amplifier and discriminator to bracket the Cs-137 photopeak are presented in Figure 55. Because the apparent peak energy shifted with detector temperature (Figure 56), retuning the electronics was necessary before each fluid dynamics test.

The first scan performed was a "zero" scan on an empty reactor. The no-flow gamma-ray absorption data and subsequent data under operating conditions could then be used to calculate the average density at each point. As will be detailed in a later section, knowledge of the catalyst bed expansion and liquid density then allows calculation of all component phase volume fractions.

On 7/11/80, the empty reactor (zero) gamma-ray scan was taken. After tuning the detector electronics, absorption measurements were obtained at 2" axial intervals, with some 1" interval data near the bottom of the column. The density monitoring system was able to scan elevations between approximately 3 and 14 ft above the bottom flange of the PDU reactor.

Figure 57 illustrates the variation in count rate with detector elevation. At each location, the count rate displayed is the average of at least two separate measurements taken for a 20-second interval. Standard deviations were on the order of $\pm 1\%$. At elevations of 4 ft and 6 ft, sharp drops in the average zero count rate may be seen. These were caused by external pipes and flanges connected to the reactor. Data taken here will be most sensitive to errors in traverse position during the operating test scans. Therefore, results from these locations may possibly not be suitable for use.

Gamma-ray scans on the operating PDU were taken at 14 test periods during the run. Because the reactor now contained dense, absorbing material, the average gamma-ray counts dropped. The average gamma-ray count rate was taken over 30-second test intervals, again with at least two measurements at each elevation. Three scans were also taken during the shutdown sequence (two on 9/24/80 at 2:12 PM and 7:52 PM, and one on 9/25/80 at 11:08 AM) to measure the absorption of the settled catalyst bed.

In addition, some interference was encountered when the sled was at the top of the column and was exposed to a second gamma-ray source (the level detector of the high-pressure separator) which was being used. This marked increase in count rate with elevation was not observed in any of the PDU tests or shutdown scans. Therefore, a modified zero scan, also shown in Figure 57, was used in the data analysis.

Density and Viscosity of PDU Reactor Liquid Slurry Samples

Sixteen PDU reactor liquid slurry samples were characterized at Battelle Memorial Institute to measure their densities and viscosities at process conditions. Battelle's experimental apparatus appears in Figure 58. Liquid was charged from the sample bomb into the viscometer while maintaining the pressure at 2,000-3,000 psig. A bob (Item 9) was alternately raised and lowered through the liquid-filled chamber. A magnetic coupling (Items 4 and 5) eliminated leaks or mechanical interference from shaft seals. Full details of the testing technique appear in Battelle's final report (Appendix C). During the several months that the experiments were performed, Battelle made some modifications to the viscometer to improve its accuracy and reliability. After these improvements were completed, several samples were rerun. For all these repeated cases, the later data were judged to be more accurate and were therefore used in our analysis.

The slurry samples were generally charged to the viscometer at a low temperature (around 400°K), just sufficient to allow the liquid to flow easily. The viscometer temperature was maintained at a constant value while the bob was moved up and down by the magnetic drive at different speeds. Measurement of the force to move the bob at a given velocity allowed calculation of the shear rate and the shear stress. Because the bob moved vertically, the density of the slurry could be calculated from the buoyancy effects. The temperature was then raised to the next measurement value up to a maximum of 725°K (845°F), slightly above the PDU reactor normal operating temperature.

Analysis of initial data indicated that the rheological behavior of these slurries could be modeled by the Bingham model:

$$\tau = \tau(0) + \dot{\gamma} * \text{Plastic Viscosity} \quad (14)$$

where τ = Shear stress.

$\tau(0)$ = Yield stress.

$\dot{\gamma}$ = Shear rate.

Plastic Viscosity = Rate at which shear stress increases as shear rate is increased.

For a Newtonian fluid, $\tau(0)$ is zero, and the "plastic" viscosity is the normal, shear-independent viscosity.

Figures 59 through 61 illustrate the variance of apparent plastic viscosity, yield stress, and density versus temperature. Attempts to replicate these data at lower temperatures, however, suggested that irreversible changes were occurring in the samples above 700°K. Figure 62 illustrates this phenomenon for Amoco Sample No. 8. After tests at 725°K, the measured viscosity at 700°K

is markedly lower than that obtained during initial heatup. Battelle performed an experiment to estimate the magnitude of this change over time at high temperature. Amoco Sample No. 15 was charged to the viscometer and rapidly heated to 725°K. During the next two hours, the changes in plastic viscosity, yield stress, and density were measured. Figures 63 through 65 present the results. Included for comparison are the values obtained earlier for the sample when the gradual heatup included several tests at lower temperatures.

We speculate that these changes may result from a reaction, preasphaltenes → asphaltenes, which occurs at elevated temperatures. Figure 66 presents data reported in References 55 and 56 contrasting the viscosity of asphaltenes and preasphaltenes. The dashed portions of the lines are extrapolated. Clearly, small decreases in preasphaltene content can translate into significant viscosity decreases. Although the plotted data were developed for Synthoil process liquids, similar trends for viscosity could be expected for the components of H-Coal slurries as well.

The viscosity and yield stress, strongly influenced by intermolecular attractive forces, will be more sensitive to chemical composition than the density. Because Amoco Sample No. 15 had the highest resid content of all the samples tested (31.3%), it is probably the most reactive. Hence, the changes in viscosity observed for this sample in the Battelle viscometer experiments represent an upper limit.

The Oak Ridge National Laboratory viscometer used a different design, as shown in Figure 67. Slurry liquids were again removed from the sample container at low temperature. The liquid was then passed through the preheater coil so that the material reached the test temperature (719°K = 834°F) just as it left the coil array. The liquid then passed through a capillary tube viscometer, where the pressure drop for flow was measured over a known length of tubing. After passing through the viscometer, the liquid sample was set aside and not reused. A previously calibrated gamma-ray apparatus determined the liquid density at the process conditions. Measurement of pressure drop at several flow rates allowed calculation of parameters for a rheological model. ORNL's analysis indicated that either a power law or Bingham plastic viscosity model could adequately describe the data.

Based on the preheater dimensions and ORNL's reported flow rates, a plot illustrating the maximum time the sample spends at different temperatures may be developed. Such a plot appears in Figure 68. By contrast, a time-temperature history for the Battelle test sequence also appears here. Clearly, the ORNL design, with its shorter residence times, is less likely to degrade samples during measurement. ORNL's final report appears in Appendix D.

These reaction effects dictated a new strategy for determining the physical properties at PDU reactor conditions. Figure 69 illustrates Battelle data obtained at lower temperatures for Samples 3 and 4, when experiments were performed to test the effect of changing the hydrogen partial pressure. Within the ± 10% error limits ascribed to the measurement technique by Battelle, no degradation in viscosity is apparent up to 675°K. Therefore,

the following method was selected to estimate the physical properties at process conditions:

Use only data taken at or below $T = 650^{\circ}\text{K}$. Extrapolate to 719°K using the following models:

Viscosity: Arrhenius model (activated exponential).

Density and Yield Stress: Linear variation with temperature.

For the tests where a sample either was not taken or was lost in processing, correlation techniques were used to estimate the properties. The correlation parameter chosen was the quantity

$$100\% - \% \text{ Ash} - \% \text{ Coal} - \% \text{ Resid}$$

which corresponds with the extent of conversion to light, low-viscosity products. These correlations are shown in Figures 70 through 72.

Table XIX presents the final values of density and viscosity assigned to the samples. Table XIX also compares the Battelle and ORNL sample results. With the exception of Amoco Sample No. 11 versus ORNL No. 4 (which were taken at low liquid and high gas flow rate), agreement between the two sets of values is reasonable. Note also that the viscosity of the samples as determined by Battelle ranges from 0.9 to 3.4 cp. By comparison, the viscosity of kerosene at 70°F is 1.4 cp, while the viscosity of kerosene/coal char slurries ranges from 3.8 to 4.9 cp.

Analysis and modeling of the PDU-10 data will require use of some fluid dynamics correlations, which will be discussed in a later section. Appendix E documents the method used to combine the plastic viscosity and yield stress into a pseudo-viscosity. The majority of force suspending a catalyst particle will be viscous shear forces rather than yield stresses. This will simplify later analyses by allowing the slurry to be modeled as a Newtonian fluid. This in turn will allow correlations developed for Newtonian fluids such as water to be directly applied to the PDU experimental data.

Data Transformations

The following methods were used to calculate linear gas and slurry velocities from the metered values.

Gas Velocity.--HRI routinely calculates superficial gas velocities at the PDU reactor inlet and outlet for its daily material balance reports. These calculations assume ideal gas behavior. The average of these two velocities (which typically were within 10% of each other) was used for data analysis.

Slurry Velocity.--The superficial liquid velocity is computed by summing its two components, slurry feed and slurry recycle:

$$U_1 = \frac{\dot{V}_{\text{Slurry Feed}} + \dot{V}_{\text{Internal Recycle}}}{A} \quad (15)$$

where $\dot{V}$ = Volumetric flow rate, ft³/sec.
 U_1 = Slurry superficial velocity, ft/sec.
 A = PDU cross-sectional area, $\pi/4 (8.5")^2$

At reactor conditions, the fresh slurry feed is assumed to have the same density as the recycle slurry. This assumption was based on comparison of room temperature densities of the slurry mix tank (Table XVII) with ORNL measurements of recycle slurry densities at room temperature (Table 5 of Appendix D). Densities for both materials were comparable.

Then:

$$\dot{V}_{\text{Slurry Feed}} = \frac{\text{Slurry Feed Rate, Lb/Hr}}{\rho \times 3,600} \quad (16)$$

The internal recycle rate was monitored by a Venturi meter, previously calibrated with water and then adjusted to an assumed slurry density of 0.85. After slurry densities became available from Battelle and ORNL, the flow rate was adjusted.

$$\dot{V}_{\text{Internal Recycle}} = (\text{Meter gpm/ft}^2) \sqrt{0.85/\rho_{\text{Actual}}} \cdot A \cdot 2.228 \cdot 10^{-3} \frac{\text{Ft}^3/\text{Sec}}{\text{GPM}} \quad (17)$$

Catalyst Bed Expansions

Catalyst bed expansions were determined by gamma-ray absorption measurements. An increase in the ratio (I/I_0), the number of gamma-ray counts under test conditions divided by the comparable count rate at that elevation for an empty reactor, corresponded to the interface between the dense catalyst bed and the less dense dilute phase. In some cases, this interface was quite sharp (Figure 73); for some other tests, the catalyst density slowly decreased with higher elevation, producing a diffuse dense-/dilute-phase interface, as shown in Figure 74. This phenomenon was also observed during some cold-flow tests using Amocat-1A catalyst, as was discussed in Section III.

Per cent catalyst bed expansions were calculated using the formula

$$\% \text{ Bed Expansion} = 100 * \frac{(\text{Expanded Bed Height} - \text{Initial Bed Height})}{(\text{Initial Bed Height} - 8")} \quad (18)$$

Figure 75 details how elevation values were defined for the PDU reactor. Zero was chosen to be the flange line at the bottom of the reactor vessel. The 8" offset accounts for the height taken up by the plenum chamber and distributor plate (2).

On startup, the PDU reactor was charged with presulfided Amocat-1A catalyst to an initial settled bed height of 96". At the end of the standard PDU test period (Period 41-A), approximately 17 lb of slurry-soaked catalyst was withdrawn from the system. This reduction of inventory was selected

to allow safe operation over a wider range of process variables. The settled initial bed height for all the fluid dynamics tests taken during Periods 41-B and later was 78", as determined by the gamma-ray scans performed during the shutdown sequence.

A listing of catalyst bed expansions for the 14 tests is in Table XX. Also included are the liquid and gas flow rates and the estimated viscosity and density of the reactor slurry liquid.

By choosing selected tests, consistency checks on the data are possible. Table XXI compares four tests where the nominal slurry viscosity was roughly constant at 2.6 cp. In going from Test 4 to Test 6 to Test 14 at constant gas velocity, the expected trend of increasing bed expansion with increasing liquid velocity is apparent. Comparing Test 14 with Test 13, where the liquid velocity remained constant and the gas velocity was decreased, the bed expansion decreased from 96% to 73%. Both these responses have been observed in cold-flow studies, as discussed in Section III.

Comparison of the magnitude of PDU catalyst bed expansions with those observed in cold-flow studies, however, reveals that the expansions in the PDU are significantly higher than other pilot plant tests with fluids of the same nominal viscosity. Run 224 was performed in the Amoco fluid dynamics cold-flow pilot plant to study the fluid dynamics of the system using Amocat-1A catalyst and a 25 wt% slurry of coal char fines in kerosene. The estimated slurry viscosity is 4.5 cp. Table XXII compares the observed bed expansions during PDU-10 with those for cold-flow Run 224. In all cases, the PDU bed expansions are equal to or (usually) are greater than the cold-flow test bed expansions, even though the maximum measured PDU slurry viscosity was roughly 3.5 cp. Discussions of reasons for this discrepancy will be presented shortly.

Phase Volume Fractions

A primary goal of the experimental program was to measure the volume fractions (holdups) occupied by the gas, liquid slurry, and catalyst phases for model development. The gamma-ray elevator described earlier was the chief instrument to obtain these data.

When a collimated beam of gamma-rays passes through matter, it is attenuated at an exponential rate (64):

$$I/I_0 = e^{-\rho\mu L} \quad (19)$$

where I = Attenuated intensity.
 I_0 = Original intensity.
 ρ = Material density.
 μ = Material mass absorption coefficient.
 L = Material thickness.

For gamma radiation from a Cs-137 source, the mass absorption coefficients for most elements are constant. However, hydrogen, because of its higher number of electrons per unit mass, has a mass absorption coefficient roughly twice as large as other elements (64).

Mass Absorption Coefficients for the PDU Fluid Dynamics Tests.--Mass absorption coefficients for the PDU catalyst and liquids were determined by combining the empty reactor zero gamma-ray scan data with the data taken during the shutdown (9/24/80 at 7:52 PM). Because temperatures were close to ambient, the following formulas could be used:

In the Catalyst Bed:

$$\frac{-\ln(I/I_0)}{L} = \rho_L \epsilon_L \mu_L + \rho_C \epsilon_C \mu_C \quad (20)$$

Above the Catalyst Bed:

$$\frac{-\ln I/I_0}{L} = \rho_L \mu_L \quad (21)$$

HRI provided the temperature history for this shutdown period and the density versus temperature relation for the shutdown oil. Figure 76 illustrates this relationship, with annotation of the three shutdown test scan times. Table XXIII details the I/I_0 versus elevation profile of the 9/24 PM test. Tables XXIV and XXV present the calculation of the liquid and catalyst mass absorption coefficients. Because ORNL studies on various pure liquids and coal-derived slurries found no significant variations of mass absorption coefficient with composition (65), these coefficients were judged applicable to all the PDU tests.

Volume Fractions.--With the mass absorption coefficients determined in the previous section and densities available (from Tables XVIII and XIX), the following relationships were used to compute the volume fractions occupied by gas, catalyst, and slurry liquid:

$$\ln(I_t/I_0) = -L(\rho_{sl} \mu_{sl} \epsilon_{sl} + \rho_g \mu_g \epsilon_g) - \rho_{st} \mu_{st} L_{st} \left[\frac{1}{(1 + \alpha \Delta T)^2} - 1 \right] \quad (22)$$

The second term on the right-hand side of the equation (very small compared with the first) arises from the fact that the zero scan is performed at ambient temperature, while the operating scans were performed at elevated temperatures.

The gas density was calculated from the ideal gas law to be 0.42 lb/ft³. The gas mass absorption coefficient was taken to be twice the liquid value.

The catalyst volume fraction ϵ_C is determined from the catalyst bed expansion:

$$\epsilon_C = \epsilon_{Co} \left(\frac{100}{100 + \% \text{ Bed Expansion}} \right) \quad (23)$$

where ϵ_{CO} (the volume fraction at settled conditions) is determined by the ratio of dry settled density to dry particle density:

$$\epsilon_{CO} = \rho_B / \rho_P \quad (24)$$

The gas holdup is computed by difference:

$$\epsilon_g = 1 - \epsilon_C - \epsilon_{sl} \quad (25)$$

Simultaneous solution of Equations 22, 23, and 24 provides values for ϵ_C , ϵ_{sl} , and ϵ_g .

Assorted experimental values used in calculating these holdups appear in Table XXVI. Calculated holdups for the dense phase (gas/slurry/catalyst) region of the reactor are tabulated in Table XXVII. Also included is the Darton-Harrison gas drift flux, defined by

$$V_{CD} = \epsilon_g (1 - \epsilon_g) (U_g / \epsilon_g - U_s / \epsilon_{sl}) \quad (26)$$

Corresponding gas/slurry dilute-phase holdups appear in Table XXVIII.

Discussion

Examination of the holdups in Table XXVII indicates several unexpected results. For Tests 1 and 2, the gamma-ray attenuation was such that the projected volume fraction of the liquid was excessively high. This caused the calculated holdup of the gas to be less than zero, clearly not a physically realistic situation.

The drift flux, V_{CD} , is a measure of how rapidly the gas bubbles are rising relative to the average liquid flow rate. A negative drift flux suggests that the bubbles are moving down rather than up. The apparent negative value for the drift flux in Test 5 is as physically unrealistic as the sub-zero gas volume fractions in Tests 1 and 2.

These errors could have been caused by

- A) Incorrect assignment of the liquid density: If the liquid were more dense, less volume would be required to provide the proper gamma-ray attenuation. For Test 1, however, two duplicate samples were taken from the separator bottoms. Battelle's independent density measurements agreed within 1% of each other.
- B) Incorrect catalyst density: At the beginning of the run, the catalyst was presulfided but was assumed to be free of contaminant ash deposits.
- C) Incorrect liquid mass absorption coefficient: For startup, atmospheric still bottoms were used as slurry liquid until sufficient recycle solvent

had been generated in the run. These atmospheric still bottoms had a high hydrogen content and a higher hydrogen donor ability than later solvent (Tables 8 and 29, Reference 2). However, this higher hydrogen content should not have caused a significant (greater than 10%) increase in the mass absorption coefficient.

- D) Reactor liquid homogeneity: If the PDU liquid flow regime were not a perfectly well-mixed CSTR, gradients in coal conversion and physical properties could exist. In this case, the sample from the separator may not be representative of the entire system. Although these property gradients may have occurred in all the tests, the runs with the most reactive conditions (freshest catalyst plus highest solvent hydrogen donor activity) would be expected to exhibit the largest inhomogeneities.

Comparison with Cold-Flow Pilot Plant Tests

Table XXIX compares the physical properties for the PDU-10 tests with corresponding values for four cold-flow pilot plant tests using coal char/kerosene slurries. Amoco Run 224 was performed using Amocat-1A catalyst to measure its fluid dynamic performance with a well-defined slurry having approximately the same fines content as the PDU slurries. Although the HRI slurries are slightly denser, the viscosity (which has a much stronger influence on such variables as catalyst bed expansion) is lower.

Table XXX contrasts the observed gas holdups in the PDU-10 tests with those at corresponding superficial velocities in the Amoco cold-flow tests. Most of the gas holdups in the PDU are significantly higher than corresponding cold-flow values.

Analysis of earlier H-Coal cold-flow pilot plant data developed under a prior contract (1) indicated two effects of liquid slurry viscosity:

- A) As slurry viscosity was increased, catalyst bed expansions increased.
- B) As slurry viscosity was increased, gas holdups decreased. This was attributed to the effect of increased viscosity in accelerating the coalescence of gas into large, rapidly rising bubbles.

The difference in viscosity of preasphaltene and asphaltene species (Figure 66) may be influencing the PDU fluid dynamic behavior. The HRI PDU slurry recycle system is designed to exclude gas and return only slurry from the reactor top. This material's composition is nominally identical with the high-pressure separator bottoms stream, which was sampled and later characterized. The fresh feed from the slurry mix tank accounted for 10-25% of the total reactor liquid flow. The viscosity of the fresh feed leaving the unit preheater, containing either suspended or partially dissolved coal, may be significantly higher than the recycle stream, which has undergone chemical reaction. If the PDU behaved as a perfectly back-mixed reactor, the separator bottoms would be representative of the material in the reactor. However, if any deviations from CSTR performance exist, then the introduction of the high-viscosity preheater outlet liquid could cause a significant gradient of viscosity and composition across the reactor.

The high incoming viscosity would cause a high bed expansion. A continuous generation of hydrogen and other light gases would ensure a large holdup of small bubbles. If this did occur, then both high bed expansions and high gas holdups could occur simultaneously. Examination of the I/I_0 ratio for several tests in Appendix F (3,6,7,8, for example) shows a far wider variation of this ratio with elevation than that observed for the shutdown tests (15 and 16) or the zero scan.

Figure 77 is a conventional plot of the drift flux versus gas holdup in the dense bed (gas/slurry/catalyst) portion of the reactor. The three tests (1, 2, and 5) which exhibited apparently negative gas holdups or drift fluxes are not included on the graph. Also shown for comparison is the line defining ideal bubbly operation developed in the pilot plant cold-flow tests with kerosene (Run 218).

Comparison of the PDU data with the composite cold-flow results illustrates that the bulk of the operating reactor gas flow is in the ideal bubbly regime. Because the slope of the V_{CD} versus ϵ_g line is related to the bubble rise velocity, the location of most PDU points below the composite line suggests that the gas bubbles in these tests are rising quite slowly. Tests 8 and 10 clearly lie in a churn turbulent area of operation. These two tests were performed at high gas velocity (0.09 ft/sec) and low or medium liquid velocities (0.050-0.075 ft/sec). The transition from ideal bubbly to churn turbulent flow had previously been observed in cold-flow tests under conditions of high gas velocity and low liquid flow rate. Test 12, performed at both high gas and high liquid superficial velocities, lies close to the ideal bubbly line, although it is clearly separated from the other tests. The effect of increasing liquid velocity in delaying the change from ideal bubbly to churn turbulent operation was noted previously (1).

The liquid slurry in Tests 8 and 12 also exhibited the highest viscosities (3.80 and 3.56 cp, respectively, as measured by Battelle). Previous data (1) established the effect of increased slurry viscosity in accelerating coalescence of gas into large bubbles with rapid rise velocities; the corresponding gas holdup decreased. Under these conditions, the calculated drift flux assumes a large value, placing operation in the churn-turbulent regime. However, Test 10, with a (Battelle-measured) viscosity of 2.11 cp, lay further in the churn turbulent region than either Test 8 or 12. It is probable that the high gas velocity influenced the fluid dynamics more than did the slurry viscosity.

VIII) MATHEMATICAL MODEL

A variety of models have been proposed in the literature to describe the fluid dynamics of a three-phase fluidized bed. A review of these models was presented in Reference 1.

No completely theoretical models were found during the literature search. Model approaches developed by other authors have tended to be in one of two categories:

- A) Empirical correlation: Phase holdups are correlated against operating variables.
- B) Semi-empirical models: Phase holdups are calculated from the results of model calculations. Specific assumptions are postulated about the behavior of bubbles, wakes, and solid phases. However, each model "law" incorporates a parameter which may be described by a correlation approach rather than an a priori calculation.

Review of the Bhatia-Epstein Model

The Bhatia-Epstein model was selected during the initial contract (1) for several perceived advantages over other models:

- A) It is able to predict both catalyst bed expansions and bed contractions when gas is introduced into a liquid-fluidized bed.
- B) It is able to describe operation both in the ideal bubbly and churn turbulent regimes.
- C) Its parameters (bubble rise velocity, wake structure, and wake composition) are amenable to a physical interpretation and may be checked by such experimental approaches as radiotracer techniques.

Figure 78 illustrates the basic features of the Bhatia-Epstein model. The system is divided into three component phases: gas bubbles, wakes behind the bubbles, and particulate (catalyst plus liquid slurry) phases. The equations of the model are presented in Table XXXI. Equation 1a is not strictly an independent equation but may be derived from the other nine. A short discussion after each equation indicates which are definitions and which incorporate specific models or assumptions.

A key independent variable of the model is the bubble rise velocity, U_{tb} . This variable, relating how rapidly bubbles rise relative to the liquid, will control the gas holdup. Additionally, because the bubbles are assumed to drag their wakes behind them at the same velocity, a mechanism is incorporated which allows liquid to be transported through the system at a higher velocity.

A second model parameter is related to the wake structure. This is the wake volume ratio, K_0 , the ratio of the wake volume to the gas bubble volume for a single gas bubble at an infinitesimal gas volume fraction. Experimental studies of the relationship between wake and bubble volumes have been performed in liquids. Henriksen and Ostergaard (66) noted that the wake volume behind a single bubble was that which completed the sphere whose upper surface was defined by the bubble top. In turn, the bubble shape was strongly influenced by the viscosity of the liquid. These observations are incorporated in a semi-empirical fashion into the model by defining upper and lower limits to the parameter.

The wake behind the bubbles is assumed to entrain both slurry and catalyst. The amount of catalyst carried by the wake is described by the parameter

X_k . If X_k is 0, the wake is assumed to be composed solely of liquid. For $X_k = 1$, the ratio of solid content to liquid content in the wake is the same as that existing in the particulate phase, where slurry fluidization is expanding the catalyst bed.

The parameter X_k was discussed by El-Temtamy and Epstein (67) in a paper which appeared after the basic model was published. We find from our cold-flow studies that the Bhatia-Epstein model can describe the experimental results by using the published correlation for X_k . In the PDU-10 modeling, X_k was taken to be an independent variable along with U_{tB} and K_0 .

Model Parameters

Because it is assumed in the Bhatia-Epstein model that the slurry-fluidized portion of the bed may be described by the Richardson-Zaki correlation, values of the catalyst terminal velocity (U_t) and the index (n) must be available. In the cold-flow pilot plant, these two parameters are obtained by performing a series of two-phase slurry fluidization experiments, where changes in the bed expansion are measured as a function of superficial slurry velocity. The slurry composition remains constant for the tests, and there is no gas flow.

During the PDU experimental program, however, it was not possible to determine these parameters. The density and viscosity of the liquid slurry changed as reactor operating conditions were altered during the test period. Additionally, failure to maintain continuous gas flow through the inlet lines would have resulted in coking and blockage.

A parallel analysis of liquid/solid fluidization data in the literature was performed under this contract. A major conclusion of the study (full report attached in Appendix G) is that the relation between the drag coefficient and Reynolds number for an infinitely long cylinder can be applied to the finite catalyst extrudates used in the PDU reactor by proper choice of the diameter, d . Use of these correlations then allows the two Richardson-Zaki parameters (U_t and n) to be estimated solely from physical properties.

The slurry density (ρ_l) and viscosity (μ), along with the catalyst density (ρ_s) and diameter (d) are used to calculate the Galileo number Ga :

$$Ga = \frac{d^3 \cdot \rho_l \cdot g \cdot (\rho_s - \rho_l)}{\mu^2} \quad (27)$$

Although the slurry is a Bingham plastic (58), the analysis in Appendix E indicated that the bulk of the forces are related to the fluid's plastic viscosity. Hence, this treatment (which is strictly proper only for a Newtonian fluid) is valid here. With the Galileo number established, the terminal Reynolds number for a single particle is calculated. The literature correlation for this is shown in Figures 2 and 8 of Appendix G.

The Reynolds number, Re , is defined by

$$Re = \frac{d * u_t * \rho_l}{\mu} \quad (28)$$

The particle terminal velocity, u_t , is the only unknown.

Richardson and Zaki presented a correlation for the index n of spheres in their original paper. Figures 9 and 10 of Appendix G show that this correlation is applicable to cylinders as well.

These calculations were performed for all 14 PDU-10 tests. Table XXXII tabulates the individual fluid properties (density and viscosity) and the predicted terminal Reynolds numbers, terminal velocities (U_t), and Richardson-Zaki index (n) for each test in the PDU experimental program.

Figure 79 illustrates the range of Galileo numbers over which the Reynolds number was predicted using the literature correlation. Some experimentally measured cold-flow pilot plant data points are included for comparison. Since both the Galileo number and the terminal Reynolds number can be measured for these cold-flow tests, the individual data points may not fall precisely on the line summarizing prior literature data. Figure 80 is the corresponding prediction for the index n in the PDU-10 tests. Again, experimentally determined values of n for four cold-flow slurry fluidization tests are also presented. With the exception of Test 221, the agreement between the slurry data points and the Richardson-Zaki correlation for n is quite good.

Because the Bhatia-Epstein model incorporates the predicted parameters U_t and n in Equation 4 of Table XXXI, the sensitivity of the model predictions to changes in the fluid viscosity was also explored. As was speculated in Section VII, the average viscosity in the catalyst bed could have been higher than that assigned to the separator bottoms liquid. Therefore, each viscosity was multiplied by 4. This magnitude of increase was selected to raise the PDU liquid viscosities to the maximum level tested during the prior contract (1). Test Series 420 and 427, documented in Reference 1, employed mineral oil at a temperature of 100°F to fluidize a catalyst bed. The liquid viscosity was 14.6 cp. Comparison of these bed expansions with those for a 4 cp fluid indicated that the viscosity increased bed expansion by a factor of about 2.5. This is roughly the maximum discrepancy between cold-flow and PDU tests noted in Table XXII.

Increasing the viscosity affects the Galileo number (Equation 27). Hence, the terminal Reynolds number and the Richardson-Zaki index n will need to be recalculated for the high-viscosity case. Table XXXIII summarizes the new calculations, which assume each test's viscosity to be the Battelle value multiplied by 4. Figures 81 and 82 compare the newly calculated Galileo numbers for the PDU tests and include the cold-flow tests as a point of reference. As expected, increasing the viscosity does decrease the terminal velocity.

Cold-Flow Tests

The Bhatia-Epstein model was applied to analysis of the data from both the cold-flow (kerosene/coal fines slurry) tests and the HRI studies. Tables XXXV

to XXXVIII contain the observed volume fractions and X_k wake concentration ratio (predicted by correlation in Reference 67) and list the two parameters U_{tb} (bubble terminal velocity) and K_0 (the single bubble wake volume ratio) for the cold-flow, three-phase fluidization tests.

The quantity Δ is a measure of how well the model is able to fit the data and is defined by the relation

$$\Delta = \text{Sum}[\epsilon(\text{Predicted}) - \epsilon(\text{Observed})] \quad (29)$$

A zero value of Δ indicates that when given the listed parameters, the model was able to predict the observed volume fractions with no error.

Bubble diameters were calculated from the bubble rise velocities using the Peebles and Garber correlation (68). Table XXXIV presents the four equations for the effective radius $r(e)$ which apply for different Reynolds number ranges. The data were developed for single isolated bubbles in a liquid medium. The correlation predicts a maximum bubble velocity at the boundary between Regions 2 and 3 (where further increases in the bubble diameter cause the bubble to rise more slowly).

The rise velocity of single bubbles is controlled by a balance of the viscous drag or surface tension force and buoyancy effects. Application to the three fluidized-bed experiments introduces two additional unknowns:

- A) The Peebles and Garber experiments did not address the effect of a finite gas holdup upon the average bubble rise velocity. Bhatia (69) correlated a variety of experimental work and suggested that swarms of bubbles rose less rapidly than single bubbles. Other authors (50) have postulated the opposite effect, with non-zero gas holdups increasing the gas/liquid relative velocity.
- B) In their correlations, Peebles and Garber use fluid properties such as the density, viscosity, and surface tension. In the three-phase system, choice of values to assign these variables is somewhat arbitrary. A macroscopic viscosity may be assigned to the fluidized catalyst bed based on its resistance to stirring. However, the drag that individual particles exert on a gas bubble may be distinctly different than that of a liquid which is homogeneous on a molecular scale.

These effects are visible upon examination of the predicted bubble diameters in Tables XXXV to XXXVIII. For cases where bubble rise velocities above 0.8 ft/sec were predicted, no corresponding diameter could be calculated by the Peebles and Garber correlation. Previous radiotracer experiments with kerosene/coal char fines (1), however, indicated that some gas bubbles were rising through the bed with velocities of approximately 1 ft/sec.

Figures 83 through 86 correlate the terminal velocity versus $(U_g - U_1)$ for the four cold-flow test series. This choice of the form for the independent correlating variable was discussed in Reference 1. Briefly, previous experiments had noted that as gas velocity was increased in catalyst beds fluidized

either by kerosene/coal char slurries or by high-viscosity fluids such as mineral oil, the nature of the gas flow changed from ideal bubbly to churn turbulent. Increasing the liquid velocity tended to stabilize the flow against this transition. Because churn turbulent operation is characterized by rapid bubble coalescence and high bubble rise velocities, the Bhatia-Epstein model bubble rise velocity U_{tb} could be expected to increase in this region. Prior data (1), as well as the currently developed data shown here, support this view.

In these four figures, the correlation line developed for cold-flow tests with a 17.8% coal char fines/kerosene slurry from Reference 1 are presented for comparison. Bubble rise velocities from kerosene-only fluidization tests are also shown. For all four tests (221-224), the behavior of the bubble rise velocity versus $(U_g - U_l)$ appears to track that previously determined for the 17.8% coal char slurry. As slurry content increases, average bubble rise velocities also increase. This is consistent with the effect of the increased viscosity in promoting coalescence of gas bubbles. The magnitude of the scatter in the model fit becomes evident with the larger number of tests to which the model was applied.

The wake structure effects are described by the wake volume ratio, K_o . If the wake volume is large and the bubbles are rapidly rising, the wake may entrain significant amounts of catalyst and liquid. This in turn may cause the catalyst bed to contract with further increases in gas velocity (4). Alternatively, when the wake volumes are small and the bubble rise velocities are low, wake effects will not be as important in determining the component holdups.

Figures 87 to 90 present the model's single bubble wake volume ratio data plotted against liquid superficial velocity for the four cold-flow tests. The choice of liquid velocity for this relationship was developed in Reference 1. Following the approach of Henriksen and Ostergaard (66) and El-Temtamy and Epstein (67), the wake volume ratio behind the gas bubbles is expected to be controlled by the "viscosity" of the fluidized bed. As the catalyst bed expands, its viscosity decreases (66). The primary variable controlling bed expansion is the liquid velocity (U_l), with gas velocity playing a secondary role. For this reason, the wake volume ratio is correlated against liquid velocity.

The wake volume ratio correlation line developed for cold-flow tests with a 17.8% coal char fines/kerosene (1) slurry are presented in Figures 87 through 90 for comparison. Some wake volume ratios from the four cold-flow tests are lower than those developed in Reference 1. However, the trend of increasing wake volume with increasing liquid velocity is observed.

Application to HRI PDU-10 Tests

This model was then applied to the PDU tests. Of the 14 three-phase tests performed, three were not suitable for analysis by the Bhatia-Epstein model:

Tests 1 and 2, where the gas volume fractions were less than zero.

Test 5, where the drift flux was less than zero. The structure of the model requires bubbles to rise faster than the surrounding liquid, and a negative drift flux corresponds to the case of the bubbles rising more slowly.

The Richardson-Zaki parameters U_t and n are used in the Bhatia-Epstein model to describe the effect of liquid velocity on catalyst bed expansion. These therefore will have a major effect on predictions of total catalyst and liquid volume fraction, while the bubble rise velocity will primarily control the gas-phase holdup. Hence, if the model is able to predict gas volume fractions properly but cannot match the observed catalyst and liquid fractions, one solution may lie in modifying U_t and n .

The model was initially tested using the first set of Richardson-Zaki parameters in Table XXXII. These values were derived by assuming that the characterizations of separator liquid samples developed by Battelle could model the liquid present in the reactor's catalyst bed. Table XXXIX (with significant non-zero values for Δ in Tests 9, 10, and 11) illustrates that the model was unable to match many of the observed catalyst and liquid volume fractions. These failures, coupled with the catalyst bed expansions which were higher than expected, led us to examine the effect of increasing the viscosity in the reactor.

To simulate the added viscosity of the incoming slurry from the preheater, the product viscosities were multiplied by 4. New values of the Richardson-Zaki parameters corresponding to the higher viscosity are presented in Table XXXIII. Incorporating this assumption proved much more successful in matching the bulk of the observed data. Table XXXX compares the model predictions with observed volume fractions. The model is now able to match successfully all the experimental points.

As was consistent with the drift flux plot (Figure 77), most of the PDU runs may be characterized by a low bubble rise velocity, low wake effects, and a high gas holdup. Only two tests (Tests 8 and 10) exhibit high bubble rise velocities and large values of X_k , indicating significant wake effects. A direct comparison of the single bubble wake volume ratios, K_0 , does not show significant differences; however, the exponential weighting with gas holdup (Equation 8 of Table XXXI) occurs over a much wider range than was previously noted in cold-flow tests. Table XXXXI illustrates the effect. Tests 8 and 10 have a significant ratio of wake volume to bubble volume. These two tests were previously identified as being in the churn turbulent regime by the drift flux analysis. Test 13 has a high ratio of wake volume to bubble volume; however, because the bubble rise velocity for this test is low, wake effects will not be significant.

For tests such as Tests 9, 11, and 13, the predicted bubble rise velocities are of the same order of magnitude as $2U_g$. At these low bubble velocities, the applicability of Equation 9 of Table XXXI may be questionable. Other variants of model for relative gas/liquid velocity were briefly tested; however, they all predicted relatively low bubble rise velocities for these high-gas-holdup tests.

Comparison of PDU and Cold-Flow Pilot Plant Model Results

A primary aim of this investigation was to assess the suitability of the Bhatia-Epstein model for description of the fluid dynamics in the PDU reactor and to relate the model parameters for the operating reactor to those developed for the cold-flow pilot plant. The bubble rise velocities calculated using the high (4x) viscosity Richardson-Zaki numbers from the PDU tests are contrasted in Figure 91 with those developed in the cold-flow unit Run 224. Figure 92 enlarges the region where the bulk of the PDU and pilot plant data lie.

Cold-flow Run 224 was selected to be most representative of the PDU-10 test, since it used Amocat-1A catalyst and had 25 wt% fines suspended in the liquid, a concentration comparable to the PDU reactor slurries. Two data points from the PDU run (Test 8, in the churn turbulent regime, and Test 13, where the gas flow was judged to be ideal bubbly) do lie within the data range for the cold flow results. Test 10 lies far above the cold-flow data range. The slurry from Test 8 did have the highest viscosity observed (3.8 cp from Battelle's measurements), but several other tests had viscosities higher than the 2.74 cp of Test 13. Test 10, with the highest bubble rise velocity, had an intermediate viscosity value of 2.11 cp. The bulk of the PDU tests exhibit lower bubble rise velocities. This is a direct consequence of the higher gas holdups and lower drift fluxes of the PDU tests as compared with the cold-flow (Run 224) tests.

The failure of the bubble rise velocities to correlate with changes in viscosity again suggests that chemical reaction or other factors unique to the PDU reactor are exerting a strong influence on the gas bubble behavior. For example, reactor pressure alone can affect bubble coalescence phenomena and therefore bubble size and gas holdup. Recent data obtained for the EDS coal liquefaction process (70) showed that gas holdup in the actual reactor operated at higher pressure is significantly higher than the gas holdup in the lower-pressure cold-flow unit. The gas holdup observed in their cold-flow pilot plant was approximately 60% higher at 75 psig versus 20 psig. Although these results (70) were obtained for a gas-slurry reactor with no catalyst present, the trends observed are in agreement with our findings of higher gas holdup in the PDU (high pressure) reactor as compared with the atmospheric pressure cold-flow unit.

Values of K_0 (the single bubble wake volume ratio) from the PDU tests are contrasted with cold-flow Run 224 tests in Figure 93. A direct comparison of these two items, however, must be tempered by including the effect of the gas holdup. The ratio of wake volume to bubble volume will be decreased by the exponential weighting in Equation 8 of Table XXXI. Because the PDU runs generally exhibited higher gas volume fractions than those of the cold-flow tests, the wake volumes will be decreased more.

Wake volume ratios for Run 224 calculated by Equation 8 of Table XXXI are listed in Table XXXXII and may be compared with the PDU data in Table XXXXI. With the exception of the three PDU-10 experiments (8, 10, and 13), the wake volume to bubble volume ratios for Run 224 appear comparable to those for the PDU tests.

IX) MODEL APPLICATION

Use of the Bhatia Epstein model for prediction of volume fractions from operating conditions was discussed previously (1). The three parameters (U_{tb} , K_0 , and X_k) are provided to the model as input, along with gas and liquid velocities. Other necessary data, such as the Richardson Zaki parameters (U_t and n) may be predicted using the technique in Section VIII.

The application of this model to an operating liquefaction reactor is complicated by the variation of slurry density and viscosity; over the 46 operating periods of PDU-10, significant changes in these two slurry properties were observed. Inducing these changes and observing their effect was an objective of the PDU-10 run. In a full scale commercial operation, the ranges of fluid properties would not be as broad after the initial startup.

For modelling purposes, Amoco Test 6, coming at the end of the PDU-10 normal run may be taken as most representative. The values of bubble rise velocity, wake volume ratio, and wake concentration ratio from this test are recommended for use. Implicit in these values, however, are the effects of the Amocat I-A catalyst properties, coal and ash concentration, and the reactivity and yield structure of the Wyodak coal tested in PDU-10. Application of this model to other coals or catalysts should proceed only after suitable tests, such as physical inspection and micro-autoclave experiments provide the basis for estimation of slurry product properties.

X) CONCLUSIONS

The overall goal of our research efforts is to develop a fundamental understanding of the fluid dynamics occurring in the three-phase, ebullated-bed H-Coal system, as well as obtain data useful in the design, operation, and control of the system. Several important areas have been investigated under this DOE contract, and the results are summarized here.

- A) The fluid dynamics occurring in HRI's H-Coal process development unit (PDU) during Run PDU-10 were measured and compared with Amoco Oil cold-flow fluidization results. It was found that catalyst bed expansions and gas holdups are higher in the PDU than those observed in the cold-flow tests for slurries having the same nominal viscosity.
- B) Analysis of the PDU results shows that the differences in A) can be explained by assuming that the viscosity in the reactor is effectively four times greater than the viscosity of the slurry sample obtained from the pressure letdown vessel. It may be that viscosity gradients rather than a uniform viscosity exist in the reactor. It is known from results published in the literature that a small amount of asphaltenes added to an oil has a dramatic effect on the viscosity of the oil.

- C) Comparison of PDU results with cold-flow results shows that the bulk of the operating reactor gas flow lies in the ideal bubbly regime. It also appears that the gas bubbles in these PDU tests are rising quite slowly. Only two of the operating points in our test program on the PDU were found to lie in the churn turbulent regime.
- D) Two- and three-phase fluidization experiments were carried out in Amoco's cold-flow fluid dynamics unit. The data base now includes coal char/kerosene slurry concentrations of 4.0, 9.8, and 20.7 vol% in addition to the 15.5 and 17.8 vol% data from our earlier work. Both HDS-2A and Amocat-1A catalysts were used in the tests. Bed expansion is primarily a function of slurry velocity, with gas velocity having only a weak effect. Bed contractions have been observed in some cases at sufficiently high gas velocity.
- E) Gas and liquid holdups were found to be uniform across the cross-section of the Amoco cold-flow fluid dynamics pilot plant.
- F) A significant degree of backmixing was found to occur in the H-Coal system as measured in the Amoco cold-flow unit. Dispersion coefficients and Peclet numbers were found to lie in the ranges 70 to 130 cm²/sec and 3.6 to 9.9, respectively.
- G) Three techniques were developed for the study of the bubble dynamics occurring in three-phase fluidized beds: 1) A laser light beam probe for measuring the behavior of bubbles greater than 120 microns; 2) A laser holographic technique for determining the size, shape, and position of bubbles in the bed; and 3) A resistivity probe for determining bubble frequencies and a qualitative measure of the flow regime.
- H) The bubble size studies in G) showed that the design of the inlet distributor played an important role in the breakup of bubbles entering the fluidized bed. It was shown that smaller bubbles can be generated by this particular inlet distributor than could be produced by the breakup of bubbles by solid particles within the bed.
- I) An equilibrium bubble size model was developed based on data obtained using the light beam probe.
- J) A viscometer was adapted for measurement of the viscosity of coal slurries at high temperature and pressure.

ACKNOWLEDGMENTS

Completion of the tasks of this study involved the efforts of many individuals. Of the several organizations involved in the various phases of the H-Coal fluid dynamics program, we wish to acknowledge the assistance and cooperation of the following individuals:

Hydrocarbon Research, Inc: E. Harris, D. Tanner, M. Merdinger and P. Stepanoff in scheduling and performing the fluid dynamics tests during the PDU-10 experimental run, and for obtaining samples and operating data from the reactor.

Amoco Research Center: C. F. Washington and J. E. Demps for performing the cold-flow H-Coal experiments in the Amoco pilot plant and assistance in the PDU-10 phase. A. P. Vander Klay, W. A. Kommendant, D. G. LeBlanc and J. A. Olson for their contributions in the design and construction of the apparatus. I. A. Vasalos and J. W. Gorman assisted in the design of the liquid mixing experiments and in the data analysis. Professor R. Aris of the University of Minnesota participated in fruitful discussions regarding data analysis from the mixing experiments. M. Lamski for an excellent job of preparing this manuscript. J. W. Jones, R. S. Hurlbut for their valuable computer assistance; and I. Ginsburgh for his thought-provoking ideas.

Battelle Memorial Institute: J. Droege for his modifications to the Battelle viscometer to extend its capabilities to the measurement of the density and viscosity of coal liquid slurries at reactor process conditions.

Oak Ridge National Laboratories: R. Hightower for his willing assistance in sharing details of experimental techniques and results of ORNL's viscosity and density measurements on H-Coal reactor slurries.

Northwestern University: The insights, new approaches, and experimental techniques contributed by Professors S. G. Bankhoff, M. C. Yuen, and R. S. Tankin were valuable in elucidating fundamental details of three-phase fluidization and in identifying new methods for detailed studies of bubble behavior.

REFERENCES

- 1) Vasalos, I. A., E. M. Bild, D. N. Rundell, and D. F. Tatterson, H-Coal Fluid Dynamics Final Technical Progress Report, DOE Contract No. DE-AC05-77ET-10149, 4/16/80.
- 2) Hydrocarbon Research, Incorporated, "PDU Run 10," DOE Contract No. DE-AC22-80PC-30026, 9/81.
- 3) Darton, R. C., and D. Harrison, "Gas and Liquid Holdup in Three-Phase Fluidization," Chem Eng Sci, **30**, 581 (1975).
- 4) Stewart, P. S. B., and J. F. Davidson, "Three-Phase Fluidization: Water Particles and Air," Chem Eng Sci, **19**, 319 (1964).
- 5) Ostergaard, K., "On Bed Porosity in Gas-Liquid Fluidization," Chem Eng Sci, **20**, 165 (1965).
- 6) Hills, J. H., "Radial non-Uniformity of Velocity and Voidage in a Bubble Column," Trans Inst Chem Engrs, **52**, 1 (1974).
- 7) Tsung, G. H., "Radial Void Fraction Distribution in Bubbly Flow," MS thesis, Chemical Engineering department, Northwestern University, Evanston, Illinois (1981).
- 8) Linneweber, K. W., and E. Blass, "Messung Ortlicher Gas und Feststoffgehalte in Blasensaulen mit in der Flüssigkeit Suspendiertem Feststoff," Chem Ing Tech, **54**, No. 7 (1982).
- 9) Wen, C. Y., and L. T. Fan, "Models for Flow Systems and Chemical Reactors," Chemical Processing and Engineering Monograph Series, Dekkar, New York (1975).

- 10) Shah, Y. T., G. J. Stiegel, and M. M. Sharma, "Backmixing in Gas-Liquid Reactors," AIChE J, 24, No. 3, 369 (1978).
- 11) Ostergaard, K., "Gas-Liquid Particle Operation in Chemical Reaction Engineering," Advances in Chemical Engineering, 7, 71, Academic Press, New York (1968).
- 12) Bischoff, K. B., "Mixing and Contacting in Chemical Reactors," Ind Eng Chem, 58, No. 11, 18 (1966).
- 13) Bickel, T. E., and M. G. Thomas, "Catalyst Deactivation in the H-Coal Coal Liquefaction Process I: Catalyst Residence Time Distribution," Ind Eng Chem Proc Des Dev, 21, 377 (1982).
- 14) Joshi, J. B., "Axial Mixing in Multiphase Contactor--A Unified Correlation," Trans Inst Chem Engrs, 58 (1980).
- 15) Aoyama, Y., K. Ogushi, K. Koide, and H. Kubota, "Liquid Mixing in Concurrent Bubble Columns," J Chem Eng Japan, 1, No. 2, 158 (1968).
- 16) Deckwer, W. D., R. Burckhart, and C. Zoll, "Mixing and Mass Transfer in Tall Bubble Columns," Chem Eng Sci, 29, 177 (1974).
- 17) Eissa, S. H., M. M. El-Halwagi, and M. A. Saleh, "Axial and Radial Mixing in a Concurrent Bubble Column," Ind Eng Chem Proc Des Dev, 10, No. 1, 31 (1971).
- 18) Kato, Y., and A. Nishiwaki, "Longitudinal Dispersion Coefficient of a Liquid in a Bubble Column," Int Chem Eng, 12, No. 1, 182 (1972).
- 19) Hikita, H., and H. Kikukawa, "Liquid-Phase Mixing in Bubble Columns: Effect of Liquid Properties," Chem Eng J, 8, 191 (1974).
- 20) Reith, T., S. Renken, and B. A. Israel, "Gas Holdup and Axial Mixing in the Fluid Phase of Bubble Column," Chem Eng Sci, 2, 619 (1968).
- 21) Towell, G. D., and G. H. Ackerman, "Axial Mixing of Liquid and Gas in Large Bubble Reactors," Proceedings of the Fifth European/Second International Symposium on Chemical Reaction Engineering, p B3-1, Amsterdam (1972).
- 22) Eissa, S. H., and K. Schugerl, "Holdup and Backmixing Investigation in Concurrent and Countercurrent Bubble Columns," Chem Eng Sci, 30, 1251 (1975).
- 23) Cova, D. R., "Axial Mixing in the Liquid Phase in Gas-Sparged Columns," Ind Eng Chem Proc Des and Dev, 13, No. 3 (1974).
- 24) Alexander, B. F., and Y. T. Shah, "Axial Dispersion Coefficient in Bubble Columns," Chem Eng J, 11, 153 (1976).
- 25) Argo, W. B., and D. R. Cova, "Longitudinal Mixing in Gas-Sparged Tubular Reactors," Ind Eng Chem Proc Des Dev, 4, No. 4, 352 (1965).
- 26) Pilhofer, T. H., H. F. Bach, and K. H. Mangartz, "Fifth International Symposium on Chemical Reaction Engineering, Houston, Texas," 372 (1978).

- 27) Baird, M. H., and R. G. Rice, "Axial Dispersion in Large Unbaffled Columns," Chem Eng J, 9, 171 (1975).
- 28) Ohki, Y., and H. Inoue, "Longitudinal Mixing of the Liquid Phase in Bubble Columns," Chem Eng Sci, 25, 1 (1970).
- 29) Parulekar, S. J., and Y. T. Shah, "Steady-State Behavior of Gas-Liquid-Solid Fluidized-Bed Reactors," Chem Eng J, 20, 21 (1980).
- 30) El-Temtamy, S. A., Y. O. El-Sharnouki, and M. M. El-Halwagi, "Liquid Dispersion in Gas-Liquid Fluidized Beds, Part I: Axial Dispersion. The Axially Dispersed Plug-Flow Model," Chem Eng J, 18, 151 (1979).
- 31) Ostergaard, K., "Holdup, Mass Transfer, and Mixing in Three-Phase Fluidization," AIChE Symposium Series, No. 176, 74, 82 (1978).
- 32) Cherry, R. S., A. C. Sharon, and Z. P. Chen, "Parameters Influencing Dispersion in a Three-Phase Fluidized Bed," ORNL Report ORNL/MIT-254 (1977).
- 33) Schugerl, K., "Experimental Comparison of Mixing Processes in Two- and Three-Phase Fluidized Beds," Proceedings of the International Symposium on Fluidization, 782 (1967).
- 34) Kato, Y., T. Fukuda, and S. Tanaka, "The Behavior of Suspended Solid Particles and Liquid in Bubble Columns," J Chem Eng Japan, 5, No. 2, 112 (1972).
- 35) Hu, T., T. Chi, and H. Chao, "Holdup and Axial Mixing in Three-Phase Fluidization," Fluidization Science and Technology Papers, China-Japan Symposium, 179 (1982).
- 36) Michelsen, M. L., and K. Ostergaard, "Holdup and Fluid Mixing in Gas-Liquid Fluidized Beds," Chem Eng J, 1, 37 (1970).
- 37) Kara, S., B. G. Keller, Y. T. Shah, and N. L. Carr, "Hydrodynamics and Axial Mixing in a Three-Phase Bubble Column," Ind Eng Chem Proc Des Dev, 21, 584 (1982).
- 38) Panvelker, S. V., J. W. Tierney, and Y. T. Shah, "Backmixing in a SRC Dissolver," Chem Eng Sci, 37, No. 10, 1582 (1982).
- 39) Ying, D. H. S., R. Sivasubramanian, S. F. Moujaes, and E. N. Givens, "Gas/Slurry Flow in Coal Liquefaction Processes," Final Technical Progress Report, DOE-TIC14801-30, Air Products and Chemicals, Incorporated (1982).
- 40) Horvath, M., C. Pikior, and S. P. Cavers, "Effect of Vertical Alignment on Continuous-Phase Axial Mixing in a Pilot Liquid/Liquid Spray Column," AIChE J, 28, No. 5, 858 (1982).
- 41) Flaschel, E., P. F. Fauquex, and A. Renken, "Zum Verhalten von Flüssigkeits-Wirbelschichten mit Einbauten," Chem Ing Tech, 54, No. 1, 54 (1982).

- 42) Joshi, J. B., and M. M. Sharma, "Mass Transfer Characteristics of Horizontal Sparged Contactors," Trans Inst Chem Engrs, 54, 42 (1976).
- 43) Schaefer, R. J., D. N. Rundell, and J. K. Shou, "Study of Ebullated Bed Fluid Dynamics for H-Coal," Quarterly Progress Report No. 9, July 1-September 30, 1982.
- 44) Schaefer, R. J., D. N. Rundell, and J. K. Shou, "Study of Ebullated Bed Fluid Dynamics for H-Coal," Quarterly Progress Report No. 10, October 1-December 31, 1982.
- 45) Shah, Y. T., Gas-Liquid-Solid Reactor Design, McGraw Hill, New York (1979).
- 46) Lee, J. C., Proc Third European Symp Chem Reac Eng, 211, Pergamon Press (1965).
- 47) Meernik, P. R., "An Optical Technique for the Determination of Bubble Size Distributions and its Application to a Three-Phase Fluidized Bed System," PhD dissertation, Northwestern University, Evanston, Illinois (1983).
- 48) Peterson, D. A., "Bed Expansion and Pressure Drop Measurements Used to Determine Volume Fractions in a Three-Phase Fluidized Bed," MS thesis, Northwestern University, Evanston, Illinois (1982).
- 49) Richardson, J. F., and W. N. Zaki, "Sedimentation and Fluidization," Trans Inst Chem Engrs, 32, 35 (1954).
- 50) Davidson, J. F., and D. Harrison, "The Behavior of a Continuously Bubbling Fluidized Bed," Chem Eng Sci, 21, 731 (1966).
- 51) Wallis, G. B., One-Dimensional Two-Phase Flow, McGraw Hill, New York (1969).
- 52) Schaefer, R. J., and D. N. Rundell, Quarterly Progress Report No. 1, "Study of Ebullated Bed Fluid Dynamics for H-Coal," Contract No. DE-AC22-80PC30026, December, 1980.
- 53) Hightower, J. R. (ORNL), letter to R. J. Schaefer (Amoco), Final Report, "H-Coal Product Physical Properties Measurement," 7/8/81.
- 54) Schaefer, R. J., and D. N. Rundell, Quarterly Progress Report No. 2, "Study of Ebullated Bed Fluid Dynamics for H-Coal," Contract DE-AC22-80PC30026, 1/81.
- 55) Pittsburgh Energy Resource Center, "The Synthoil Process," presented at the Symposium on Clean Fuels from Coal; IGT, Chicago, Illinois; 6/75.
- 56) Pittsburgh Energy Research Center, PERC/QTR-76/2 (1976).
- 57) Schaefer, R. J., and D. N. Rundell, Quarterly Progress Report No. 4, "Study of Ebullated Bed Fluid Dynamics for H-Coal," Contract No. DE-AC22-80PC30026, 7/81.

- 58) Droege, J. W., et al., "Final Report on Viscosity of H-Coal Samples," Battelle Columbus Laboratories, 6/30/81.
- 59) Levenspiel, O., Chemical Reaction Engineering, Second Edition, Chapter 9, John Wiley & Sons, Incorporated.
- 60) Ostergaard, K., and M. L. Michelsen, "On the Use of the Imperfect Tracer Pulse Method for Determination of Holdup and Axial Mixing," Canadian J Chem Eng, 47, 107 (1969).
- 61) Hopkins, M. J., A. J. Sheppard, and P. Eisenklans, "The Use of Transfer Function in Evaluating Residence Time Distribution Curves," Chem Eng Sci, 24, 1131 (1969).
- 62) Harris, E. C. (HRI), letter to D. N. Rundell (Amoco), "Reports of Tasks Performed, Contract 9929-P-126," 10/28/80.
- 63) Johanson, E. S., and A. G. Comolli, "Bench Scale Catalyst Evaluation Program," Hydrocarbon Research, Incorporated, Report FE-2547-28, 12/78.
- 64) Kohl, J., R. D. Zentener, and H. R. Lukens, Radioisotope Applications Engineering, Chapter 9, Van Nostrand (Princeton, New Jersey) (1961).
- 65) Oak Ridge National Laboratory Report ORNL-5612, "Fossil Energy Program, Quarterly Progress Report for the Period Ending September 30, 1979," 1/80.
- 66) Henriksen, H. K., and K. Ostergaard, "Characteristics of Large Two-Dimensional Air Bubbles in Liquids and in Three-Phase Fluidized Beds," Chem Eng J, 7, 141 (1974).
- 67) El-Temtamy, S. A., and N. Epstein, "Bubble Wake Solids Content in Three-Phase Fluidized Beds," Int J Multiphase Flow, 4 (1978).
- 68) Peebles, F. N., and H. J. Garber, Chem Eng Prog, 49, 2 (1953).
- 69) Bhatia, V. K., "Gas Holdup of a Bubble Swarm in Two-Phase Vertical Flow," AIChE J, 15, 466 (1969).
- 70) DOE Report, "EDS Coal Liquefaction Process Development-Phase V, Annual Technical Progress Report for July, 1980 - June 30, 1981," Contract No. FC01-77ET10069.

NOMENCLATURE

A	Cross-sectional area of the column
BE	Catalyst bed expansion, %
C	Concentration, ppm
d	Particle diameter, cm
d_m	Sauter mean bubble diameter
d_3	Particle diameter
d_t	Column diameter
$C(s)$	Laplace transform of C
C_D	Drag coefficient
D	Reactor diameter, feet
D_1	Dispersion coefficient, cm^2/sec
$F(s)$	Laplace transfer function
g	Gravitational acceleration constant, $980.6 \text{ cm}/\text{sec}^2$
Ga	Galileo number
H	Catalyst bed height
I_0	Gamma-ray intensity through empty reactor, counts/sec
I_1	Gamma-ray intensity through kerosene, counts/sec
I_m	Gamma-ray intensity through test conditions, counts/sec
K_0	Wake volume ratio, volume of wake/volume of bubble
L	Distance between sampling trays, 157.5 cm or material thickness
M	Mass of dry catalyst
n	Richardson-Zaki index
Pe	Peclet number
Q	Amount of tracer injected into the system
R	Radial position index, R/R_0
R_i	Radial sampling position
R_0	Radius of fluidization column
Re_t	Particle Reynolds number
S	Standard deviation or square root of the variance in log scale
ΔT	PDU reactor temperature minus ambient temperature, $^{\circ}\text{F}$
T	Sampling time corresponding to C, sec
T'	Sampling time corresponding to log-normal mean
T''	Sampling time corresponding to one log-normal standard deviation
T_m	Mean residence time, sec
U_g	Superficial gas velocity, cm/sec
U_l	Superficial liquid velocity, cm/sec

U_t	Particle terminal velocity, cm/sec
U_{tb}	Bubble terminal velocity, cm/sec
U_s	Gas/liquid slip velocity, cm/sec
U_z	Average linear liquid velocity, cm/sec
$\dot{V}$	Volumetric liquid feed rate, ft ³ /sec
V_{CD}	Drift flux, cm/sec
X_k	Solid concentration ratio in wake
z	Axial position, cm
Z	Axial position, dimensionless (z/L)

Greek

α	Thermal expansion coefficient, in/in °F
θ	Sampling time, dimensionless
σ	Second moment or square root of the variance, or surface tension
σ_θ	σ/T_m
μ	First moment or mean of the RTD curve, or viscosity in centipoise
ϵ_c	Volume fraction catalyst, dimensionless
ϵ_{co}	Settled catalyst volume fraction, dimensionless
ϵ_f	Volume fraction fines, dimensionless
ϵ_g	Volume fraction gas, dimensionless
ϵ_l	Volume fraction liquid, dimensionless
ϵ_w	Volume fraction wake, dimensionless
ϵ_{sl}	Volume fraction slurry = $\epsilon_l + \epsilon_f$
ρ_p	Catalyst dry particle density, g/cm ³
ρ_c	Catalyst soaked particle density, g/cm ³
ρ_f	Fines density, g/cm ³
ρ_l	Liquid density, g/cm ³
ρ_{sl}	Slurry density, g/cm ³
$\Delta\rho$	Solid/liquid density difference
ρ_B	Catalyst bulk density, g/cm ³
μ_c	Catalyst mass absorption coefficient
μ_f	Fines mass absorption coefficient
μ_l	Liquid mass absorption coefficient
ω_f	Weight percent fines
τ	Shear stress, gm/cm-sec
τ_o	Yield stress, gm/cm-sec
η_{pl}	Platic viscosity, cp
$\dot{\gamma}$	Shear rate, sec ⁻¹
Δ	Difference (in predicted and observed volume fractions)

TABLE I

SUMMARY OF COLD-FLOW EXPERIMENTAL RUNS

<u>Run No.</u>	<u>Catalyst</u>	<u>Gas</u>	<u>Liquid</u>	<u>Fines, Vol% (Wt%)</u>	<u>Temperature, OF</u>
218	HDS-2A	Nitrogen	Kerosene	0.0 (0.0)	76
221	HDS-2A	Nitrogen	Kerosene	4.0 (7.11)	72-83
222	HDS-2A	Nitrogen	Kerosene	9.8 (16.7)	70-72
223	HDS-2A	Nitrogen	Kerosene	20.7 (32.4)	65-81
224	Amocat-1A	Nitrogen	Kerosene	15.4 (25.2)	68-69

Liquid Velocity Range, Ft/SecGas Velocity Range, Ft/Sec

218	0.072-0.212	0-0.223
221	0.052-0.210	0-0.136
222	0.048-0.195	0-0.224
223	0.051-0.207	0-0.213
224	0.046-0.173	0-0.152

TABLE II

RICHARDSON-ZAKI CORRELATION PARAMETERS

<u>Run No.</u>	<u>218</u>	<u>221</u>	<u>222</u>	<u>223</u>	<u>224</u>
Fines, Wt%	0.0	7.11	16.7	32.44	25.2
Fines, Vol%	0.0	4.0	9.8	20.7	15.4
Viscosity, cp	1.39	3.8	4.2	4.9	4.5
Catalyst	-----HDS-2A-----				Amocat-1A
Soaked Particle Density, Lb/Ft ³	-----107.9-----				86.89
Length, Inches	-----0.247-----				0.274
Diameter, Inches	-----0.062-----				0.062
U _t , Ft/Sec	0.43	0.355	0.412	0.326	0.350
n	2.60	2.07	2.88	3.11	3.06

TABLE III

COLD-FLOW ZERO SCAN DATA
(REACTOR FILLED WITH KEROSENE ONLY)

Average Scan <u>(cps)</u>	Standard Deviation <u>(cps)</u>	Radial Position <u>(R_i/R_o)</u>
378.17	3.72	-0.75
213.43	7.95	-0.50
288.67	7.10	-0.25
276.20	3.82	0
281.80	5.75	0.25
245.50	6.28	0.50
163.67	6.09	0.75

TABLE IV
H-COAL FLUIDIZATION RADIAL SCAN DATA

RUN490-5 FINES=25 WT% GAS VEL.=0.1 FT/S LIQ. VEL.=0.1 FT/S

ELEVATION (IN)	AVERAGE SCAN (CPS)	STANDARD DEV (CPS)	RADIAL POSITION RI/RO
30	319.8	6.7	-0.75
30	186.8	2.9	-0.50
30	217.4	2.9	-0.25
30	207.8	4.4	0.0
30	214.6	4.3	0.25
30	225.8	6.5	0.50
30	172.6	7.6	0.75
50	315.0	2.3	-0.75
50	183.6	1.1	-0.50
50	223.2	8.7	-0.25
50	206.6	3.0	0.0
50	206.6	4.2	0.25
50	187.0	6.9	0.50
50	142.8	5.4	0.75
70	326.0	5.8	-0.75
70	190.2	4.2	-0.50
70	224.2	6.0	-0.25
70	212.0	3.8	0.0
70	209.2	4.1	0.25
70	185.2	5.4	0.50
70	148.8	3.5	0.75
90	364.6	10.3	-0.75
90	238.6	3.0	-0.50
90	293.6	3.9	-0.25
90	279.8	8.1	0.0
90	280.8	3.9	0.25
90	199.2	3.7	0.50
90	273.0	6.2	0.75
110	350.6	2.4	-0.75
110	227.4	4.0	-0.50
110	290.2	4.5	-0.25
110	277.4	4.7	0.0
110	279.2	4.4	0.25
110	197.6	1.7	0.50
110	255.4	2.7	0.75

TABLE IV
H-COAL FLUIDIZATION RADIAL SCAN DATA
-2-

RUN490-6 FINES=25 WT% GAS VEL. =.08 FT/S LIQ. VEL. =.125 FT/S

ELEVATION (IN)	AVERAGE SCAN (CPS)	STANDARD DEV (CPS)	RADIAL POSITION RI/RO
30	327.2	5.4	-0.75
30	201.2	3.7	-0.50
30	230.6	3.5	-0.25
30	225.2	5.3	0.0
30	225.6	6.3	0.25
30	216.8	4.3	0.50
30	169.0	1.9	0.75
50	324.0	3.0	-0.75
50	183.8	3.6	-0.50
50	226.6	5.6	-0.25
50	222.6	2.9	0.0
50	220.6	5.1	0.25
50	215.4	3.0	0.50
50	151.8	3.3	0.75
70	331.8	4.1	-0.75
70	175.4	3.8	-0.50
70	234.0	4.4	-0.25
70	215.6	3.2	0.0
70	215.6	3.6	0.25
70	195.4	4.7	0.50
70	140.4	3.0	0.75
90	349.6	2.4	-0.75
90	206.8	2.2	-0.50
90	254.2	2.8	-0.25
90	239.2	16.6	0.0
90	238.0	4.3	0.25
90	179.8	7.1	0.50
90	150.8	6.5	0.75
110	358.6	4.2	-0.75
110	249.8	5.4	-0.50
110	289.6	3.4	-0.25
110	277.4	8.9	0.0
110	285.6	5.8	0.25
110	193.2	3.1	0.50
110	216.2	1.6	0.75

TABLE IV
H-COAL FLUIDIZATION RADIAL SCAN DATA

-3-

RUN490-7 FINES=25 WTX GAS VEL. =.08 FT/S LIQ. VEL. =0.1 FT/S

ELEVATION (IN)	AVERAGE SCAN (CPS)	STANDARD DEV (CPS)	RADIAL POSITION RI/RO
30	311.6	5.1	-0.75
30	190.8	1.8	-0.50
30	222.2	5.1	-0.25
30	209.6	3.8	0.0
30	210.8	5.6	0.25
30	203.0	4.3	0.50
30	163.2	4.6	0.75
50	318.2	3.5	-0.75
50	171.4	2.4	-0.50
50	221.2	2.4	-0.25
50	210.0	6.2	0.0
50	209.6	6.1	0.25
50	209.2	6.0	0.50
50	144.8	4.8	0.75
70	322.2	4.3	-0.75
70	168.2	5.6	-0.50
70	226.6	2.1	-0.25
70	214.6	3.0	0.0
70	207.8	4.7	0.25
70	190.2	1.5	0.50
70	141.4	3.4	0.75
90	365.8	6.7	-0.75
90	226.6	4.3	-0.50
90	289.0	4.9	-0.25
90	277.0	4.7	0.0
90	277.2	4.1	0.25
90	186.4	7.1	0.50
90	269.0	2.2	0.75
110	353.0	5.4	-0.75
110	220.4	3.8	-0.50
110	284.6	5.9	-0.25
110	277.4	4.3	0.0
110	276.0	8.2	0.25
110	190.6	3.1	0.50
110	212.8	4.1	0.75

TABLE IV
H-COAL FLUIDIZATION RADIAL SCAN DATA
-4-

RUN490-B FINE8=25 WT% GAS VEL.=0.1 FT/S LIQ. VEL.=.125 FT/S

ELEVATION (IN)	AVERAGE SCAN (CPS)	STANDARD DEV (CPS)	RADIAL POSITION RI/RO
30	328.8	0.8	-0.75
30	206.6	4.8	-0.50
30	236.8	5.8	-0.25
30	223.2	7.9	0.0
30	226.8	2.3	0.25
30	223.6	4.6	0.50
30	163.0	3.2	0.75
50	324.4	4.9	-0.75
50	173.0	1.9	-0.50
50	240.0	6.7	-0.25
50	218.4	3.3	0.0
50	217.8	3.4	0.25
50	205.4	3.8	0.50
50	138.2	2.8	0.75
70	341.0	4.4	-0.75
70	174.0	2.7	-0.50
70	233.4	5.0	-0.25
70	219.0	5.2	0.0
70	216.2	5.4	0.25
70	177.2	4.9	0.50
70	139.8	5.0	0.75
90	342.0	3.5	-0.75
90	212.6	1.9	-0.50
90	253.4	5.2	-0.25
90	234.8	5.0	0.0
90	239.0	2.5	0.25
90	180.0	1.6	0.50
90	284.4	1.3	0.75
110	360.0	3.5	-0.75
110	244.8	3.1	-0.50
110	283.8	2.6	-0.25
110	282.2	2.2	0.0
110	281.4	4.4	0.25
110	196.8	3.3	0.50
110	222.0	3.4	0.75

RADIAL PHASE HOLDUPS FOR RUN491-5

BED HEIGHT=70 IN. GAS FLOW RATE=0.10 FT/S LIQ. FLOW RATE=0.10 FT/S

ELEVATION INCH	VOLUME FRACTION			RADIAL POSITION RI/RO
	CATALYST	SLURRY	GAS	
30	0.331	0.570	0.100	-0.75
30	0.331	0.563	0.107	-0.50
30	0.331	0.572	0.097	-0.25
30	0.331	0.572	0.098	0.0
30	0.331	0.572	0.098	0.25
30	0.331	0.559	0.111	0.50
30	0.331	0.546	0.123	0.75
50	0.331	0.571	0.098	-0.75
50	0.331	0.564	0.105	-0.50
50	0.331	0.570	0.097	-0.25
50	0.331	0.572	0.097	0.0
50	0.331	0.574	0.095	0.25
50	0.331	0.574	0.095	0.50
50	0.331	0.566	0.103	0.75
70	0.331	0.568	0.102	-0.75
70	0.331	0.561	0.108	-0.50
70	0.331	0.570	0.099	-0.25
70	0.331	0.570	0.099	0.0
70	0.331	0.573	0.096	0.25
70	0.331	0.575	0.095	0.50
70	0.331	0.562	0.107	0.75
90	0.0	0.881	0.119	-0.75
90	0.0	0.868	0.132	-0.50
90	0.0	0.876	0.124	-0.25
90	0.0	0.876	0.124	0.0
90	0.0	0.877	0.123	0.25
90	0.0	0.894	0.106	0.50
90	0.0	0.823	0.177	0.75
110	0.0	0.885	0.115	-0.75
110	0.0	0.872	0.129	-0.50
110	0.0	0.876	0.124	-0.25
110	0.0	0.876	0.124	0.0
110	0.0	0.877	0.123	0.25
110	0.0	0.894	0.106	0.50
110	0.0	0.830	0.170	0.75

RADIAL PHASE HOLDUPS FOR RUN491-6

-2-

BED HEIGHT=74 IN. GAS FLOW RATE=0.08 FT/S LIQ. FLOW RATE=0.125FT/S

ELEVATION INCH	VOLUME FRACTION			RADIAL POSITION RI/RO
	CATALYST	SLURRY	GAS	
30	0.313	0.585	0.102	-0.75
30	0.313	0.574	0.113	-0.50
30	0.313	0.586	0.102	-0.25
30	0.313	0.584	0.103	0.0
30	0.313	0.586	0.102	0.25
30	0.313	0.579	0.108	0.50
30	0.313	0.566	0.121	0.75
50	0.313	0.586	0.101	-0.75
50	0.313	0.582	0.106	-0.50
50	0.313	0.587	0.100	-0.25
50	0.313	0.585	0.103	0.0
50	0.313	0.587	0.100	0.25
50	0.313	0.580	0.107	0.50
50	0.313	0.577	0.110	0.75
70	0.313	0.583	0.104	-0.75
70	0.313	0.585	0.102	-0.50
70	0.313	0.583	0.103	-0.25
70	0.313	0.587	0.100	0.0
70	0.313	0.589	0.098	0.25
70	0.313	0.588	0.099	0.50
70	0.313	0.586	0.102	0.75
90	0.0	0.885	0.115	-0.75
90	0.0	0.879	0.121	-0.50
90	0.0	0.886	0.114	-0.25
90	0.0	0.887	0.113	0.0
90	0.0	0.889	0.111	0.25
90	0.0	0.902	0.098	0.50
90	0.0	0.885	0.115	0.75
110	0.0	0.862	0.118	-0.75
110	0.0	0.864	0.136	-0.50
110	0.0	0.877	0.123	-0.25
110	0.0	0.876	0.124	0.0
110	0.0	0.876	0.124	0.25
110	0.0	0.896	0.104	0.50
110	0.0	0.847	0.153	0.75

RADIAL PHASE HOLDUPS FOR RUN491-7

-3-

BED HEIGHT=70 IN. GAS FLOW RATE=0.08 FT/S LIQ. FLOW RATE=0.10 FT/S

ELEVATION INCH	VOLUME FRACTION			RADIAL POSITION RI/RO
	CATALYST	SLURRY	GAS	
30	0.331	0.572	0.097	-0.75
30	0.331	0.561	0.108	-0.50
30	0.331	0.571	0.099	-0.25
30	0.331	0.571	0.098	0.0
30	0.331	0.573	0.096	0.25
30	0.331	0.567	0.102	0.50
30	0.331	0.552	0.117	0.75
50	0.331	0.570	0.099	-0.75
50	0.331	0.570	0.100	-0.50
50	0.331	0.571	0.098	-0.25
50	0.331	0.571	0.098	0.0
50	0.331	0.573	0.096	0.25
50	0.331	0.565	0.105	0.50
50	0.331	0.565	0.104	0.75
70	0.331	0.569	0.100	-0.75
70	0.331	0.571	0.098	-0.50
70	0.331	0.569	0.100	-0.25
70	0.331	0.570	0.100	0.0
70	0.331	0.574	0.095	0.25
70	0.331	0.572	0.097	0.50
70	0.331	0.567	0.102	0.75
90	0.0	0.880	0.120	-0.75
90	0.0	0.872	0.128	-0.50
90	0.0	0.877	0.123	-0.25
90	0.0	0.877	0.123	0.0
90	0.0	0.878	0.122	0.25
90	0.0	0.899	0.101	0.50
90	0.0	0.824	0.176	0.75
110	0.0	0.884	0.116	-0.75
110	0.0	0.874	0.126	-0.50
110	0.0	0.878	0.122	-0.25
110	0.0	0.876	0.124	0.0
110	0.0	0.878	0.122	0.25
110	0.0	0.897	0.103	0.50
110	0.0	0.849	0.151	0.75

RADIAL PHASE HOLDUPS FOR RUN491-8

-4-

BED HEIGHT=74 IN. GAS FLOW RATE=0.10 FT/S LIQ. FLOW RATE=0.125FT/S

ELEVATION INCH	VOLUME FRACTION			RADIAL POSITION RI/RO
	CATALYST	SLURRY	GAS	
30	0.313	0.584	0.103	-0.75
30	0.313	0.572	0.115	-0.50
30	0.313	0.584	0.103	-0.25
30	0.313	0.584	0.103	0.0
30	0.313	0.585	0.102	0.25
30	0.313	0.577	0.110	0.50
30	0.313	0.570	0.117	0.75
50	0.313	0.586	0.102	-0.75
50	0.313	0.586	0.101	-0.50
50	0.313	0.583	0.104	-0.25
50	0.313	0.586	0.101	0.0
50	0.313	0.588	0.099	0.25
50	0.313	0.584	0.103	0.50
50	0.313	0.587	0.100	0.75
70	0.313	0.580	0.107	-0.75
70	0.313	0.586	0.101	-0.50
70	0.313	0.585	0.102	-0.25
70	0.313	0.586	0.102	0.0
70	0.313	0.589	0.099	0.25
70	0.313	0.596	0.091	0.50
70	0.313	0.586	0.101	0.75
90	0.0	0.887	0.113	-0.75
90	0.0	0.877	0.120	-0.50
90	0.0	0.866	0.114	-0.25
90	0.0	0.888	0.112	0.0
90	0.0	0.889	0.111	0.25
90	0.0	0.902	0.098	0.50
90	0.0	0.810	0.182	0.75
110	0.0	0.882	0.118	-0.75
110	0.0	0.866	0.134	-0.50
110	0.0	0.878	0.122	-0.25
110	0.0	0.875	0.125	0.0
110	0.0	0.877	0.123	0.25
110	0.0	0.895	0.105	0.50
110	0.0	0.845	0.155	0.75

TABLE VI
SUMMARY OF LIQUID-PHASE DISPERSION COEFFICIENT MODELS

Reference	Model	System
1) Towns and Ackerman (1972) (21)	$D_L = 73.5 d_c^{1.5} u_g^{0.5} (d_c \tau_c, u_g \tau_c/n)$	Air-Water
2) Deckwer, et al. (1974) (16)	$D_L = 0.678 d_c^{1.4} u_g^{0.3} (d_c \tau_c, u_g \tau_c/n)$	Air-Water
3) Cova (1974) (23)	$D_L = 73.7 u_g^{0.32} d_c^{1.07} \text{ (for } u_g \tau_c/n, 0.19/\text{cm}^2 \text{, for } d_c = 1.8 \text{ in.)}$ $D_L = 23.6 u_g^{0.45} d_c^{1.40} \text{ (for } d_c = 0.75 \text{ in.)}$	Methyl acetone, Cyclohexanol, Ethanol, Etc.)
4) Birkle and Kikukawa (1974) (19)	$D_L = (0.114 + 0.523 u_g^{0.77}) d_c^{1.25} u_g^{-0.12}$	Air (Water, NaOH, Sugar Solutions)
5) Kato and Nishiwaki (1972) (18)	$D_L = \frac{1}{1 + 6.5 (F_r)^{0.8}} \cdot u_g = d_c \tau_c, F_r = \frac{u_g}{\sqrt{g d_c}}$	Air-Water
6) Welch, et al. (1968) (20)	$F_g = 3.0 \pm 0.3 \text{ LE } 0.1 \leq u_g \leq 0.45 \text{ m/sec}$ and $d_c = 0.14 \text{ and } 0.29 \text{ m}$	Air (Water, Salt Solution)
7) Oishi and Inoue (1970) (28)	$D_L = 75.4 d_c^{1.2} u_g^{1.2} + 17,000 d_c \text{ (Bubble Regime)}$ $D_L = \frac{0.14 d_c}{(1 - c_g)} \text{ (Coalesced Bubble Slug Flow)}$	Air-Water
8) Baird and Rice (1975) (27)	$D_L = 0.35 d_c^{1.33} [9(u_g \tau_c/n)(1 - c_g)]^{1/3}$	Air-Water
9) Pilhofer, et al. (1978) (26)	$D_L = (0.15 + 0.69 u_g^{0.77}) (d_c^{1.25} / u_g^{1/3})$	Air-Water
10) Kato, et al. (1982) (37)	$D_L = 240.1 u_g^{0.205} u_g^{-0.235} (u_g \tau_c/n)$	Air-Water
11) Joshi (1980) (14)	$D_L = 0.33 d_c u_g \text{ (where } u_g = 1.31 [9 d_c (u_g - g u_g^{1/3})]^{1/3}$	Air-Water
Three-Phase Bubble Column		
12) Kato, et al. (1972) (34)	$F_g = \frac{u_g d_c}{13 F_r} = \frac{1}{1 + 8 F_r^{0.85}}, F_r = \frac{u_g}{\sqrt{g d_c}}$	Air-Water Slurry-(Glass Sphere, Copper Powder, Resin)
13) Kato, et al. (1982) (37)	$D_L = 179.6 u_g^{0.336} u_g^{1.0} \text{ for } d_p = 10 \mu\text{m}$ $D_L = 285.3 u_g^{0.262} u_g^{1.0} \text{ for } d_p = 30 \mu\text{m}$ $D_L = 165.9 u_g^{0.249} u_g^{1.0} \text{ for } d_p = 70 \mu\text{m}$ $F_g = 0.48 u_g^{0.157} u_g^{0.315} d_c^{-0.418} \text{ for } u_g \geq 5 \text{ cm/sec}$ $F_g = 11.58 u_g^{0.269} u_g^{1.375} d_c^{-0.212} \text{ for } u_g < 5 \text{ cm/sec}$	Air-(Water, Glycerine, Ethyl Acetate)-Glass Sphere
14) Hu, et al. (1982) (35)		Air-Water-Glass Sphere
15) Joshi (1980) (14)	$D_L = 0.29 d_c (u_g + u_g)$ where $u_g = 1.31 [9 d_c (u_g - g u_g^{1/3})]^{1/3}$ $- c_g u_g^{1/3}$	Air-Water-Glass Sphere Literature Data

*Units used in the models are (dim, cm/s, m/s, m/s) unless otherwise stated.

JMS/ml: 4/21/83

TABLE VII
DENSE-PHASE SLURRY MIXING RESULTS

<u>Pilot Plant Run No.</u>	<u>Superficial Gas Velocity (cm/Sec)</u>	<u>Superficial Liquid Velocity (cm/Sec)</u>	<u>Dispersion Coefficient (cm²/Sec)</u>	<u>Peclet Number</u>	<u>Radial Sampling Position (R/R₀)</u>
490-6	2.74	2.96	58.8	9.48	-1.0
490-7	1.62	2.96	49.4	9.2	-1.0
490-8	0.0	2.96	9.10	54.0	-1.0
490-9	1.62	4.42	39.3	16.2	-1.0
490-10	2.76	2.96	105.0	5.20	-0.5
490-11	2.80	2.96	76.0	9.90	0
490-12	2.99	2.96	209.0	3.63	0.5
490-13	2.77	2.96	132.0	7.18	0.5
490-14	0.0	2.95	12.5	40.9	0.5
490-15	0.0	3.05	7.68	57.3	0
490-16	0.0	2.96	13.3	44.2	-0.5
490-17	4.05	2.96	123.4	4.92	0
490-18	3.11	2.96	87.7	8.34	0
490-19	1.92	2.96	58.9	9.86	0
490-20	2.90	2.95	72.2	7.39	0
490-21	4.54	2.89	129.0	4.60	-1.0
490-22	2.89	3.81	119.0	9.66	0
490-23	2.87	2.90	94.3	6.84	0

R/R₀ = 0: Liquid samples drawn from the center of the reactor.
 = -1.0: Liquid samples drawn from the near-side wall of the reactor.
 = +1.0: Liquid samples drawn from the far-side wall of the reactor.

JKS/ml
 3/23/83

TABLE VIII
LEAST SQUARES EVALUATION
OF SLURRY DISPERSION CORRELATIONS

<u>Coefficient</u>	<u>Estimate</u>	<u>SE of Estimate</u>	<u>T-Value</u>
<u>Correlation 1: $Pe = A + [B + D(U_1 - 0.1) + E(R)] \exp(-C U_g)^*$</u>			
A	5.01	1.24	4.0
B	40.1	8.12	4.9
C	35.9	9.63	3.7
D	12.5	575	0.0
E	0.00368	11.5	0.0

Residual Root Mean Square = 0.32, df = 13.

Correlation 2: $Pe = A + [B + D(U_1 - 0.1)] \exp(-C U_g)$

A	3.11	3.17	1.0
B	26.25	7.68	3.4
C	21.28	10.9	2.0
D	427.46	67.7	0.6

Residual Root Mean Square = 0.53, df = 14.

Correlation 3: $Pe = A + B * \exp(-C U_g)$

A	5.05	1.11	4.5
B	42.91	6.50	6.6
C	35.48	7.65	4.6

Residual Root Mean Square = 0.27, df = 13.

Correlation 4: $Pc = A * \exp(-B U_g)$

A	40.26	6.61	6.1
B	17.83	1.91	9.3

Residual Root Mean Square = 0.35, df = 14.

U_g and U_1 are in ft/sec.

*R = radial position index = R_i/R_o , where R_i = sampling point radius and R_o = ID of column.

TABLE IX

SLURRY-PHASE DISPERSION MODEL STATISTICS

$$\ln(Pe) = \ln(a + b e^{C U_g})$$

$$\text{Min } \sum_{C=1}^n (\ln Pe_i - \ln Pe)^2$$

<u>Model</u>	<u>Coef- ficient</u>	<u>Estimate</u>	<u>Standard Error of Estimate</u>	<u>95% Confidence Interval</u>
4	b	40.26	6.61	26.1 → 54.4
	c	-17.83	1.91	-13.7 → -21.9
Residual Root Mean Square Error = 0.356, df = 14.				
3	a	5.05	1.11	2.65 → 7.45
	b	42.91	6.50	28.9 → 56.9
	c	-35.48	7.65	-19.0 → -52.0
Residual Root Mean Square Error = 0.272, df = 13.				

U_g in ft/sec.

TABLE X
LACK-OF-FIT TEST RESULTS
FOR SLURRY-PHASE DISPERSION MODELS

1) Error Estimate from $U_g = 0$ Tests (Assumes Effect of U_1 & R_1/R_0 are Negligible)

<u>U_1</u>	<u>Test</u>	<u>Pe</u>	<u>ln (Pe)</u>
2.96	490-8	54.0	3.9890
2.95	490-14	40.9	3.7111
3.05	490-15	57.3	4.0483
2.96	490-16	44.2	<u>3.7887</u>

$$\bar{x} = 3.8843$$

$$s = 0.1602, 3 \text{ df}$$

$$SSE = 3 (0.1602)^2 = 0.07698$$

2) Model 4 Lack-of-Fit Test

	<u>SS</u>	<u>df</u>	<u>MS</u>	<u>E (MS)</u>
Regression SSR	1.78004	14		
Error SSE	<u>0.07698</u>	<u>3</u>	0.02566	σ_o^2
Difference	1.70306	11	0.15482	$\sigma_o^2 + \sigma_{LF}^2$

$$F_3^{11} = \frac{0.15482}{0.02566} = 6.03 \quad \underline{0.05 < \alpha < 0.10}$$

α = Significance level.

TABLE X
LACK-OF-FIT TEST RESULTS
FOR SLURRY-PHASE DISPERSION MODELS

-2-

3) Model 3 Lack-of-Fit Test

	<u>SS</u>	<u>df</u>	<u>MS</u>	<u>E (MS)</u>
Regression SSR	0.96131	13		
Error SSE	<u>0.07698</u>	<u>3</u>	0.02566	σ_o^2
Difference	0.88433	10	0.088433	$\sigma_o^2 + \sigma_{LF}^2$

$$F_3^{10} = \frac{0.08843}{0.02566} = 3.44$$

$$\underline{0.10 < \alpha < 0.30}$$

TABLE XI
FLUID PROPERTIES

<u>CHEMICAL</u>	<u>DENSITY</u> <u>(GM/CM³)</u>	<u>VISCOSITY</u> <u>(CP)</u>	<u>SURFACE</u> <u>TENSION</u> <u>(DYNES/CM)</u>	<u>REFRACTIVE</u> <u>INDEX</u>	<u>% IN</u> <u>MIXTURE</u>
Dipropylene Glycol Monomethyl Ether	0.95 (1)	3.3 (2)	28.8 (2)	1.419 (1)	66
Diphenyl Ether	1.07 (3)	3.9 (2)	42.0 (3)	1.579 (2)	34
Mixture	0.99 (1)	3.2 (1)	29.0 (1)	1.474 (2)	

(1) 20°C.

(2) 25°C.

(3) 30°C.

TABLE XII
PARTICLE PROPERTIES

<u>Particles</u>	<u>Density (Gm/Cm³)</u>	<u>Average Dimensions (mm)</u>	<u>Type of Glass</u>
2 x 5 mm Cylinders	2.24	Diameter 2.1, Length 5.7	Pyrex
1 mm Spheres	2.49	1.19 ± 0.10	Soda Lime
2 mm Spheres	2.49	2.04 ± 0.07	Soda Lime
3 mm Spheres	2.49	2.99 ± 0.07	Soda Lime
5 mm Spheres	2.49	4.96 ± 0.06	Soda Lime

TABLE XIII
DATA FOR BUBBLE SIZE CORRELATION

<u>Particles</u>	<u>Fluid</u>	<u>Sauter Mean Diam.</u>	<u>d_n/d_3</u>	<u>$\sigma/(\Delta\rho d_3^2 \text{ g})$</u>
5mm Spheres	Solvents	0.133 cm ¹	0.266	0.0789
3mm Spheres	Solvents	0.144 cm ¹	0.480	0.219
2mm Spheres	Solvents	0.181 cm ¹	0.905	0.493
Cylinders	Solvents	0.161 cm	0.481 ²	0.211
6mm Spheres	Water	0.24 cm ³	0.400	0.132

Sphere Density = 2.49 gm/cm³

Cylinder Density = 2.24 gm/cm³

Solvent Mixture Properties: Density = 0.99 gm/cm
 Surface Tension = 29. dynes/cm

Water Properties: Density = 1.00 gm/cm
 Surface Tension = 70. dynes/cm

¹Based upon data at gas fluxes below 0.5 cm/sec and bed expansions greater than 20%.

²Based upon a volume equivalent diameter of 0.335 cm.

³Based upon a mean bubble size of 0.24 cm in a water fluidized bed of 0.6 cm diameter glass particles. (Lee, 1965.)

TABLE XIV
PDU TEST SCHEDULE SUMMARY

<u>Date (1980)</u>	<u>Period</u>	<u>Slurry Feed Rate, Lb/Hr*</u>	<u>Slurry Recycle Rate, GPM/Ft²</u>	<u>Makeup H₂ SCFH</u>	<u>Recycle Gas SCFH</u>
7/30	04A	822	47.1	3280	4178
8/05	10A	809	16.5	3485	4171
9/04	27A	606	35.9	2949	4330
9/11	34A	621	18.5	2935	4389
9/19A	41B	699	41.5	2850	4550
9/19P	42A	644	29.5	2900	4470
9/20A	42B	636	19.1	2860	4500
9/20P	43A	637	18.8	3610	6010
9/21A	43B	628	19.1	2830	2200
9/21P	44A	639	30.5	3940	6360
9/22A	44B	641	30.5	2790	2350
9/22P	45A	660	37.1	3690	6390
9/23A	45B	623	41.2	2850	2100
9/23P	46A	696	41.2	2820	4340

*Dry coal basis.

DNR/ml
12/31/80

TABLE XV
PDU VISCOSITY SAMPLE SCHEDULE

<u>Sample ID</u>	<u>Period Sample Was Taken</u>	<u>Date</u>
Amoco-1	130-93-04A	7/30/80
-2	130-93-04A	7/30/80
-3	130-93-10A	8/05/80
-4	130-93-20A	8/28/80
-5 ORNL-1	130-93-27A	9/04/80
-6 ORNL-2	130-93-34A	9/11/80
-7	130-93-41B	9/19/80 AM
-8	139-93-42A	9/19/80 PM
-9 ORNL-3	130-93-42A	9/19/80 PM
-10	130-93-42B	9/20/80 AM
-11 ORNL-4	130-93-43A	9/20/80 PM
-12	130-93-43B	9/21/80 AM
-13	130-93-44A	9/21/80 PM
-14	130-93-44B	9/22/80 AM
-15	130-93-45A	9/22/80 PM
-16	130-93-45B	9/23/80 AM

DNR/ml
12/31/80

TABLE XVI

SLURRY MIX TANK SAMPLE CHARACTERIZATION

<u>Sample Date</u>	<u>Specific Gravity, g/cc</u>	<u>Liquid % Oil (1)</u>	<u>Solid</u>		<u>% THF Soluble</u>
			<u>% Coal (2)</u>	<u>Insoluble Ash Oxide Wt% of THF Insolubles</u>	
8/05/80	1.2244	40	48	13.1	12
9/04/80	1.2094	33	--	14.4	--
9/11/80	1.2374	38	41	15.2	18
9/19 AM	1.174	56	32	12.7	10
9/19 PM	1.1781	57	34	13.6	9
9/20 AM	1.1943	60	32	14.9	7
9/20 PM	1.1800	53	40	10.3	2
9/21 AM	1.1981	50	40	12.9	8
9/21 PM	1.1925	54	39	13.6	4
9/22 AM	1.2427	51	40	16.1	5
9/22 PM	1.2460	43	43	15.1	8
9/23 AM	1.2504	25	47	18.1	19
9/23 PM	1.2853	48	44	20.6	4
Water	1.00				0.

Note: 1) % oil determined from millipore filtration.
 2) Defined to be THF insoluble.

DNR/ml: 2/19/81

TABLE XVII

ESTIMATED REACTOR EFFLUENT COMPOSITION

Amoco Test No.	-01	-02	--	-03	-04	-05	-06	-07	-08	-09	-10	-11	-12	-13	-14
1980 Date	7/30	8/05	--	9/04	9/11	9/19 AM	9/19 PM	9/20 AM	9/20 PM	9/21 AM	9/21 PM	9/22 AM	9/22 PM	9/23 AM	9/23 PM
HRI Period	4B	10A	20B	27B	34B	41B	42A	42B	43A	43B	44A	44B	45A	45B	46A
Amoco Sample	1,2	3	4	5	6	7	8,9	10	11	12	13	14	15	16	
400-975°F, Wt%	59.59	34.29	50.40	49.66	39.10	54.50	51.69	52.59	52.18	45.14	54.00	52.90	43.20	44.40	45.51
975+°F, Wt%	17.89	34.68	19.11	21.01	29.49	21.90	25.19	25.72	27.17	27.56	22.48	20.12	31.33	28.48	28.30
Unconverted Coal	11.99	18.68	16.22	14.89	17.14	12.45	12.06	11.37	10.91	14.65	12.16	15.32	14.44	13.48	12.87
Ash, Wt%	9.62	11.32	12.62	13.29	12.94	10.00	9.95	9.13	8.73	11.10	9.83	10.61	10.18	12.47	11.94

TABLE XVIII

PDU-10 CATALYST PARTICLE DENSITY HISTORY

<u>Amoco Test</u>	<u>Date</u>	<u>HRI Period</u>	<u>Soaked Particle Density g/cc (4)</u>	<u>Remarks</u>
1	7/30/80	4	1.552	(1)
--	8/3/80	8	1.552	(2)
2	8/5/80	10	1.552	
3	9/4/80	27	1.616	
4	9/11/80	34	1.643	
5	9/19/80 AM	41B	1.669	
6	9/19/80 PM	42A	1.673	
7	9/20/80 AM	42B	1.673	
8	9/20/80 PM	43A	1.677	
9	9/21/80 AM	43B	1.677	
10	9/21/80 PM	44A	1.681	
11	9/22/80 AM	44B	1.681	
12	9/22/80 PM	45A	1.684	
13	9/23/80 AM	45B	1.684	
14	9/23/80 PM	46A	1.688	
15	9/24/80 AM	46B	1.688	
16	9/24/80 PM	47	1.692	(3)

(1) Assumed.

(2) Table IV in Reference 54.

(3) Table 12 in Reference 2.

(4) All values are interpolated except for the 8/3/80 sample and the 9/24/80 Sample 16.

TABLE XIX

PDU REACTOR LIQUID VISCOSITIES AND DENSITIES

Sample ID	Period Sample Was Taken	Sample Date	Battelle			ORNL		
			T_o (1)	η_{pl} (2)	ρ (3)	T_o (4)	η_{pl} (5)	ρ (6)
Amoco-1	130-93-04A	7/30/80	0.115	1.40	0.832			
-2	130-93-04A	7/30/80	0.201	1.31	0.842			
-3	130-93-10A	8/05/80	0.134	3.12	0.947			
-4	130-93-20A	8/28/80	0.139	1.92	0.917			
-5	ORNL-1	130-93-27A (7)	0.056	1.56	0.941	0.1270	1.683	0.908
-6	ORNL-2	130-93-34A	0.142	2.38	0.987	0.3067	3.027	0.985
-7 (8)		130-93-41B	0.115	1.85	0.880			
-8		130-93-42A	0.117	2.90	0.903			
-9	ORNL-3	130-93-42A	0.062	1.95	0.901	0.2324	1.730	0.882
-10		130-93-42B	0.072	2.11	0.883			
-11	ORNL-4	130-93-43A	0.308	3.12	0.936	0.1911	1.070	0.838
-12		130-93-43B	0.065	1.66	0.894			
-13 (9)		130-93-44A	0.115	1.85	0.880			
-14		130-93-44B	0.002	0.90	0.914			
-15		130-93-45A	0.085	3.37	0.954			
-16		130-93-45B	0.048	2.63	0.947			
-17	(10)	130-93-46A	0.105	2.37	0.933			

Notes

- (1) N/m^2 .
- (2) $mPa.s$ (cp).
- (3) g/cc .
- (4) N/m^2 .
- (5) $mPa.s$.
- (6) g/cc .
- (7) Amoco-5 taken 9/4/80 at 1600 hours (Period 27A). ORNL-1 taken 9/4/80 at 1830 hours (Period 27B).
- (8) Sample was contaminated during testing and discarded. Properties estimated.
- (9) Sample bomb contained only gas. Properties estimated.
- (10) No sample taken in Period 46A. Properties estimated.

TABLE XX

X BED EXPANSION FOR HRI PDU-10 TESTS

CATALYST : AMOCAT-1A
 GAS : HYDROGEN
 LIQUID : COAL/OIL
 TEMPERATURE : 815. DEG F

Amoco Test No.	HRI Period	Liquid Flow Rate. Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	X Bed Expansion	Slurry Viscosity centipoise	Slurry Density g/cc
HRI-01	04B	0.119	0.070	161.	74.	1.71	0.837
-02	10A	0.045	0.071	148.	59.	3.42	0.947
-03	27B	0.085	0.065	148.	59.	1.69	0.941
-04	34B	0.046	0.065	148.	59.	2.69	0.987
-05	41B	0.102	0.066	151.	104.	2.11	0.880
-06	42A	0.073	0.064	124.	66.	2.50	0.902
-07	42B	0.051	0.065	112.	49.	2.27	0.888
-08	43A	0.050	0.086	124.	66.	3.80	0.900
-09	43B	0.050	0.043	112.	49.	1.8	0.894
-10	44A	0.076	0.091	157.	113.	2.11	0.880
-11	44B	0.075	0.045	117.	56.	0.91	0.914
-12	45A	0.088	0.090	161.	119.	3.36	0.934
-13	45B	0.096	0.042	129.	73.	2.74	0.947
-14	46A	0.098	0.065	145.	96.	2.6	0.938
-15	47A	-----	-----	78.	0.	---	1.058
-16	47A	-----	-----	78.	0.		1.080

TABLE XXI
 RESPONSE OF CATALYST BED EXPANSION
TO PROCESS VARIABLES

<u>Test Period</u>	<u>4 34B</u>	<u>6 42A</u>	<u>14 46A</u>	<u>13 45B</u>
Plastic Viscosity, cp	2.69	2.50	2.60	2.74
Liquid Velocity, Ft/Sec	0.046	0.073	0.098	0.096
Gas Velocity, Ft/Sec	0.065	0.064	0.065	0.042
% Catalyst Bed Expansion	59	66	96	73

TABLE XXII

COMPARISON OF CATALYST BED EXPANSIONS:
PDU PILOT PLANT VERSUS COLD-FLOW PILOT PLANT (RUN 224)

88-

PDU Test	PDU-10 Experiments		Test 224-	Cold-Flow Experiments		% Bed Expansion	
	U_1 , Ft/Sec	U_g , Ft/Sec		U_1 , Ft/Sec	U_g , Ft/Sec	PDU.	224
1	0.119	0.070	15	0.110	0.067	74	87
2	0.045	0.071	11	0.048	0.070	59	22
3	0.085	0.065	13	0.083	0.068	59	51
4	0.046	0.065	11	0.048	0.070	59	22
5	0.102	0.066	14	0.094	0.067	104	73
			15	0.110	0.067	104	93
6	0.073	0.064	12	0.072	0.069	66	38
7	0.051	0.065	11	0.048	0.070	49	22
8	0.050	0.086	16	0.045	0.090	66	11
9	0.050	0.043	10	0.047	0.043	49	7
10	0.076	0.091	17	0.074	0.090	113	42
11	0.075	0.045	9	0.073	0.041	56	40
12	0.088	0.090	18	0.083	0.090	119	49
13	0.096	0.042	7	0.093	0.046	73	71
14	0.097	0.065	14	0.094	0.067	96	73

TABLE XXIII

PDU Fluid Dynamics Data

Date: 9/24/80	7:52 PM
HRI Period:	47A
Slurry Feed, Lb/Hr	0.
Recycle, GPM/Ft ²	0.
H ₂ Feed Gas, SCFH	0.
Recycle Gas, SCFH	0.
Bed Height, Feet	6.50

Elev, Ft.	I-Zero CPS (1)	I-Flow CPS	I/I0	
3.12	2497.1	448.0	0.1794	
3.62	2492.5	445.6	0.1788	
4.13	1206.1	327.9	0.2719	
4.52	2496.2	408.8	0.1638	
5.06	2195.1	545.3	0.2484	
5.59	2360.5	522.5	0.2213	
6.09	2540.7	556.1	0.2189	
6.61	2074.1	505.6	0.2438	
7.13	2527.8	676.7	0.2677	
7.66	2543.7	682.4	0.2683	
8.16	2439.9	682.8	0.2799	
8.68	2402.5	663.5	0.2762	
8.96	2705.5	660.3	0.2441	
9.49	2409.2	661.0	0.2744	
10.01	2476.2	686.3	0.2772	
10.53	2432.0	674.8	0.2775	
11.03	2384.9	656.4	0.2752	
11.55	2482.2	664.0	0.2675	
12.07	2533.7	690.7	0.2726	
12.59	2453.5	660.8	0.2693	
13.09	2453.0	653.9	0.2666	
13.73	2453.0	677.5	0.2762	
14.14	2453.0	672.5	0.2742	
14.70	2453.0	664.5	0.2709	

↑

Catalyst
Bed

↓

↑

Liquid Only

↓

Note: (1). Values of I0 were interpolated from original zero scan data.

TABLE XXIV

CALCULATION OF OIL MASS ABSORPTION COEFFICIENT

<u>Amoco</u> <u>Test ID</u>	<u>Average</u> <u>Liquid</u> <u>Temp, °F</u>	<u>Liquid</u> <u>Density,</u> <u>g/cc (1)</u>	<u>L,</u> <u>In.</u>	<u>I/I_o</u>	<u>μ_L</u> <u>Ft²/lbm (2)</u>
924PM	203	1.080	8.5	0.2743 (3)	0.0271

- (1) See Figure 76 for variation of density with temperature.
- (2) Calculated from Equation 21, correcting for proper units.
- (3) Average between 9.49' and 11.55' elevation. See Table XXIII.

TABLE XXV
CALCULATION OF CATALYST MASS ABSORPTION COEFFICIENT

<u>Variable</u>	<u>Value</u>	<u>Reference</u>
I/I_0	0.2249	Table XXIII, 9/24/80 PM, 4.13-6.09' Elevation
L	8.5"	--
ϵ_C	0.6019	*
ϵ_L	0.3981	(1 - ϵ_C)
μ_L	0.0271 Ft ² /Lb	Table XXIV
ρ_L	67.5 Lb/Ft ³	Table XXIV
ρ_C	105.75 Lb/Ft ³	Table XVIII
μ_C	0.0217 Ft ² /Lb	Equation 20

*Calculated using $\rho_p = 1.186$ g/cc, $\rho_B = 0.714$ g/cc (Reference 63).

Various densities reported in Table 28, Reference 2, bracketed this value.

TABLE XXVI
MISCELLANEOUS DATA

		<u>Value Used</u>
α	Steel linear expansion coefficient	$6.7 \cdot 10^{-6}$ in./in. $^{\circ}$ F
L_{st}	2X thickness of PDU steel wall at 77 $^{\circ}$ F	2.75"
ρ_{st}	Steel density	490 lb/ft 3
ΔT	Temperature difference between ambient and process conditions	740 $^{\circ}$ F
μ_{gas}	Gas mass absorption coefficient	0.0542 ft 2 /lbm
μ_{sl}	Slurry mass absorption coefficient	0.0271 ft 2 /lbm
μ_c	Catalyst mass absorption coefficient	0.0217 ft 2 /lbm
μ_{st}	Steel mass absorption coefficient	0.0271 ft 2 /lbm

TABLE XXVII

CALCULATED HRI PDU HOLDUPS, DENSE PHASE

Amoco Test Number	HRI Period	Catalyst		Amocat-1A			
		Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	Gas	Hydrogen	Coal/Oil	Temperature
				ϵ_c	ϵ_s	ϵ_g	V_{CD} , mm/Sec
HRI-01	04B	0.119	0.070	0.346	0.786	-0.132	0.0
-02	10A	0.045	0.071	0.378	0.675	-0.053	0.0
-03	27B	0.085	0.065	0.378	0.467	0.155	9.4
-04	34B	0.046	0.065	0.378	0.430	0.191	11.0
-05	41B	0.102	0.066	0.295	0.333	0.372	-9.1
-06	42A	0.073	0.064	0.363	0.438	0.199	7.5
-07	42B	0.051	0.065	0.405	0.310	0.285	4.0
-08	43A	0.050	0.086	0.363	0.566	0.070	22.5
-09	43B	0.050	0.043	0.405	0.347	0.247	1.7
-10	44A	0.076	0.091	0.283	0.690	0.027	26.2
-11	44B	0.075	0.045	0.387	0.456	0.158	4.9
-12	45A	0.088	0.090	0.275	0.553	0.172	15.8
-13	45B	0.096	0.042	0.348	0.520	0.132	4.7
-14	46A	0.098	0.065	0.308	0.495	0.198	6.3

TABLE XXVIII

CALCULATED HRI PDU HOLDUPS, DILUTE PHASE

		Catalyst Gas Liquid Temperature	Amocat-1A Hydrogen Coal/Oil 815°F		
Amoco Test Number	HRI Period	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	<u>ε_l</u>	<u>ε_g</u>
-01	04B	0.119	0.070	0.823	0.177
-02	10A	0.045	0.071	0.763	0.237
-03	27B	0.085	0.065	0.776	0.224
-04	34D	0.046	0.065	0.756	0.244
-05	41B	0.102	0.066	0.571	0.429
-06	42A	0.073	0.064	0.659	0.341
-07	42B	0.051	0.065	0.496	0.504
-08	43A	0.050	0.086	0.810	0.190
-09	43B	0.050	0.043	0.575	0.425
-10	44A	0.076	0.091	0.927	0.073
-11	44D	0.075	0.045	0.630	0.370
-12	45A	0.088	0.090	0.778	0.222
-13	45B	0.096	0.042	0.727	0.273
-14	46A	0.098	0.065	0.768	0.232

TABLE XXIX
COMPARISON OF SLURRY AND CATALYST PROPERTIES
OF AMOCO COLD-FLOW AND HRI PDU TESTS

	<u>Amoco</u>	<u>Amoco</u>	<u>Amoco</u>	<u>Amoco</u>	<u>HRI</u>
Run	221	222	223	224	PDU-10
Slurry Fines, Wt%	7.11	16.7	32.44	25.0	~25.0
Slurry Density, g/cc	0.82	0.85	0.93	0.89	0.84-0.99
Slurry Viscosity, cp	3.8	4.2	4.9	4.5	0.9-3.5
Catalyst	----- HDS-1442A-----			----- Amocat-1A-----	

TABLE XXX

**AMOCAT-1A COMPARISON OF FLUID DYNAMIC VARIABLES:
PDU PILOT PLANT VERSUS COLD-FLOW PILOT PLANT (RUN 224)**

PDU			Run 224			ϵ_g	
Test	U_1	U_g	Test	U_1	U_g	PDU	224
1	0.119	0.070	15	0.110	0.067	--	0.09
2	0.045	0.071	21	0.048	0.070	--	0.03
3	0.085	0.065	13	0.083	0.068	0.15	0.08
4	0.046	0.065	11	0.048	0.070	0.19	0.03
5	0.102	0.066	14	0.094	0.067	--	0.09
6	0.073	0.064	15	0.110	0.067	--	0.09
7	0.051	0.065	12	0.072	0.068	0.20	0.10
8	0.050	0.086	11	0.048	0.070	0.25	0.03
9	0.050	0.043	16	0.045	0.010	0.07	0.06
10	0.076	0.091	10	0.047	0.043	0.25	0.06
11	0.075	0.045	17	0.074	0.090	0.03	0.09
12	0.088	0.090	9	0.073	0.041	0.16	0.05
13	0.096	0.042	18	0.083	0.090	0.17	0.09
14	0.097	0.065	7	0.093	0.046	0.13	0.06
			14	0.094	0.067	0.20	0.08

TABLE XXXI

BHATIA-EPSTEIN MODEL

<u>Equation</u>	<u>Comment</u>
1) $\epsilon_c + \epsilon_g + \epsilon_l = 1$	Gas + liquid + solid phase volume fractions add up to unity.
1a) $\epsilon_f + \epsilon_g + \epsilon_w = 1$	Fluidized bed + gas + wake phase volume fractions sum to unity.
2) $\epsilon_l = \epsilon_k (1 - X_k) + \epsilon_{lf}'' (1 - \epsilon_g - \epsilon_k + X_k \epsilon_k)$	Distribution of liquid between wake and fluidized liquid/solid phase.
3) $U_g = V_g \epsilon_g$	Defines linear gas velocity V_g . Assumes bubbles rise in plug flow.
4) $\epsilon_{lf}'' = \left[\frac{U_1 - V_g \epsilon_k (1 - X_k)}{U_t (1 - \epsilon_g - \epsilon_k)} \right]^{1/n}$	Assumes that Richardson-Zaki correlation describes behavior of particulate liquid fluidized phase. Modifications to U_1/U_t account for existence of gas bubble and wake phases and transport of catalyst and liquid in bubble wakes.
5) $V_g = \frac{U_1 + U_g + \epsilon_{lf}'' (1 - \epsilon_g - \epsilon_k) V_{g1}'''}{1 - \epsilon_c}$	A definition of V_{g1}''' , where $V_{g1}''' =$ relative velocity between gas in the bubble phase and liquid in the particulate phase. The manner in which V_{g1}''' is related to the bubble rise velocity will be discussed in the text.
6) Variation of (ϵ_k/ϵ_g) two-phase with operating conditions.	Independent model parameter. Correlate against U_1 .
7) Prediction of X_k .	Independent model parameter or literature correlation (Reference 67).
8) $(\epsilon_k/\epsilon_g)_{\text{three-phase}} = K_0 e^{-5.08 \epsilon_g}$ Where $K_0 = (\epsilon_k/\epsilon_g)$ for two-phase gas/liquid bed.	Wake volume ratio in a three-phase fluidized bed.

(Table Continued)

TABLE XXXI

BHATIA-EPSTEIN MODEL

-2-

86-

Equation

9) $v_{gl}''' = U_{tb} + 2U_g$

Comment

Relationship between bubble rise velocity and relative gas/liquid velocity. (This format was originally proposed by Towell (21) for two-phase systems to account for systematic circulation of the liquid.

TABLE XXXII

PREDICTED RICHARDSON-ZAKI TERMINAL VELOCITIES (U_t) AND INDICES (n)

<u>Amoco Test Number</u>	<u>HRI Period</u>	<u>Catalyst Density, g/cc</u>	<u>Slurry Density, g/cc</u>	<u>Slurry Viscosity, cp</u>	<u>Galileo Number</u>	<u>Terminal Reynolds Number</u>	<u>Terminal Velocity, Ft/Sec</u>	<u>Index n</u>
1	04B	1.55	0.837	0.0171	$7.49 \cdot 10^3$	100.5	0.48	2.81
2	10A	1.55	0.947	0.0342	$1.79 \cdot 10^3$	45.4	0.38	3.04
3	27B	1.62	0.941	0.0169	$8.14 \cdot 10^3$	105.1	0.44	2.79
4	34B	1.64	0.987	0.0269	$3.27 \cdot 10^3$	63.94	0.406	2.93
5	41B	1.67	0.880	0.0211	$5.71 \cdot 10^3$	86.82	0.484	2.85
6	42A	1.67	0.902	0.0250	$4.08 \cdot 10^3$	72.20	0.466	2.90
7	42B	1.67	0.888	0.0227	$4.955 \cdot 10^3$	80.38	0.478	2.87
8	43A	1.68	0.900	0.0380	$1.77 \cdot 10^3$	45.08	0.443	3.04
9	43B	1.68	0.894	0.0180	$7.91 \cdot 10^3$	103.47	0.484	2.80
10	44A	1.68	0.880	0.0211	$5.78 \cdot 10^3$	87.49	0.488	2.85
11	44B	1.68	0.914	0.0091	$3.11 \cdot 10^4$	209.6	0.485	2.61
12	45A	1.68	0.954	0.0356	$2.00 \cdot 10^3$	48.46	0.421	3.02
13	45B	1.68	0.947	0.0274	$3.41 \cdot 10^3$	65.35	0.440	2.92
14	46A	1.69	0.938	0.0260	$3.81 \cdot 10^3$	69.54	0.449	2.91

Note: Slurry viscosities as measured by Battelle.

TABLE XXXIII

PREDICTED RICHARDSON-ZAKI TERMINAL VELOCITIES (U_t) AND INDICES (n). ($\mu = 4 \mu$ Battelle)

<u>Amoco Test Number</u>	<u>HRI Period</u>	<u>Catalyst Density, g/cc</u>	<u>Slurry Density, g/cc</u>	<u>Slurry Viscosity, cp</u>	<u>Galileo Number</u>	<u>Terminal Reynolds Number</u>	<u>Terminal Velocity, Ft/Sec</u>	<u>Index, n</u>
1	04B	1.55	0.837	0.0683	$4.68 \cdot 10^2$	20.3	0.386	3.29
2	10A	1.55	0.947	0.1368	$1.12 \cdot 10^2$	7.97	0.268	3.62
3	27B	1.62	0.941	0.0676	$5.088 \cdot 10^2$	21.33	0.356	3.28
4	34B	1.64	0.987	0.1076	$2.047 \cdot 10^2$	11.92	0.303	3.47
5	41B	1.67	0.880	0.0844	$3.567 \cdot 10^2$	17.07	0.381	3.35
6	42A	1.67	0.902	0.0999	$2.55 \cdot 10^2$	13.75	0.355	3.42
7	42B	1.67	0.888	0.0908	$3.097 \cdot 10^2$	15.59	0.371	3.38
8	43A	1.68	0.900	0.152	$1.11 \cdot 10^2$	7.91	0.311	3.62
9	43B	1.68	0.894	0.0719	$4.94 \cdot 10^2$	20.94	0.392	3.28
10	44A	1.68	0.880	0.0843	$3.62 \cdot 10^2$	17.21	0.384	3.35
11	44B	1.68	0.914	0.0364	$1.93 \cdot 10^3$	47.41	0.439	3.03
12	45A	1.68	0.954	0.1424	$1.256 \cdot 10^2$	8.61	0.299	3.58
13	45B	1.68	0.947	0.1096	$2.13 \cdot 10^2$	12.23	0.329	3.46
14	46A	1.69	0.938	0.1040	$2.38 \cdot 10^2$	13.16	0.339	3.43

Note: Slurry viscosities = 4X values measured by Battelle.

TABLE XXXIV

TERMINAL VELOCITY OF ISOLATED BUBBLES IN LIQUIDS

<u>Region</u>	<u>Terminal Velocity, U_t</u>	<u>Range of Applicability</u>
1	$\frac{2r_e^2(\rho_l - \rho_g)g}{9\mu_l}$	$Re_b < 2$ where Re_b bubble Reynolds number = $\frac{2\rho_l U_t r_e}{\mu_l}$
2	$0.33g^{0.76} \left(\frac{\rho_l}{\mu_l}\right)^{0.52} r_e^{1.28}$	$2 < Re_b < 4.02M^{-0.214}$ where M Morton Number = $\frac{8\mu_l^4}{\rho_l \sigma^3}$
3	$1.35 \left(\frac{\sigma}{\rho_l r_e}\right)^{1/2}$	$4.02M^{-0.214} < Re_b < 3.10M^{-1/4}$ or $16.32M^{0.144} < G < 5.75$ where $G = \frac{8 r_e^4 U_t^4 \rho_l^3}{\sigma^3}$
4	$1.18 \left(\frac{g \sigma}{\rho_l}\right)^{0.25}$	$3.10 G < Re_b$ $5.75 < G$

Reference: Peebles, F. N., and Garber, H. J., "Studies on the Motion of Gas Bubbles in Liquids," Chemical Engineering Progress, 49, 2 (1953).

(5/ 9/83)

TABLE XXXV

BHATIA-EPSTEIN MODEL WITH PEBBLES/GARBER CORRELATION

RUN : 221

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 4.0 VOL %
 COAL CHAR CONC: 7.1 WT %
 TEMPERATURE : 83. DEG F

Test	U1 Ft/Sec	Ug Ft/Sec	Uth Ft/Sec	Xt	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1 Ft/Sec	Dh Cm
					Obs	Pred	Obs	Pred	Obs	Pred				
-25	0.052	0.049	0.322	0.229	0.53	0.54	0.38	0.37	0.09	0.09	0.02	0.107	-0.003	0.136
-26	0.055	0.079	0.545	0.544	0.52	0.52	0.39	0.38	0.10	0.10	0.02	0.104	0.024	0.206
-27	0.053	0.103	0.709	0.656	0.54	0.53	0.36	0.37	0.10	0.10	0.02	0.114	0.050	0.253
-28	0.052	0.128	0.907	0.730	0.52	0.53	0.38	0.37	0.10	0.10	0.03	0.103	0.075	(1)
-29	0.080	0.047	0.391	0.355	0.46	0.46	0.47	0.47	0.07	0.07	0.01	0.111	-0.033	0.159
-30	0.080	0.077	0.324	0.330	0.44	0.44	0.44	0.44	0.12	0.12	0.00	0.285	-0.002	0.137
-31	0.080	0.104	0.353	0.410	0.43	0.43	0.43	0.43	0.15	0.15	0.00	0.494	0.024	0.147
-32	0.079	0.129	0.462	0.539	0.43	0.43	0.42	0.42	0.15	0.15	0.00	0.513	0.050	0.181
-33	0.106	0.048	0.749	0.633	0.38	0.41	0.57	0.54	0.05	0.05	0.05	0.103	-0.058	(1)
-34	0.106	0.080	0.426	0.464	0.38	0.37	0.51	0.52	0.10	0.10	0.02	0.112	-0.026	0.170
-35	0.106	0.108	0.422	0.379	0.38	0.45	0.47	0.40	0.15	0.16	0.14	2.327	0.002	0.169
-36	0.107	0.134	0.404	0.463	0.37	0.37	0.46	0.46	0.17	0.17	0.00	1.124	0.027	0.163
-37	0.131	0.050	0.582	0.525	0.36	0.36	0.58	0.58	0.06	0.06	0.00	0.929	-0.082	0.217
-38	0.132	0.083	0.427	0.417	0.35	0.35	0.55	0.55	0.11	0.11	0.00	1.220	-0.049	0.170
-39	0.134	0.111	0.399	0.405	0.33	0.33	0.52	0.52	0.14	0.14	0.00	1.426	-0.022	0.161
-40	0.132	0.136	0.967	0.135	0.40	0.39	0.41	0.51	0.20	0.10	0.21	0.586	0.004	(1)
-41	0.161	0.053	0.649	0.545	0.31	0.31	0.63	0.63	0.06	0.06	0.00	1.678	-0.108	0.236
-42	0.159	0.087	0.554	0.511	0.39	0.30	0.60	0.60	0.10	0.10	0.00	1.667	-0.073	0.209
-43	0.158	0.116	0.546	0.528	0.29	0.29	0.58	0.58	0.12	0.12	0.00	1.605	-0.042	0.206
-44	0.177	0.052	0.672	0.572	0.28	0.28	0.67	0.67	0.05	0.05	0.00	1.493	-0.125	0.243
-45	0.181	0.086	0.670	0.573	0.25	0.26	0.65	0.65	0.09	0.09	0.00	1.805	-0.095	0.242
-46	0.206	0.052	0.525	0.416	0.24	0.24	0.70	0.70	0.06	0.06	0.00	2.572	-0.154	0.200
-47	0.201	0.077	0.504	0.417	0.24	0.24	0.67	0.67	0.09	0.09	0.00	2.319	-0.124	0.194
-48	0.102	0.068	0.524	0.524	0.38	0.40	0.54	0.52	0.08	0.08	0.03	0.111	-0.034	0.200
-49	0.102	0.106	0.379	0.433	0.38	0.38	0.48	0.48	0.14	0.14	0.00	0.609	0.004	0.155
-50	0.102	0.134	0.414	0.508	0.37	0.37	0.47	0.47	0.16	0.16	0.00	0.728	0.033	0.166
-51	0.131	0.135	0.375	0.464	0.31	0.31	0.53	0.52	0.16	0.16	0.01	1.187	0.003	0.154

Note: (1). Out of range for Peebles and Garber correlation.

(5/ 9/83)

TABLE XXXVI

BHATIA-EPSTEIN MODEL WITH PEEBLES/GARBER CORRELATION

RUN : 222

CATALYST : HDS2A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 9.8 VOL %
COAL CHAR CONC: 16.7 WT %
TEMPERATURE : 72. DEG F

Test	U1 Ft/Sec	Ug Ft/Sec	Uth Ft/Sec	Xk	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1 Ft/Sec	Db Cm
					Obs	Pred	Obs	Pred	Obs	Pred				
- 7	0.049	0.049	0.903	0.635	0.47	0.49	0.48	0.46	0.04	0.04	0.05	0.105	-0.000	(1)
- 8	0.072	0.049	0.454	0.314	0.43	0.43	0.50	0.50	0.07	0.07	0.00	0.559	-0.023	0.183
- 9	0.088	0.050	0.521	0.417	0.35	0.38	0.58	0.56	0.06	0.07	0.05	0.102	-0.038	0.203
-10	0.122	0.051	0.410	0.323	0.32	0.31	0.61	0.62	0.07	0.07	0.02	0.105	-0.071	0.169
-11	0.146	0.052	0.462	0.327	0.28	0.28	0.65	0.65	0.07	0.07	0.00	0.642	-0.093	0.185
-12	0.171	0.054	0.362	0.185	0.24	0.24	0.68	0.68	0.08	0.08	0.00	0.796	-0.117	0.153
-13	0.191	-0.054	0.368	0.198	0.21	0.21	0.71	0.71	0.08	0.08	0.00	0.904	-0.136	0.155
-15	0.047	-0.079	0.941	0.665	0.49	0.49	0.44	0.44	0.07	0.07	0.00	0.195	0.032	(1)
-16	0.074	0.077	0.586	0.497	0.40	0.40	0.51	0.51	0.09	0.09	0.00	0.197	0.003	0.223
-17	0.098	0.075	0.367	0.193	0.37	0.37	0.51	0.51	0.12	0.12	0.00	1.121	-0.023	0.155
-18	0.122	0.078	0.313	0.149	0.30	0.30	0.57	0.57	0.12	0.12	0.00	0.880	-0.044	0.137
-19	0.145	0.080	0.356	0.255	0.26	0.26	0.63	0.63	0.11	0.11	0.00	0.717	-0.065	0.151
-20	0.170	0.081	0.507	0.424	0.23	0.23	0.67	0.67	0.09	0.09	0.00	0.821	-0.089	0.199
-21	0.195	0.078	0.500	0.276	0.20	0.27	0.70	0.63	0.10	0.10	0.13	3.500	-0.117	0.197
-22	0.049	0.107	0.801	0.615	0.51	0.51	0.39	0.39	0.10	0.10	0.00	0.859	0.058	(1)
-23	0.073	0.107	0.678	0.578	0.41	0.41	0.49	0.49	0.11	0.11	0.00	0.494	0.034	0.250
-24	0.097	-0.108	0.388	0.289	0.37	0.37	0.48	0.48	0.15	0.15	0.00	1.342	0.011	0.161
-25	0.122	0.109	0.408	0.376	0.29	0.29	0.57	0.57	0.14	0.14	0.00	0.804	-0.012	0.168
-26	0.145	0.102	0.325	0.256	0.25	0.25	0.61	0.61	0.14	0.14	0.00	0.852	-0.043	0.141
-27	0.168	0.102	0.350	0.286	0.22	0.22	0.64	0.64	0.13	0.13	0.00	1.038	-0.066	0.149
-28	0.188	0.102	0.401	0.334	0.20	0.20	0.67	0.67	0.12	0.12	0.00	1.235	-0.086	0.166
-29	0.048	0.126	0.966	0.683	0.49	0.49	0.41	0.41	0.10	0.10	0.00	0.544	0.078	(1)
-30	0.073	0.128	0.834	0.661	0.40	0.40	0.50	0.50	0.11	0.11	0.00	0.315	0.055	(1)

Note: (1). Out of range for Peebles and Garber correlation.

(5/ 9/83)

TABLE XXXVI (cont'd)

BHATIA-EPSTEIN MODEL WITH PEEBLES/GARBER CORRELATION

RUN : 222
CATALYST : HDS2A
GAS : NITROGEN
LIQUID : KEFOSENE
COAL CHAR CONC: 9.8 VOL %
COAL CHAR CONC: 16.7 WT %
TEMPERATURE : 70. DEG F

Test	U1 Ft/Sec	Ug Ft/Sec	Uth Ft/Sec	Xk	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1 Ft/Sec	Db Cm
					Obs	Pred	Obs	Pred	Obs	Pred				
-31	0.096	0.131	0.481	0.460	0.34	0.34	0.51	0.51	0.15	0.15	3.00	0.852	0.034	0.191
-32	0.121	0.134	0.355	0.318	0.29	0.29	0.53	0.53	0.18	0.18	3.00	1.235	0.014	0.151
-33	0.132	0.137	0.399	0.451	0.25	0.25	0.60	0.50	0.16	0.16	3.00	0.159	0.005	0.165
-34	0.049	0.149	1.914	0.835	0.47	0.48	0.46	0.45	0.07	0.06	3.02	0.105	0.100	(1)
-35	0.073	0.153	0.969	0.710	0.39	0.39	0.50	0.50	0.11	0.11	0.00	0.229	0.080	(1)
-36	0.098	0.157	0.640	0.590	0.34	0.33	0.52	0.52	0.15	0.15	0.00	0.741	0.059	0.239
-37	0.097	0.104	0.435	0.403	0.34	0.34	0.53	0.53	0.13	0.13	0.00	0.544	0.007	0.177
-38	0.050	0.046	0.703	0.540	0.47	0.49	0.48	0.46	0.05	0.05	0.03	0.107	-0.004	0.257
-39	0.122	0.117	0.332	0.267	0.29	0.29	0.54	0.54	0.16	0.16	0.00	1.128	-0.005	0.143
-40	0.120	0.155	0.451	0.424	0.31	0.31	0.51	0.51	0.18	0.18	0.00	1.657	0.035	0.182
-41	0.144	0.160	0.387	0.396	0.25	0.25	0.56	0.56	0.19	0.19	0.00	1.394	0.016	0.161
-42	0.171	0.166	0.422	0.421	0.21	0.21	0.61	0.61	0.18	0.18	0.00	1.518	-0.005	0.172
-43	0.169	0.127	0.240	0.130	0.22	0.22	0.60	0.60	0.18	0.18	0.00	1.430	-0.042	0.111
-44	0.049	0.178	1.263	0.760	0.49	0.49	0.40	0.40	0.11	0.11	0.00	0.636	0.129	(1)
-45	0.074	0.185	1.063	0.738	0.40	0.40	0.48	0.48	0.12	0.12	0.00	0.552	0.112	(1)
-46	0.099	0.186	0.874	0.670	0.36	0.36	0.50	0.50	0.14	0.14	0.00	1.197	0.088	(1)
-47	0.121	0.186	0.485	0.551	0.31	0.31	0.51	0.45	0.18	0.24	0.11	3.497	0.065	0.192
-48	0.146	0.179	0.432	0.444	0.25	0.25	0.55	0.55	0.20	0.20	0.00	1.624	0.033	0.176
-49	0.169	0.187	0.425	0.466	0.20	0.20	0.60	0.60	0.19	0.19	0.00	1.465	0.018	0.173
-50	0.049	0.218	1.231	0.758	0.51	0.51	0.35	0.35	0.13	0.13	0.00	0.959	0.170	(1)
-51	0.073	0.213	1.176	0.764	0.40	0.40	0.47	0.47	0.13	0.13	0.00	0.594	0.140	(1)
-52	0.098	0.224	1.093	0.730	0.36	0.36	0.49	0.49	0.15	0.15	0.00	1.303	0.126	(1)
-53	0.049	0.050	0.868	0.622	0.47	0.49	0.48	0.46	0.05	0.05	0.04	0.108	0.000	(1)
-54	0.075	0.178	1.179	0.743	0.41	0.41	0.47	0.48	0.11	0.11	0.01	0.743	0.103	(1)
-55	0.098	0.183	0.889	0.684	0.35	0.34	0.52	0.52	0.14	0.14	0.00	0.823	0.085	(1)
-56	0.116	0.217	0.732	0.593	0.35	0.35	0.46	0.46	0.19	0.19	0.00	2.169	0.101	(1)

Note: (1). Out of range for Peebles and Garber correlation.

(5/ 9/83)

TABLE XXXVII

BHATIA-EPSTEIN MODEL WITH PEEBLES/GARBER CORRELATION

RUN : 223

CATALYST : HDS2A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 20.7 VOL %
COAL CHAR CONC: 32.4 WT %
TEMPERATURE : 81. DEG F

Test	U1 Ft/Sec	Ug Ft/Sec	Utb Ft/Sec	Xk	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1 Ft/Sec	Db Cm
					Obs	Pred	Obs	Pred	Obs	Pred				
- 7	0.052	0.047	0.636	0.585	0.44	0.44	0.50	0.50	0.06	0.06	0.00	0.975	-0.005	0.245
- 8	0.077	0.048	0.401	0.401	0.34	0.34	0.58	0.58	0.08	0.08	0.00	0.440	-0.029	0.171
- 9	0.100	0.049	0.354	0.300	0.30	0.30	0.61	0.61	0.09	0.09	0.00	1.170	-0.051	0.155
-10	0.126	0.050	0.354	0.423	0.24	0.23	0.69	0.68	0.07	0.08	0.01	0.772	-0.076	0.155
-11	0.146	0.051	0.373	0.349	0.21	0.21	0.71	0.71	0.08	0.08	0.00	1.093	-0.095	0.161
-12	0.049	0.078	1.891	0.862	0.44	0.43	0.52	0.53	0.04	0.04	0.02	0.116	0.028	(1)
-13	0.063	0.078	3.852	0.701	0.39	0.44	0.54	0.54	0.07	0.02	0.11	0.655	0.015	(1)
-14	0.087	0.079	0.489	0.497	0.33	0.33	0.57	0.57	0.11	0.11	0.00	1.206	-0.009	0.199
-15	0.109	0.079	0.505	0.521	0.27	0.27	0.63	0.63	0.10	0.10	0.00	1.072	-0.030	0.204
-16	0.133	0.079	0.406	0.425	0.23	0.23	0.66	0.66	0.11	0.11	0.00	1.398	-0.054	0.172
-17	0.048	0.101	1.227	0.790	0.44	0.45	0.49	0.49	0.07	0.07	0.00	0.643	0.053	(1)
-18	0.064	0.102	1.104	0.764	0.40	0.40	0.52	0.52	0.08	0.08	0.00	1.051	0.038	(1)
-19	0.088	0.102	0.727	0.664	0.33	0.33	0.57	0.57	0.10	0.10	0.00	1.222	0.014	(1)
-20	0.126	0.108	0.408	0.479	0.22	0.22	0.63	0.63	0.14	0.14	0.00	1.263	-0.019	0.173
-21	0.151	0.106	0.371	0.431	0.19	0.19	0.67	0.67	0.15	0.15	0.00	1.647	-0.045	0.161
-22	0.051	0.128	1.393	0.814	0.45	0.45	0.47	0.47	0.08	0.08	0.00	1.104	0.077	(1)
-23	0.076	0.126	1.020	0.762	0.35	0.35	0.56	0.56	0.09	0.09	0.00	0.846	0.030	(1)
-24	0.100	0.130	0.875	0.717	0.30	0.30	0.59	0.59	0.11	0.11	0.00	1.420	0.030	(1)
-25	0.126	0.129	0.593	0.602	0.24	0.24	0.62	0.62	0.14	0.14	0.00	1.724	0.002	0.232
-26	0.151	0.128	0.439	0.492	0.19	0.19	0.65	0.65	0.16	0.16	0.00	1.980	-0.023	0.183
-27	0.051	0.145	1.272	0.807	0.43	0.43	0.48	0.48	0.09	0.09	0.00	0.738	0.094	(1)
-28	0.076	0.151	1.310	0.810	0.35	0.35	0.56	0.56	0.09	0.09	0.00	0.837	0.075	(1)
-29	0.100	0.156	1.231	0.793	0.30	0.30	0.60	0.60	0.10	0.10	0.00	1.316	0.056	(1)
-30	0.126	0.160	0.836	0.698	0.25	0.25	0.61	0.61	0.14	0.14	0.00	2.068	0.034	(1)

Note: (1). Out of range for Peebles and Garber correlation.

(5/ 9/83)

TABLE XXXVII (cont'd)

BHATIA-EPSTEIN MODEL WITH PEEBLES/GARBER CORRELATION

RUN : 223

CATALYST : HDS2A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 20.7 VOL %
COAL CHAR CONC: 32.4 WT %
TEMPERATURE : 65. DEG F

Test	U1 Ft/Sec	Ug Ft/Sec	Uth Ft/Sec	Xt	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1 Ft/Sec	Db Cm
					Obs	Pred	Obs	Pred	Obs	Pred				
-31	0.051	0.173	1.565	0.847	0.39	0.40	0.52	0.51	0.09	0.09	0.02	0.106	0.122	(1)
-32	0.076	0.183	1.577	0.852	0.34	0.33	0.57	0.58	0.09	0.09	0.03	0.107	0.107	(1)
-33	0.100	0.184	1.206	0.789	0.31	0.31	0.57	0.57	0.12	0.12	0.00	1.840	0.083	(1)
-34	0.126	0.185	1.023	0.735	0.26	0.26	0.59	0.59	0.14	0.14	0.00	2.988	0.059	(1)
-35	0.177	0.056	0.446	0.417	0.17	0.17	0.75	0.75	0.08	0.08	0.00	1.899	-0.121	0.185
-36	0.052	0.205	1.283	0.816	0.43	0.43	0.44	0.44	0.12	0.12	0.00	1.059	0.153	(1)
-37	0.051	0.203	1.313	0.818	0.43	0.43	0.45	0.45	0.12	0.12	0.00	0.949	0.152	(1)
-38	0.076	0.210	1.567	0.843	0.35	0.35	0.54	0.54	0.11	0.11	0.00	1.043	0.134	(1)
-39	0.100	0.213	1.488	0.810	0.34	0.34	0.53	0.53	0.13	0.13	0.00	2.551	0.113	(1)
-42	0.100	0.111	0.741	0.672	0.29	0.29	0.60	0.60	0.11	0.11	0.00	1.237	0.011	(1)
-44	0.051	0.053	0.646	0.607	0.43	0.43	0.51	0.51	0.06	0.06	0.00	0.511	0.002	0.248
-45	0.126	0.168	0.849	0.702	0.25	0.25	0.61	0.61	0.14	0.14	0.00	2.191	0.041	(1)
-53	0.147	0.158	0.606	0.573	0.22	0.22	0.61	0.61	0.17	0.17	0.00	2.783	0.010	0.235
-54	0.177	0.145	0.541	0.512	0.17	0.17	0.66	0.66	0.17	0.17	0.00	3.071	-0.032	0.216
-55	0.177	0.104	0.480	0.428	0.19	0.19	0.68	0.68	0.14	0.14	0.00	3.021	-0.073	0.196
-56	0.177	0.085	0.493	0.432	0.19	0.19	0.70	0.70	0.11	0.11	0.00	2.853	-0.093	0.200
-59	0.199	0.058	0.517	0.453	0.15	0.15	0.78	0.78	0.07	0.07	0.00	2.672	-0.142	0.208
-60	0.199	0.072	0.434	0.386	0.15	0.15	0.76	0.76	0.10	0.10	0.00	2.470	-0.127	0.181
-61	0.198	0.119	0.518	0.480	0.15	0.15	0.71	0.71	0.14	0.14	0.00	3.117	-0.079	0.209
-62	0.198	0.131	0.561	0.509	0.15	0.15	0.70	0.70	0.15	0.15	0.00	3.283	-0.067	0.222
-63	0.207	0.144	1.072	0.687	0.15	0.15	0.73	0.73	0.12	0.12	0.00	4.303	-0.062	(1)
-64	0.198	0.087	0.502	0.446	0.16	0.16	0.73	0.73	0.11	0.11	0.00	3.053	-0.111	0.203

Note: (1). Out of range for Peebles and Garber correlation.

TABLE XXXVIII

BHATIA-EPSTEIN MODEL WITH PEBBLES/GARBER CORRELATION

RUN : 224

CATALYST : AMOCAT-1A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 15.4 VOL %
 COAL CHAR CONC: 25.0 WT %
 TEMPERATURE : 68. DEG F

Test	U1 Ft/Sec	Ug Ft/Sec	Utb Ft/Sec	Xk	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1 Ft/Sec	Db Cm
					Obs	Pred	Obs	Pred	Obs	Pred				
- 6	0.112	0.057	0.522	0.455	0.30	0.30	0.62	0.62	0.08	0.08	0.00	1.448	-0.056	0.206
- 7	0.093	0.046	0.514	0.460	0.34	0.34	0.60	0.60	0.06	0.06	0.00	0.958	-0.047	0.203
- 8	0.083	0.040	0.575	0.506	0.37	0.37	0.58	0.58	0.05	0.05	0.00	0.991	-0.043	0.222
- 9	0.073	0.041	0.594	0.503	0.41	0.41	0.53	0.53	0.05	0.06	0.00	1.557	-0.031	0.228
-10	0.047	0.043	0.677	0.536	0.54	0.54	0.40	0.40	0.06	0.06	0.00	1.891	-0.004	0.252
-11	0.048	0.070	1.939	0.850	0.47	0.47	0.49	0.49	0.03	0.03	0.00	0.735	0.022	(1)
-12	0.072	0.069	0.515	0.453	0.42	0.42	0.48	0.48	0.10	0.10	0.00	1.596	-0.003	0.204
-13	0.083	0.068	0.649	0.545	0.38	0.38	0.53	0.54	0.08	0.08	0.00	1.533	-0.014	0.244
-14	0.094	0.067	0.587	0.531	0.33	0.33	0.58	0.58	0.08	0.08	0.00	1.120	-0.027	0.226
-15	0.110	0.067	0.482	0.440	0.30	0.30	0.61	0.61	0.09	0.09	0.00	1.309	-0.043	0.193
-16	0.045	0.090	1.219	0.759	0.52	0.52	0.41	0.41	0.06	0.06	0.00	1.414	0.044	(1)
-17	0.074	0.090	0.772	0.630	0.41	0.41	0.50	0.50	0.09	0.09	0.00	1.529	0.016	(1)
-18	0.083	0.090	0.780	0.624	0.39	0.39	0.52	0.52	0.09	0.09	0.00	1.715	0.007	(1)
-19	0.093	0.090	0.569	0.537	0.34	0.34	0.55	0.55	0.11	0.11	0.00	1.411	-0.004	0.220
-20	0.112	0.089	0.481	0.454	0.30	0.30	0.58	0.58	0.12	0.12	0.00	1.644	-0.023	0.193
-21	0.113	0.102	0.523	0.508	0.29	0.29	0.58	0.59	0.13	0.13	0.00	1.511	-0.010	0.206
-22	0.094	0.101	0.740	0.628	0.34	0.34	0.56	0.56	0.10	0.10	0.00	1.410	0.007	(1)
-23	0.083	0.100	0.914	0.699	0.37	0.37	0.54	0.54	0.09	0.09	0.00	1.365	0.018	(1)
-24	0.073	0.100	0.994	0.705	0.42	0.42	0.50	0.50	0.08	0.08	0.00	1.658	0.028	(1)
-25	0.046	0.100	1.235	0.759	0.52	0.52	0.41	0.41	0.07	0.07	0.00	1.449	0.054	(1)
-26	0.046	0.148	1.718	0.827	0.54	0.54	0.38	0.38	0.08	0.08	0.00	1.650	0.102	(1)
-27	0.073	0.151	1.165	0.767	0.39	0.39	0.50	0.50	0.10	0.10	0.00	1.274	0.078	(1)
-28	0.083	0.152	1.144	0.745	0.39	0.40	0.49	0.49	0.11	0.11	0.00	1.907	0.069	(1)
-32	0.093	0.150	1.043	0.714	0.37	0.37	0.51	0.51	0.12	0.12	0.00	2.111	0.057	(1)
-33	0.112	0.152	0.765	0.607	0.34	0.34	0.51	0.51	0.15	0.15	0.00	2.650	0.040	(1)
-34	0.140	0.151	0.382	0.535	0.26	0.18	0.57	0.66	0.17	0.17	0.17	0.004	0.011	0.161
-35	0.140	0.096	0.289	0.342	0.23	0.19	0.63	0.67	0.14	0.14	0.08	0.054	-0.044	0.129
-36	0.140	0.084	0.369	0.359	0.23	0.23	0.65	0.65	0.12	0.12	0.00	1.369	-0.055	0.157
-37	0.143	0.067	0.385	0.310	0.24	0.24	0.66	0.66	0.10	0.10	0.00	1.460	-0.076	0.162
-38	0.140	0.039	0.344	0.266	0.24	0.23	0.69	0.71	0.06	0.06	0.03	0.116	-0.102	0.148

Note: (1). Out of range for Peebles and Garber correlation.

(5/ 5/83)

TABLE XXXIX

EMATIA-EPSTEIN MODEL WITH PEBBLES/GARBER CORRELATION

RUN : HRI PDU-10 TESTS

CATALYST : AMOCAT-1A

GAS : HYDROGEN

LIQUID : COAL/OIL

TEMPERATURE : 815. DEG F

Test	U1	Ug	Utb	Xk	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1	Db
	Ft/Sec	Ft/Sec	Ft/Sec		Obs	Pred	Obs	Pred	Obs	Pred			Ft/Sec	Cm
-03	0.085	0.065	0.337	C.583	0.38	0.38	0.47	0.47	0.15	0.15	0.00	4.751	-0.020	0.096
-04	0.046	0.065	0.108	C.016	0.38	0.40	0.43	0.41	0.19	0.19	0.04	0.104	0.019	0.047
-06	0.073	0.064	0.030	C.018	0.36	0.34	0.44	0.46	0.20	0.20	0.04	0.110	-0.009	0.022
-07	0.051	0.065	0.099	C.169	0.41	0.41	0.31	0.31	0.29	0.29	0.00	3.861	0.014	0.043
-08	0.050	0.086	1.940	C.999	0.36	0.36	0.57	0.57	0.07	0.07	0.00	8.684	0.036	(2)
-09	0.050	0.043	0.000	C.112	0.41	0.41	0.35	0.38	0.25	0.21	0.07	0.298	-0.007	0.002
-10	0.076	0.091	2.219	C.997	0.28	0.29	0.69	0.64	0.03	0.07	0.10	9.793	0.015	(2)
-11	0.075	0.045	4.207	C.546	0.39	0.51	0.46	0.48	0.16	0.01	0.30	0.103	-0.030	(2)
-12	0.088	0.090	0.247	C.987	0.28	0.27	0.55	0.55	0.17	0.17	0.00	1.808	0.002	0.101
-13	0.096	0.042	0.205	C.328	0.35	0.35	0.52	0.52	0.13	0.13	0.00	4.965	-0.054	0.079
-14	0.098	0.065	0.187	C.339	0.31	0.31	0.49	0.50	0.20	0.20	0.00	4.925	-0.033	0.072

Note: (1). PDU Reactor slurry viscosities assumed to be equal to Battelle measured viscosities of separator bottoms streams.

(2). Out of range for Peebles and Garber correlation.

TABLE XXXX

BHATIA-EPSTEIN MODEL WITH PEBBLES/GARBER CORRELATION

RUN : HRI PBU-10 Tests

CATALYST : AMCAT
 GAS : HYDROGEN
 LIQUID : COAL/OIL
 TEMPERATURE : 815. DEG F

Test	U1 Ft/Sec	Ug Ft/Sec	Utb Ft/Sec	Xt	Eps(Cat)		Eps(Liq)		Eps(Gas)		Delta	Ko	Ug-U1 Ft/sec	Db cm
					Obs	Pred	Obs	Pred	Obs	Pred				
-03	0.085	0.065	0.291	0.318	0.38	0.38	0.47	0.47	0.15	0.15	0.00	3.504	-0.020	0.150
-04	0.046	0.065	0.297	0.660	0.38	0.38	0.43	0.43	0.19	0.19	0.00	4.273	0.019	0.181
-06	0.073	0.064	0.224	0.321	0.36	0.36	0.44	0.44	0.20	0.20	0.00	4.204	-0.009	0.146
-07	0.051	0.065	0.114	0.128	0.41	0.42	0.31	0.31	0.29	0.27	0.03	3.233	0.014	0.084
-08	0.050	0.086	1.307	0.946	0.36	0.36	0.57	0.56	0.07	0.07	0.00	4.312	0.036	(1)
-09	0.050	0.043	0.052	0.011	0.41	0.40	0.35	0.35	0.25	0.25	0.01	4.130	-0.007	0.049
-10	0.076	0.091	3.130	1.000	0.28	0.28	0.69	0.68	0.03	0.04	0.02	9.994	0.015	(1)
-11	0.075	0.045	0.156	0.196	0.39	0.39	0.46	0.46	0.16	0.16	0.00	3.741	-0.030	0.073
-12	0.088	0.090	0.301	0.395	0.28	0.28	0.55	0.55	0.17	0.17	0.00	2.257	0.002	0.208
-13	0.096	0.042	0.286	0.310	0.35	0.35	0.52	0.52	0.13	0.13	0.00	5.981	-0.054	0.180
-14	0.098	0.065	0.229	0.245	0.31	0.31	0.49	0.49	0.20	0.20	0.00	5.055	-0.033	0.149

Notes:

- (1). Out of Range of Peebles and Garber Correlation
- (2). Reactor liquid viscosity assumed to be 4 times separator bottoms viscosity measured by Battelle

TABLE XXXXI

EFFECTIVE WAKE VOLUME RATIOS FOR BHATIA-EPSTEIN MODEL:
HRI PDU-10 TESTS

<u>Test Number</u>	<u>Gas Volume Fraction ϵ_g</u>	<u>Wake Ratio 2-Phase, K_0</u>	<u>Wake Volume to Bubble Volume Ratio, $K_0 \text{ exp}$ $(-5.08 \epsilon_g)$</u>
3	0.15	3.504	1.64
4	0.19	4.273	1.63
6	0.20	4.204	1.52
7	0.29	3.233	0.74
8	0.07	4.312	3.02
9	0.25	4.130	1.16
10	0.03	9.994	8.58
11	0.16	3.741	1.66
12	0.17	2.257	0.95
13	0.13	5.981	3.09
14	0.20	5.055	1.83

TABLE XXXXII

EFFECTIVE WAKE VOLUME RATIOS
 FOR BHATIA-EPSTEIN MODEL:
COLD-FLOW RUN 224 TESTS

<u>Test Number</u>	<u>ϵ_{gas} Gas Holdup</u>	<u>K_o</u>	<u>$\epsilon_w/\epsilon_{\text{gas}}$ Wake Volume Ratio</u>
-6	0.076	1.450	0.986
-7	0.064	0.972	0.702
-8	0.053	1.002	0.765
-9	0.055	1.551	1.173
-10	0.056	1.901	1.430
-11	0.033	0.749	0.633
-12	0.097	1.596	0.975
-13	0.082	1.592	1.050
-14	0.082	1.131	0.746
-15	0.092	1.313	0.823
-16	0.065	1.398	1.005
-17	0.092	1.530	0.959
-18	0.092	1.731	1.085
-19	0.108	1.389	0.802
-20	0.118	1.659	0.911
-21	0.126	1.516	0.799
-22	0.101	1.430	0.856
-23	0.086	1.353	0.874
-24	0.084	1.658	1.082
-25	0.072	1.456	1.010
-26	0.078	1.663	1.119
-27	0.103	1.303	0.772
-28	0.111	1.916	1.090
-32	0.119	2.118	1.157
-33	0.151	2.636	1.224
-34	0.167	2.636	1.129
-35	0.139	1.605	0.792
-36	0.121	1.371	0.741
-37	0.101	1.451	0.869
-38	0.062	1.048	0.765

TABLE XXXXI

EFFECTIVE WAKE VOLUME RATIOS FOR BHATIA-EPSTEIN MODEL:
HRI PDU-10 TESTS

<u>Test Number</u>	<u>Gas Volume Fraction ϵ_g</u>	<u>Wake Ratio, 2-Phase, K_O</u>	<u>Wake Volume to Bubble Volume Ratio, $K_O \text{ exp}$ $(-5.08 \epsilon_g)$</u>
3	0.15	3.504	1.64
4	0.19	4.273	1.63
6	0.20	4.204	1.52
7	0.29	3.233	0.74
8	0.07	4.312	3.02
9	0.25	4.130	1.16
10	0.03	9.994	8.58
11	0.16	3.741	1.66
12	0.17	2.257	0.95
13	0.13	5.981	3.09
14	0.20	5.055	1.83

TABLE XXXXII

EFFECTIVE WAKE VOLUME RATIOS
 FOR BHATIA-EPSTEIN MODEL:
COLD-FLOW RUN 224 TESTS

<u>Test Number</u>	<u>ϵ_{gas}, Gas Holdup</u>	<u>K_o</u>	<u>$\epsilon_w/\epsilon_{\text{gas}}$, Wake Volume Ratio</u>
- 6	0.076	1.450	0.986
- 7	0.064	0.972	0.702
- 8	0.053	1.002	0.765
- 9	0.055	1.551	1.173
-10	0.056	1.901	1.430
-11	0.033	0.749	0.633
-12	0.097	1.596	0.975
-13	0.082	1.592	1.050
-14	0.082	1.131	0.746
-15	0.092	1.313	0.823
-16	0.065	1.398	1.005
-17	0.092	1.530	0.959
-18	0.092	1.731	1.085
-19	0.108	1.389	0.802
-20	0.118	1.659	0.911
-21	0.126	1.516	0.799
-22	0.101	1.430	0.856
-23	0.086	1.353	0.874
-24	0.084	1.658	1.082
-25	0.072	1.456	1.010
-26	0.078	1.663	1.119
-27	0.103	1.303	0.772
-28	0.111	1.916	1.090
-32	0.119	2.118	1.157
-33	0.151	2.636	1.224
-34	0.167	2.636	1.129
-35	0.139	1.605	0.792
-36	0.121	1.371	0.741
-37	0.101	1.451	0.869
-38	0.062	1.048	0.765

Figure 1
H-Coal PDU Reactor

Figure 2

Ebullated Bed Composed of 3 Phases

Figure 3

Schematic Diagram of the Fluid Dynamics Unit

Figure 4.

Gamma Ray Scans Liquid Velocity 0.07 ft/sec

FIGURE 5

RICHARDSON ZAKI CORRELATION
RUN AU77-223
KEROSENE / 20.7 VOL % COAL CHAR / HDS-2A CATALYST

Figure 6

**Schematic Diagram of Drift Flux vs.
Gas Holdup: Darton and Harrison (3)**
(Gas/liquid/solid fluidization)

FIGURE 7

DRIFT FLUX DENSE PHASE, COLD FLOW RUN 218 **KEROSENE / NO FINES / HDS-2A CATALYST**

FIGURE 8

**DRIFT FLUX DENSE PHASE, COLD FLOW RUN 221
KEROSENE / 4 VOL % COAL CHAR FINES / HDS-2A CATALYST**

FIGURE 9

DRIFT FLUX DENSE PHASE, COLD FLOW RUN 222
KEROSENE / 9.8 VOL % COAL CHAR FINES / HDS-2A CATALYST

FIGURE 10

**DRIFT FLUX DENSE PHASE, COLD FLOW RUN 223
KEROSENE / 20.7 VOL % COAL CHAR FINES / HDS-2A CATALYST**

FIGURE 11

DRIFT FLUX DENSE PHASE, COLD FLOW RUN 224
KEROSENE / 15.4 VOL % COAL CHAR FINES / AMOCAT-1A CATALYST

Figure 12

Fines Concentration Effect on Gas Holdup

FIGURE 13

BED EXPANSION VS LIQUID VELOCITY
 $U_{gas} = 0.0 \text{ FT/SEC}$
EFFECT OF FINES CONCENTRATION

FIGURE 14

BED EXPANSION VS LIQUID VELOCITY
 $U_{\text{gas}} = 0.100 \text{ FT/SEC}$
EFFECT OF FINES CONCENTRATION

FIGURE 15

BED EXPANSION VS GAS VELOCITY
COLD FLOW RUN 222 --- KEROSENE /
16.7 WT% COAL CHAR FINES / HDS-2A CATALYST

FIGURE 16

BED EXPANSION VS GAS VELOCITY
COLD FLOW RUN 224 -- KEROSENE /
25.0 WT % COAL CHAR FINES / AMOCAT-1A CATALYST

Figure 17

Comparison of Axial Dispersion Coefficient for Various Multi-Phase Fluidization Systems

-130

Figure 18

Adsorption of Thiophene by Coal Char Fines

Figure 19

Input Pulse and the Resulting C-Curves for Tracer Injection at the Slurry Inlet Before the Bubble Cap Distributor

Figure 20

Schematic Diagram of Amoco Cold Flow Unit Modified for Liquid Mixing Experiment

AMOCO PILCT PLANT SAMPLING SYSTEM DETAILS

AMOCO PILOT PLANT SAMPLING SYSTEM TRAY

AMOCO PILOT PLANT SAMPLING PROBE

FIGURE 25
CONCENTRATION—TIME DISTRIBUTION CURVE
FOR LIQUID MIXING RUN 490-8

-138

Slurry Dispersion Data Analysis

Figure 27

H-Coal Slurry Dispersion Correlations

Figure 28

Liquid Mixing Material Balance

FIGURE 29
CONCENTRATION-TIME DISTRIBUTION CURVE
FOR LIQUID MIXING RUN 490-17

Figure 30 - Schematic Diagram of Experimental Apparatus.

Figure 31 - Metal Spool Piece.

Figure 32 - Inlet Distributor Section.

Figure 33 - Overflow Cup and Spiral Entrainment Separator.

Figure 34 - Solid Phase Volume Fraction

FIGURE 34A. Gas Holdup
(Liquid Flux = 4.80 ft/min.)

FIGURE 34B. Gas Holdup
(Liquid Flux = 6.45 ft/min.)

FIGURE 34C. Gas Holdup
(Liquid Flux = 8.10 ft/min.)

FIGURE 34D. Gas Holdup
(Liquid Flux = 9.75 ft/min.)

Figure 35 - Cross-Sectional View of a Light Beam Probe

Figure 36 - Schematic of Light Beam Probes Inserted into the Column

$$u = v \cdot \cos(\theta)$$

$$\tau = 2 \cdot r \cdot \cos(\theta) / v$$

Figure 37 - Bubble/Beam Intersection Geometry

Figure 38 - Typical Signals from Light Beam Probe
(50 Microseconds between Data Points)

Figure 39 - Bubble Size Distribution (Cylinders , LF = 4.01 cm/sec , GF = 0.44 cm/sec)

Figure 40 - Sauter Mean Diameters (Cylinders)

Figure 41 - Dimensionless Mean Bubble Diameter Versus the Inverse of the Eotvos Number

Figure 42 - OPTICAL SET-UP (OFF-AXIS HOLOGRAPHY) TO DETERMINE BUBBLE SIZES AND DISTRIBUTIONS.

Figure 43 - Holographic Analysis System

Figure 44 - Horizontal/Vertical Line Generator - External Connections and Signal Conditioning

Figure 45 - Horizontal/Vertical Line Generator Circuit Diagram

Figure 46 - Bubble Size Distribution with Gas Injection into the Liquid Inlet Line
($U_1 = 1.60$ cm/sec, $U_g = 0.05$ cm/sec)

Figure 47 - Surface Area Distribution with Gas Injection into the Liquid Inlet Line
($U_1 = 1.60$ cm/sec, $U_g = 0.05$ cm/sec)

Figure 48 - Void Fraction Distribution with Gas Injection into the Liquid Inlet Line
($U_1 = 1.60$ cm/sec, $U_g = 0.05$ cm/sec)

- 1 - 0.254 mm D music wire
- 2 - 1.02 mm I.D., 0.15 mm thick teflon tube
- 3 - 2.42 mm O.D. stainless steel tube
- 4 - Epoxy for sealing
- 5 - 6.3 mm O.D. stainless steel tube
- 6 - Rubber tube for fitting
- 7 - 12.7 mm O.D. stainless steel tube

Figure 49 - Resistivity Probe

Figure 50 - Diagram of the Probe Circuit

Figure 51

PDU Reactor System and Sampling Location

Figure 52

Side View of PDU Density Gauge Assembly

Legend

- 1 500 mc Cs-137 source
- 2 PDU reactor
- 3 Amoco scintillation detector
- 4 HRI Geiger/Muller detector

Figure 53

Top View of PDU Density Gauge Assembly

Legend

- 1 500 mc Cs-137 source
- 2 PDU reactor
- 3 Amoco scintillation detector
- 4 HRI Geiger/Muller detector
- 5 Traverse sled assembly

Figure 54

Gamma Ray Detection System Schematic

Notes

The SCA puts out a pulse if the signal from the amplifier is in the proper energy range. One pulse corresponds to one detected gamma-ray photon.

The rate meter is an analog indicator of the SCA pulse rate (counts/sec).

Figure 55

Energy Window Criteria

1. Lower edge: Minimum between background and Cs^{137} peak.
2. Upper edge: Defined as the energy where count rate = 10% of maximum rate.

Figure 56

Apparent Shift of Cs-137 Photopeak with Detector Temperature

FIGURE 57

PDU-10 ZERO GAMMA RAY SCAN

BATTELLE VISCOMETER SCHEMATIC

FIGURE 59

**PDU-10 REACTOR LIQUID SLURRY
VISCOSITY VS TEMPERATURE , AMOCO SAMPLE #5**

FIGURE 60

**PDU-10 REACTOR LIQUID SLURRY
YIELD STRESS VS TEMPERATURE , AMOCO SAMPLE #5**

FIGURE 61

PDU-10 REACTOR LIQUID SLURRY DENSITY VS TEMPERATURE , AMOCO SAMPLE #5

FIGURE 62

**PDU-10 REACTOR LIQUID SLURRY
HYSTERESIS OF VISCOSITY VS TEMPERATURE , AMOCO SAMPLE #8**

FIGURE 63

CHANGE OF PLASTIC VISCOSITY WITH TIME AT 725 K PDU LIQUID SAMPLE AMOCO-15

FIGURE 64

**CHANGE OF YIELD STRESS
WITH TIME AT 725 K
PDU LIQUID SAMPLE AMOCO-15**

FIGURE 65

CHANGE OF SLURRY DENSITY WITH TIME AT 725 K PDU LIQUID SAMPLE AMOCO-15

FIGURE 66

COMPARISON OF VISCOSITY OF ASPHALTENES AND PREASPHALTENES

Figure 67

OAK RIDGE NATIONAL LABORATORY
VISCOMETER SCHEMATIC

Schematic flow diagram of coal liquids flow system.

Schematic flow diagram of pipeline viscometer.

FIGURE 68

COMPARISON OF BATTELLE AND ORNL VISCOMETER TEMPERATURE HISTORIES

Figure 69

-182 TRENDS OF PLASTIC VISCOSITY VERSUS TIME (BATTELLE VISCOMETER)

FIGURE 70

PDU-10 REACTOR LIQUID SLURRY VISCOSITY CORRELATION

FIGURE 71

PDU-10 REACTOR LIQUID SLURRY DENSITY CORRELATION

FIGURE 72

PDU-10 REACTOR LIQUID SLURRY YIELD STRESS CORRELATION

FIGURE 73

**PDU-10 GAMMA RAY SCAN
I/I0 RATIO, TEST NUMBER 07
EXAMPLE OF SHARP CATALYST INTERFACE**

FIGURE 74

**PDU-10 GAMMA RAY SCAN
I/I₀ RATIO, TEST NUMBER 10
EXAMPLE OF DIFFUSE CATALYST BED INTERFACE**

Figure 75

H-Coal PDU Reactor Elevation Reference

Figure 76

HRI SHUTDOWN OIL DENSITY

-189

FIGURE 77

DRIFT FLUX DENSE PHASE PDU-10 FLUID DYNAMICS TESTS

Figure 78

Bhatia-Epstein Model

- Ebullated bed assumes 3 phases
 - Gas
 - Wake
 - Particulate

Assumptions:

- Particulate phase—liquid and catalyst only:
Described by Richardson-Zaki correlation
- Bubble wake—liquids and solids:
Described by wake volume ratio K_o
- Bubble rise velocity— U_{tB}

FIGURE 79

PREDICTION OF CATALYST PARTICLE TERMINAL REYNOLDS NUMBERS FOR PDU-10 TESTS AND COMPARISON WITH COLD FLOW DATA

FIGURE 80

**PREDICTION OF CATALYST RICHARDSON-ZAKI INDEX
FOR PDU-10 TESTS
AND COMPARISON WITH COLD FLOW DATA**

FIGURE 81

PREDICTION OF CATALYST PARTICLE TERMINAL REYNOLDS NUMBERS FOR PDU-10 TESTS AND COMPARISON WITH COLD FLOW DATA

FIGURE 82

**PREDICTION OF CATALYST RICHARDSON-ZAKI INDEX
FOR PDU-10 TESTS
AND COMPARISON WITH COLD FLOW DATA**

FIGURE 83

BHATIA EPSTEIN MODEL, RUN221
KEROSENE / 4 VOL % COAL CHAR / HDS-2A CATALYST
BUBBLE RISE VELOCITY CORRELATION

FIGURE 84

BHATIA EPSTEIN MODEL, RUN222
KEROSENE / 9.8 VOL % COAL CHAR / HDS-2A CATALYST
BUBBLE RISE VELOCITY CORRELATION

FIGURE 85

BHATIA EPSTEIN MODEL, RUN223
KEROSENE / 20.7 VOL % COAL CHAR / HDS-2A CATALYST
BUBBLE RISE VELOCITY CORRELATION

FIGURE 86

BHATIA EPSTEIN MODEL, RUN224
KEROSENE / 15.4 VOL % COAL CHAR / AMOCAT-1A CATALYST
BUBBLE RISE VELOCITY CORRELATION

Legend
 Δ RUN 224

FIGURE 87

BHATIA EPSTEIN MODEL, RUN221
KEROSENE / 4 VOL % COAL CHAR / HDS-2A CATALYST
WAKE VOLUME RATIO CORRELATION

FIGURE 88

BHATIA EPSTEIN MODEL, RUN 222
KEROSENE / 9.8 VOL % COAL CHAR / HDS-2A CATALYST
WAKE VOLUME RATIO CORRELATION

FIGURE 89

BHATIA EPSTEIN MODEL, RUN 223
KEROSENE / 20.7 VOL % COAL CHAR / HDS-2A CATALYST
WAKE VOLUME RATIO CORRELATION

FIGURE 90

BHATIA EPSTEIN MODEL, RUN 224
KEROSENE / 15.4 VOL % COAL CHAR / AMOCAT-1 A CATALYST
WAKE VOLUME RATIO CORRELATION

FIGURE 91

**COMPARISON OF BHATIA EPSTEIN BUBBLE RISE VELOCITIES
COLD FLOW RUN 224 VS HRI PDU-10 TESTS**

FIGURE 92

**BHATIA EPSTEIN MODEL
COMPARISON OF BUBBLE RISE VELOCITIES
PDU-10 VS COLD FLOW RUN 224**

FIGURE 93

**BHATIA EPSTEIN MODEL
COMPARISON OF WAKE VOLUME RATIO CORRELATION
PDU-10 VS COLD FLOW RUN 224**

APPENDIX A

**CATALYST BED EXPANSIONS
AND PHASE HOLDUPS FOR RUNS 218-224**

% BED EXPANSION FOR RUN 218

CATALYST : HDS-2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 0.0 VOL %
 COAL CHAR CONC: 0.0 WT %
 TEMPERATURE : 76. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
218- 1	0.072	0.0	56.	12.
- 2	0.090	0.0	60.	20.
- 3	0.106	0.0	63.	26.
- 4	0.132	0.0	72.	44.
- 5	0.156	0.0	80.	60.
- 6	0.181	0.0	90.	80.
- 7	0.092	0.048	65.	30.
- 8	0.091	0.099	70.	40.
- 9	0.091	0.132	72.	44.
-10	0.091	0.160	72.	44.
-11	0.091	0.193	72.	44.
-12	0.107	0.056	67.	34.
-13	0.107	0.100	75.	50.
-14	0.106	0.143	80.	60.
-15	0.106	0.200	82.	64.
-16	0.158	0.047	84.	68.
-17	0.157	0.108	92.	84.
-18	0.159	0.164	102.	104.
-19	0.180	0.048	97.	94.
-20	0.161	0.223	109.	122.
-21	0.181	0.113	102.	108.
-22	0.180	0.169	111.	127.
-23	0.212	0.051	112.	129.
-24	0.208	0.119	116.	137.

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 4.0 VOL %
 COAL CHAR CONC: 7.1 WT %
 TEMPERATURE : 83. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
221-18	0.053	0.0	47.	2.
-19	0.079	0.0	50.	9.
-20	0.103	0.0	56.	22.
-21	0.128	0.0	65.	41.
-22	0.156	0.0	73.	59.
-23	0.184	0.0	86.	87.
-24	0.210	0.0	96.	109.
-25	0.052	0.049	48.	4.
-26	0.055	0.079	49.	7.
-27	0.053	0.103	47.	2.
-28	0.052	0.128	49.	7.
-29	0.080	0.047	55.	20.
-30	0.080	0.077	58.	26.
-31	0.080	0.104	59.	28.
-32	0.079	0.129	59.	28.
-33	0.106	0.048	66.	43.
-34	0.106	0.080	66.	43.
-35	0.106	0.108	66.	43.
-36	0.107	0.134	68.	48.
-37	0.131	0.050	70.	52.
-38	0.132	0.083	73.	59.
-39	0.134	0.111	76.	65.
-40	0.132	0.136	64.	39.
-41	0.161	0.053	81.	76.
-42	0.159	0.087	84.	83.
-43	0.158	0.116	87.	89.
-44	0.177	0.052	92.	100.
-45	0.181	0.086	97.	111.
-46	0.206	0.052	106.	130.
-47	0.201	0.077	106.	130.

% BED EXPANSION FOR RUN 221

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 4.0 VOL %
 COAL CHAR CONC: 7.1 WT %
 TEMPERATURE : 72. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
221-48	0.102	0.068	66.	43.
-49	0.102	0.106	67.	46.
-50	0.102	0.134	68.	48.
-51	0.131	0.135	81.	76.
-52	0.050	0.0	47.	2.
-53	0.176	0.0	84.	83.
-54	0.201	0.0	97.	111.

% BED EXPANSION FOR RUN 222

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 9.8 VOL %
 COAL CHAR CONC: 16.7 WT %
 TEMPERATURE : 72. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
222- 1	0.049	0.0	49.	17.
- 2	0.074	0.0	53.	26.
- 3	0.096	0.0	58.	38.
- 4	0.121	0.0	64.	52.
- 5	0.144	0.0	73.	74.
- 6	0.167	0.0	89.	112.
- 7	0.049	0.049	49.	17.
- 8	0.072	0.049	54.	29.
- 9	0.088	0.050	66.	57.
-10	0.122	0.051	73.	74.
-11	0.146	0.052	83.	98.
-12	0.171	0.054	97.	131.
-13	0.191	0.054	109.	160.
-14	0.194	0.0	104.	148.
-15	0.047	0.079	47.	12.
-16	0.074	0.077	58.	38.
-17	0.098	0.075	63.	50.
-18	0.122	0.078	76.	81.
-19	0.145	0.080	88.	110.
-20	0.170	0.081	99.	136.
-21	0.195	0.078	114.	171.
-22	0.049	0.107	45.	7.
-23	0.073	0.107	57.	36.
-24	0.097	0.108	63.	50.
-25	0.122	0.109	79.	88.
-26	0.145	0.102	92.	119.
-27	0.168	0.102	103.	145.
-28	0.188	0.102	113.	169.
-29	0.048	0.126	47.	12.
-30	0.073	0.128	58.	38.

% BED EXPANSION FOR RUN 222

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 9.8 VOL %
 COAL CHAR CONC: 16.7 WT %
 TEMPERATURE : 70. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
222-31	0.096	0.131	68.	62.
-32	0.121	0.134	79.	88.
-33	0.132	0.137	94.	124.
-34	0.049	0.149	49.	17.
-35	0.073	0.153	59.	40.
-36	0.098	0.157	69.	64.
-37	0.097	0.104	69.	64.
-38	0.050	0.046	49.	17.
-39	0.122	0.117	79.	88.
-40	0.120	0.155	74.	76.
-41	0.144	0.160	93.	121.
-42	0.171	0.166	110.	162.
-43	0.169	0.127	107.	155.
-44	0.049	0.178	47.	12.
-45	0.074	0.185	58.	38.
-46	0.099	0.186	65.	55.
-47	0.121	0.186	74.	76.
-48	0.146	0.179	93.	121.
-49	0.169	0.187	113.	169.
-50	0.049	0.218	45.	7.
-51	0.073	0.213	58.	38.
-52	0.098	0.224	64.	52.
-53	0.049	0.050	49.	17.
-54	0.075	0.178	56.	33.
-55	0.098	0.183	67.	60.
-56	0.116	0.217	66.	57.

% BED EXPANSION FOR RUN 223

-213

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 20.7 VOL %
 COAL CHAR CONC: 32.4 WT %
 TEMPERATURE : 81. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
223- 1	0.051	0.0	51.	21.
- 2	0.076	0.0	62.	48.
- 3	0.100	0.0	73.	74.
- 4	0.126	0.0	89.	112.
- 5	0.152	0.0	111.	164.
- 6	0.178	0.0	132.	214.
- 7	0.052	0.047	52.	24.
- 8	0.077	0.048	68.	62.
- 9	0.100	0.049	77.	83.
-10	0.126	0.050	97.	130.
-11	0.146	0.051	111.	164.
-12	0.049	0.078	52.	24.
-13	0.063	0.078	59.	40.
-14	0.087	0.079	71.	69.
-15	0.109	0.079	86.	105.
-16	0.133	0.079	102.	143.
-17	0.048	0.101	52.	24.
-18	0.064	0.102	58.	38.
-19	0.088	0.102	71.	69.
-20	0.126	0.108	104.	148.
-21	0.151	0.106	124.	195.
-22	0.051	0.128	51.	21.
-23	0.076	0.126	66.	57.
-24	0.100	0.130	78.	86.
-25	0.126	0.129	98.	133.
-26	0.151	0.128	122.	190.
-27	0.051	0.145	54.	29.
-28	0.076	0.151	66.	57.
-29	0.100	0.156	78.	86.
-30	0.126	0.160	94.	124.

% BED EXPANSION FOR RUN 223

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 20.7 VOL %
 COAL CHAR CONC: 32.4 WT %
 TEMPERATURE : 65. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
223-31	0.051	0.173	59.	40.
-32	0.076	0.183	68.	62.
-33	0.100	0.184	75.	79.
-34	0.126	0.185	88.	110.
-35	0.177	0.056	135.	221.
-36	0.052	0.205	54.	27.
-37	0.051	0.203	54.	29.
-38	0.076	0.210	66.	57.
-39	0.100	0.213	68.	62.
-40	0.100	0.0	74.	76.
-41	0.147	0.0	104.	148.
-42	0.100	0.111	79.	88.
-43	0.051	0.0	52.	23.
-44	0.051	0.053	54.	29.
-45	0.126	0.168	93.	121.
-53	0.147	0.158	107.	155.
-54	0.177	0.145	134.	219.
-55	0.177	0.104	124.	195.
-56	0.177	0.085	124.	195.
-57	0.203	0.0	145.	245.
-59	0.199	0.058	155.	269.
-60	0.199	0.072	158.	276.
-61	0.198	0.119	157.	274.
-62	0.198	0.131	157.	274.
-63	0.207	0.144	154.	267.
-64	0.198	0.087	149.	255.
-99	0.459	0.072	139.	231.

% BED EXPANSION FOR RUN 224

CATALYST : AMOCAT1A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 15.4 VOL %
 COAL CHAR CONC: 25.0 WT %
 TEMPERATURE : 68. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
224- 1	0.049	0.0	52.	16.
- 2	0.074	0.0	63.	40.
- 3	0.079	0.0	68.	51.
- 4	0.094	0.0	72.	60.
- 5	0.112	0.0	83.	84.
- 6	0.112	0.057	86.	91.
- 7	0.093	0.046	77.	71.
- 8	0.083	0.040	71.	58.
- 9	0.073	0.041	63.	40.
-10	0.047	0.043	48.	7.
-11	0.048	0.070	55.	22.
-12	0.072	0.069	62.	38.
-13	0.083	0.068	68.	51.
-14	0.094	0.067	78.	73.
-15	0.110	0.067	87.	93.
-16	0.045	0.090	50.	11.
-17	0.074	0.090	64.	42.
-18	0.083	0.090	67.	49.
-19	0.093	0.090	77.	71.
-20	0.112	0.089	87.	93.
-21	0.113	0.102	90.	100.
-22	0.094	0.101	77.	71.
-23	0.083	0.100	70.	56.
-24	0.073	0.100	62.	38.
-25	0.046	0.100	50.	11.
-26	0.046	0.148	48.	7.
-27	0.073	0.151	66.	47.
-28	0.083	0.152	66.	47.
-29	0.141	0.0	102.	127.
-30	0.173	0.0	130.	189.

% BED EXPANSION FOR RUN 224

CATALYST : AMOCAT1A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 15.4 VOL %
 COAL CHAR CONC: 25.0 WT %
 TEMPERATURE : 69. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
224-32	0.093	0.150	70.	56.
-33	0.112	0.152	76.	69.
-34	0.140	0.151	100.	122.
-35	0.140	0.096	112.	149.
-36	0.140	0.084	112.	149.
-37	0.143	0.067	110.	144.
-38	0.140	0.039	107.	138.

CATALYST : HDS-2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 0.0 VOL %
 COAL CHAR CONC: 0.0 WT %
 TEMPERATURE : 76. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EGB	Vcd (Mm/Sec)
218- 1	0.072	0.0	0.483	0.518	0.0	0.0	0.0
- 2	0.090	0.0	0.451	0.544	0.0	0.0	0.0
- 3	0.106	0.0	0.429	0.577	0.0	0.0	0.0
- 4	0.132	0.0	0.376	0.613	0.0	0.0	0.0
- 5	0.156	0.0	0.338	0.681	0.0	0.0	0.0
- 6	0.181	0.0	0.301	0.707	0.0	0.0	0.0
- 7	0.092	0.048	0.416	0.498	0.0	0.086	8.8
- 8	0.091	0.099	0.387	0.454	0.0	0.160	17.0
- 9	0.091	0.132	0.376	0.426	0.0	0.198	21.9
-10	0.091	0.160	0.376	0.413	0.0	0.211	27.2
-11	0.091	0.193	0.376	0.385	0.0	0.239	31.7
-12	0.107	0.056	0.404	0.502	0.0	0.094	9.9
-13	0.107	0.100	0.361	0.486	0.0	0.154	17.0
-14	0.106	0.143	0.338	0.451	0.0	0.211	22.5
-15	0.106	0.200	0.330	0.423	0.0	0.247	31.7
-16	0.158	0.047	0.322	0.589	0.0	0.089	6.4
-17	0.157	0.108	0.294	0.558	0.0	0.148	17.3
-18	0.159	0.164	0.265	0.514	0.0	0.221	22.8
-19	0.180	0.048	0.279	0.641	0.0	0.080	7.3
-20	0.161	0.223	0.243	0.487	0.0	0.270	29.8
-21	0.181	0.113	0.260	0.617	0.0	0.123	20.5
-22	0.180	0.169	0.239	0.556	0.0	0.205	24.8
-23	0.212	0.051	0.237	0.713	0.0	0.051	10.3
-24	0.208	0.119	0.229	0.642	0.0	0.129	20.4

CALCULATED HOLDUPS, RUN 221: DENSE PHASE

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 4.0 VOL %
 COAL CHAR CONC: 7.1 WT %
 TEMPERATURE : 83. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec.	ECB	ELGB	Efgb	EGB	Vcd (Mm/Sec)
221-18	0.053	0.0	0.539	0.436	0.018	0.0	0.0
-19	0.079	0.0	0.507	0.459	0.019	0.0	0.0
-20	0.103	0.0	0.452	0.517	0.022	0.0	0.0
-21	0.128	0.0	0.390	0.563	0.023	0.0	0.0
-22	0.156	0.0	0.317	0.619	0.026	0.0	0.0
-23	0.184	0.0	0.255	0.668	0.028	0.0	0.0
-24	0.210	0.0	0.264	0.691	0.029	0.0	0.0
-25	0.052	0.049	0.528	0.366	0.015	0.091	10.2
-26	0.055	0.079	0.517	0.371	0.015	0.096	18.1
-27	0.053	0.103	0.539	0.348	0.014	0.098	24.4
-28	0.052	0.128	0.517	0.368	0.015	0.099	31.4
-29	0.080	0.047	0.461	0.449	0.019	0.072	9.8
-30	0.080	0.077	0.437	0.425	0.018	0.120	15.0
-31	0.080	0.104	0.429	0.408	0.017	0.145	20.0
-32	0.079	0.129	0.429	0.404	0.017	0.150	26.2
-33	0.106	0.048	0.384	0.547	0.023	0.047	11.5
-34	0.106	0.080	0.384	0.494	0.021	0.102	16.2
-35	0.106	0.108	0.384	0.449	0.019	0.149	19.4
-36	0.107	0.134	0.373	0.443	0.018	0.166	24.4
-37	0.131	0.050	0.362	0.558	0.023	0.056	10.6
-38	0.132	0.083	0.347	0.524	0.022	0.107	15.6
-39	0.134	0.111	0.333	0.503	0.021	0.143	19.6
-40	0.132	0.136	0.395	0.390	0.016	0.199	17.5
-41	0.161	0.053	0.313	0.606	0.025	0.056	11.1
-42	0.159	0.087	0.302	0.578	0.024	0.096	16.8
-43	0.158	0.116	0.291	0.561	0.023	0.125	22.0
-44	0.177	0.052	0.275	0.645	0.027	0.052	11.0
-45	0.181	0.086	0.261	0.627	0.026	0.085	17.3
-46	0.206	0.052	0.239	0.670	0.028	0.063	9.5
-47	0.201	0.077	0.239	0.642	0.027	0.092	13.7

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 4.0 VOL %
 COAL CHAR CONC: 7.1 WT %
 TEMPERATURE : 72. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EOB	Vcd (Mm/Sec)
221-48	0.102	0.068	0.384	0.514	0.021	0.081	14.8
-49	0.102	0.106	0.378	0.463	0.019	0.139	20.1
-50	0.102	0.134	0.373	0.450	0.019	0.158	25.7
-51	0.131	0.135	0.313	0.504	0.021	0.162	24.0
-52	0.050	0.0	0.539	0.435	0.018	0.0	0.0
-53	0.176	0.0	0.302	0.643	0.027	0.0	0.0
-54	0.201	0.0	0.261	0.690	0.029	0.0	0.0

CALCULATED HOLDUPS, RUN 222: DENSE PHASE

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 9.8 VOL %
 COAL CHAR CONC: 16.7 WT %
 TEMPERATURE : 72. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EGB	Vcd (Mm/Sec)
222- 1	0.049	0.0	0.472	0.475	0.052	0.0	0.0
- 2	0.074	0.0	0.436	0.495	0.054	0.0	0.0
- 3	0.096	0.0	0.399	0.543	0.059	0.0	0.0
- 4	0.121	0.0	0.361	0.607	0.066	0.0	0.0
- 5	0.144	0.0	0.317	0.622	0.068	0.0	0.0
- 6	0.167	0.0	0.260	0.646	0.071	0.0	0.0
- 7	0.049	0.049	0.472	0.436	0.048	0.045	12.9
- 8	0.072	0.049	0.428	0.450	0.049	0.073	10.8
- 9	0.088	0.050	0.350	0.527	0.058	0.065	11.5
-10	0.122	0.051	0.317	0.553	0.060	0.069	10.5
-11	0.146	0.052	0.279	0.588	0.064	0.069	10.4
-12	0.171	0.054	0.238	0.617	0.067	0.077	9.6
-13	0.191	0.054	0.212	0.642	0.070	0.076	9.6
-14	0.194	0.0	0.222	0.710	0.078	0.0	0.0
-15	0.047	0.079	0.492	0.398	0.043	0.067	20.4
-16	0.074	0.077	0.399	0.462	0.050	0.089	17.8
-17	0.098	0.075	0.367	0.464	0.051	0.118	14.2
-18	0.122	0.078	0.304	0.517	0.057	0.122	13.8
-19	0.145	0.080	0.263	0.564	0.062	0.112	14.6
-20	0.170	0.081	0.234	0.608	0.066	0.092	16.0
-21	0.195	0.078	0.203	0.628	0.069	0.100	13.7
-22	0.049	0.107	0.514	0.348	0.038	0.101	25.9
-23	0.073	0.107	0.406	0.440	0.048	0.106	24.8
-24	0.097	0.108	0.367	0.433	0.047	0.133	20.0
-25	0.122	0.109	0.293	0.513	0.056	0.138	21.0
-26	0.145	0.102	0.251	0.547	0.060	0.141	18.0
-27	0.168	0.102	0.225	0.579	0.063	0.133	17.8
-28	0.188	0.102	0.205	0.605	0.066	0.124	18.0
-29	0.048	0.126	0.492	0.367	0.040	0.100	31.4
-30	0.073	0.128	0.399	0.447	0.049	0.105	30.6

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 9.8 VOL %
 COAL CHAR CONC: 16.7 WT %
 TEMPERATURE : 70. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EOB	Vcd (Mm/Sec)
222-31	0.096	0.131	0.340	0.458	0.050	0.152	26.3
-32	0.121	0.134	0.293	0.478	0.052	0.177	23.5
-33	0.132	0.137	0.246	0.539	0.059	0.156	26.4
-34	0.049	0.149	0.472	0.417	0.046	0.065	40.6
-35	0.073	0.153	0.392	0.449	0.049	0.110	37.2
-36	0.098	0.157	0.335	0.467	0.051	0.147	33.6
-37	0.097	0.104	0.335	0.480	0.052	0.132	21.2
-38	0.050	0.046	0.472	0.429	0.047	0.052	11.7
-39	0.122	0.117	0.293	0.490	0.054	0.163	20.5
-40	0.120	0.155	0.313	0.457	0.050	0.180	28.2
-41	0.144	0.160	0.249	0.508	0.056	0.188	27.8
-42	0.171	0.166	0.210	0.546	0.060	0.184	28.5
-43	0.169	0.127	0.216	0.542	0.059	0.183	18.9
-44	0.049	0.178	0.492	0.359	0.039	0.110	44.7
-45	0.074	0.185	0.399	0.433	0.047	0.121	44.7
-46	0.099	0.186	0.356	0.451	0.049	0.144	41.2
-47	0.121	0.186	0.313	0.459	0.050	0.179	36.0
-48	0.146	0.179	0.249	0.500	0.055	0.196	31.3
-49	0.169	0.187	0.205	0.541	0.059	0.195	32.3
-50	0.049	0.218	0.514	0.318	0.035	0.134	52.7
-51	0.073	0.213	0.399	0.428	0.047	0.127	51.6
-52	0.098	0.224	0.361	0.444	0.049	0.146	50.7
-53	0.049	0.050	0.472	0.434	0.047	0.047	13.0
-54	0.075	0.178	0.413	0.427	0.047	0.114	43.1
-55	0.098	0.183	0.345	0.466	0.051	0.137	41.3
-56	0.116	0.217	0.350	0.414	0.045	0.191	41.7

CALCULATED HOLDUPS, RUN 223: DENSE PHASE

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 20.7 VOL %
 COAL CHAR CONC: 32.4 WT %
 TEMPERATURE : 81. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EGB	Vcd (Mm/Sec)
223- 1	0.051	0.0	0.453	0.439	0.115	0.0	0.0
- 2	0.076	0.0	0.373	0.504	0.132	0.0	0.0
- 3	0.100	0.0	0.317	0.548	0.143	0.0	0.0
- 4	0.126	0.0	0.260	0.586	0.153	0.0	0.0
- 5	0.152	0.0	0.208	0.632	0.165	0.0	0.0
- 6	0.178	0.0	0.175	0.659	0.172	0.0	0.0
- 7	0.052	0.047	0.445	0.393	0.103	0.060	11.8
- 8	0.077	0.048	0.340	0.461	0.120	0.079	10.5
- 9	0.100	0.049	0.300	0.487	0.127	0.086	9.7
-10	0.126	0.050	0.240	0.544	0.142	0.074	10.3
-11	0.146	0.051	0.208	0.565	0.148	0.080	9.8
-12	0.049	0.078	0.445	0.412	0.108	0.036	21.9
-13	0.063	0.078	0.392	0.424	0.111	0.073	19.6
-14	0.087	0.079	0.326	0.448	0.117	0.109	16.8
-15	0.109	0.079	0.269	0.498	0.130	0.103	16.8
-16	0.133	0.079	0.227	0.523	0.137	0.113	15.2
-17	0.048	0.101	0.445	0.385	0.101	0.069	26.7
-18	0.064	0.102	0.399	0.416	0.109	0.077	26.1
-19	0.088	0.102	0.326	0.454	0.119	0.102	23.7
-20	0.126	0.108	0.222	0.502	0.131	0.144	20.6
-21	0.151	0.106	0.187	0.530	0.138	0.146	19.1
-22	0.051	0.128	0.453	0.372	0.097	0.078	33.7
-23	0.076	0.126	0.350	0.440	0.115	0.094	31.2
-24	0.100	0.130	0.297	0.471	0.123	0.110	30.2
-25	0.126	0.129	0.236	0.495	0.129	0.140	26.3
-26	0.151	0.128	0.190	0.515	0.135	0.160	23.1
-27	0.051	0.145	0.428	0.381	0.099	0.091	37.5
-28	0.076	0.151	0.350	0.442	0.115	0.092	38.3
-29	0.100	0.156	0.297	0.478	0.125	0.101	38.3
-30	0.126	0.160	0.246	0.487	0.127	0.139	34.5

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 20.7 VOL %
 COAL CHAR CONC: 32.4 WT %
 TEMPERATURE : 65. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EGB	Vcd (Mm/Sec)
223-31	0.051	0.173	0.392	0.412	0.108	0.088	45.6
-32	0.076	0.183	0.340	0.453	0.118	0.089	47.5
-33	0.100	0.184	0.308	0.453	0.118	0.120	43.6
-34	0.126	0.185	0.263	0.471	0.123	0.144	40.4
-35	0.177	0.056	0.171	0.596	0.156	0.077	10.7
-36	0.052	0.205	0.432	0.353	0.092	0.123	50.9
-37	0.051	0.203	0.428	0.358	0.093	0.120	50.8
-38	0.076	0.210	0.350	0.430	0.112	0.107	53.0
-39	0.100	0.213	0.340	0.423	0.111	0.126	50.5
-40	0.100	0.0	0.313	0.552	0.144	0.0	0.0
-41	0.147	0.0	0.222	0.630	0.165	0.0	0.0
-42	0.100	0.111	0.293	0.476	0.124	0.107	25.4
-43	0.051	0.0	0.449	0.390	0.102	0.0	0.0
-44	0.051	0.053	0.428	0.403	0.105	0.064	13.2
-45	0.126	0.168	0.249	0.482	0.126	0.144	36.0
-53	0.147	0.158	0.216	0.484	0.127	0.173	29.2
-54	0.177	0.145	0.173	0.522	0.136	0.170	25.1
-55	0.177	0.104	0.187	0.537	0.140	0.137	18.0
-56	0.177	0.085	0.187	0.556	0.145	0.112	15.3
-57	0.203	0.0	0.160	0.689	0.180	0.0	0.0
-59	0.199	0.058	0.149	0.616	0.161	0.074	10.9
-60	0.199	0.072	0.146	0.599	0.156	0.098	12.6
-61	0.198	0.119	0.147	0.562	0.147	0.144	20.6
-62	0.198	0.131	0.147	0.556	0.145	0.152	22.8
-63	0.207	0.144	0.150	0.630	0.099	0.121	29.5
-64	0.198	0.087	0.155	0.582	0.152	0.111	15.5
-99	0.459	0.072	0.166	0.409	0.065	0.360	-36.3

CALCULATED HOLDUPS, RUN 224: DENSE PHASE

CATALYST : AMOCAT1A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 15.4 VOL %
 COAL CHAR CONC: 25.0 WT %
 TEMPERATURE : 68. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EGB	Vcd (Mm/Sec)
224- 1	0.049	0.0	0.501	0.414	0.075	0.0	0.0
- 2	0.074	0.0	0.414	0.511	0.093	0.0	0.0
- 3	0.079	0.0	0.383	0.529	0.096	0.0	0.0
- 4	0.094	0.0	0.362	0.541	0.098	0.0	0.0
- 5	0.112	0.0	0.314	0.580	0.105	0.0	0.0
- 6	0.112	0.057	0.303	0.525	0.095	0.076	12.1
- 7	0.093	0.046	0.338	0.506	0.092	0.064	10.4
- 8	0.083	0.040	0.367	0.491	0.089	0.053	9.5
- 9	0.073	0.041	0.414	0.450	0.082	0.055	9.8
-10	0.047	0.043	0.543	0.340	0.062	0.056	10.6
-11	0.048	0.070	0.474	0.418	0.076	0.033	19.8
-12	0.072	0.069	0.420	0.409	0.074	0.097	15.0
-13	0.083	0.068	0.383	0.452	0.082	0.082	15.5
-14	0.094	0.067	0.334	0.494	0.090	0.082	15.1
-15	0.110	0.067	0.300	0.515	0.093	0.092	14.0
-16	0.045	0.090	0.521	0.350	0.064	0.065	23.5
-17	0.074	0.090	0.407	0.424	0.077	0.092	21.1
-18	0.083	0.090	0.389	0.439	0.080	0.092	20.8
-19	0.093	0.090	0.338	0.469	0.085	0.108	19.5
-20	0.112	0.089	0.300	0.493	0.089	0.118	17.9
-21	0.113	0.102	0.290	0.495	0.090	0.126	20.9
-22	0.094	0.101	0.338	0.474	0.086	0.101	22.9
-23	0.083	0.100	0.372	0.459	0.083	0.086	24.3
-24	0.073	0.100	0.420	0.419	0.076	0.084	24.6
-25	0.046	0.100	0.521	0.344	0.062	0.072	26.0
-26	0.046	0.148	0.543	0.321	0.058	0.078	38.9
-27	0.073	0.151	0.395	0.425	0.077	0.103	37.3
-28	0.083	0.152	0.395	0.419	0.076	0.111	36.1
-29	0.141	0.0	0.256	0.630	0.114	0.0	0.0
-30	0.173	0.0	0.200	0.675	0.122	0.0	0.0

CATALYST : AMOCAT1A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 15.4 VOL %
COAL CHAR CONC: 25.0 WT %
TEMPERATURE : 69. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	Efgb	EOB	Vcd (Mm/Sec)
224-32	0.093	0.150	0.372	0.430	0.078	0.119	34.5
-33	0.112	0.152	0.343	0.428	0.078	0.151	30.6
-34	0.140	0.151	0.261	0.485	0.088	0.167	28.0
-35	0.140	0.096	0.233	0.532	0.097	0.139	17.0
-36	0.140	0.084	0.233	0.547	0.099	0.121	15.6
-37	0.143	0.067	0.237	0.560	0.102	0.101	12.3
-38	0.140	0.039	0.244	0.588	0.107	0.062	7.5

CALCULATED HOLDUPS, RUN 218--DILUTE PHASE

CATALYST : HDS-2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 0.0 VOL %
 COAL CHAR CONC: 0.0 WT %
 TEMPERATURE : 76. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
218- 1	0.072	0.0	0.988	0.0	0.0
- 2	0.090	0.0	0.989	0.0	0.0
- 3	0.106	0.0	0.978	0.0	0.0
- 4	0.132	0.0	0.988	0.0	0.0
- 5	0.156	0.0	0.993	0.0	0.0
- 6	0.181	0.0	0.989	0.0	0.0
- 7	0.092	0.048	0.912	0.0	0.088
- 8	0.091	0.099	0.824	0.0	0.176
- 9	0.091	0.132	0.762	0.0	0.238
-10	0.091	0.160	0.753	0.0	0.247
-11	0.091	0.193	0.730	0.0	0.270
-12	0.107	0.056	0.896	0.0	0.104
-13	0.107	0.100	0.820	0.0	0.180
-14	0.106	0.143	0.751	0.0	0.249
-15	0.106	0.200	0.710	0.0	0.290
-16	0.158	0.047	0.898	0.0	0.102
-17	0.157	0.108	0.809	0.0	0.191
-18	0.159	0.164	0.750	0.0	0.250
-19	0.180	0.048	0.910	0.0	0.090
-20	0.161	0.223	0.696	0.0	0.304
-21	0.181	0.113	0.832	0.0	0.168
-22	0.180	0.169	0.765	0.0	0.235
-23	0.212	0.051	0.914	0.0	0.086
-24	0.208	0.119	0.838	0.0	0.162

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 4.0 VOL %
 COAL CHAR CONC: 7.1 WT %
 TEMPERATURE : 83. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
221-18	0.053	0.0	0.932	0.039	0.0
-19	0.079	0.0	0.929	0.039	0.0
-20	0.103	0.0	0.928	0.039	0.0
-21	0.128	0.0	0.936	0.039	0.0
-22	0.156	0.0	0.934	0.039	0.0
-23	0.184	0.0	0.934	0.039	0.0
-24	0.210	0.0	0.933	0.039	0.0
-25	0.052	0.049	0.868	0.036	0.095
-26	0.055	0.079	0.822	0.034	0.143
-27	0.053	0.103	0.794	0.033	0.173
-28	0.052	0.128	0.768	0.032	0.200
-29	0.080	0.047	0.857	0.036	0.108
-30	0.080	0.077	0.813	0.034	0.153
-31	0.080	0.104	0.774	0.032	0.193
-32	0.079	0.129	0.754	0.031	0.214
-33	0.106	0.048	0.851	0.035	0.114
-34	0.106	0.080	0.803	0.033	0.163
-35	0.106	0.108	0.766	0.032	0.202
-36	0.107	0.134	0.730	0.030	0.239
-37	0.131	0.050	0.844	0.035	0.121
-38	0.132	0.083	0.796	0.033	0.171
-39	0.134	0.111	0.759	0.032	0.209
-40	0.132	0.136	0.723	0.030	0.247
-41	0.161	0.053	0.856	0.036	0.108
-42	0.159	0.087	0.801	0.033	0.165
-43	0.158	0.116	0.762	0.032	0.206
-44	0.177	0.052	0.863	0.036	0.101
-45	0.181	0.086	0.814	0.034	0.152
-46	0.206	0.052	0.870	0.036	0.094
-47	0.201	0.077	0.837	0.035	0.128

-224D

CALCULATED HOLDUPS, RUN 221--DILUTE PHASE

CATALYST : HDS2A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 4.0 VOL %
COAL CHAR CONC: 7.1 WT %
TEMPERATURE : 72. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
221-48	0.102	0.068	0.820	0.034	0.146
-49	0.102	0.106	0.767	0.032	0.201
-50	0.102	0.134	0.738	0.031	0.232
-51	0.131	0.135	0.732	0.031	0.237
-52	0.050	0.0	0.940	0.039	0.0
-53	0.176	0.0	0.935	0.039	0.0
-54	0.201	0.0	0.939	0.039	0.0

CALCULATED HOLDUPS, RUN 222--DILUTE PHASE

-224E

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 9.8 VOL %
 COAL CHAR CONC: 16.7 WT %
 TEMPERATURE : 72. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
222- 1	0.049	0.0	0.866	0.095	0.0
- 2	0.074	0.0	0.868	0.095	0.0
- 3	0.096	0.0	0.872	0.095	0.0
- 4	0.121	0.0	0.864	0.094	0.0
- 5	0.144	0.0	0.870	0.095	0.0
- 6	0.167	0.0	0.866	0.095	0.0
- 7	0.049	0.049	0.809	0.088	0.103
- 8	0.072	0.049	0.807	0.088	0.105
- 9	0.088	0.050	0.791	0.086	0.123
-10	0.122	0.051	0.786	0.086	0.128
-11	0.146	0.052	0.790	0.086	0.123
-12	0.171	0.054	0.798	0.087	0.115
-13	0.191	0.054	0.805	0.088	0.107
-14	0.194	0.0	0.867	0.095	0.0
-15	0.047	0.079	0.775	0.085	0.141
-16	0.074	0.077	0.766	0.084	0.150
-17	0.098	0.075	0.752	0.082	0.166
-18	0.122	0.078	0.747	0.082	0.172
-19	0.145	0.080	0.749	0.082	0.170
-20	0.170	0.081	0.756	0.083	0.162
-21	0.195	0.078	0.766	0.084	0.150
-22	0.049	0.107	0.747	0.082	0.171
-23	0.073	0.107	0.739	0.081	0.180
-24	0.097	0.108	0.723	0.079	0.198
-25	0.122	0.109	0.707	0.077	0.215
-26	0.145	0.102	0.722	0.079	0.200
-27	0.168	0.102	0.726	0.079	0.195
-28	0.188	0.102	0.743	0.081	0.176
-29	0.048	0.126	0.719	0.079	0.202
-30	0.073	0.128	0.726	0.079	0.195

CALCULATED HOLDUPS, RUN 222--DILUTE PHASE

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 9.8 VOL %
 COAL CHAR CONC: 16.7 WT %
 TEMPERATURE : 70. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
222-31	0.096	0.131	0.713	0.078	0.209
-32	0.121	0.134	0.687	0.075	0.238
-33	0.132	0.137	0.676	0.074	0.250
-34	0.049	0.149	0.719	0.079	0.202
-35	0.073	0.153	0.714	0.078	0.208
-36	0.098	0.157	0.703	0.077	0.220
-37	0.097	0.104	0.723	0.079	0.198
-38	0.050	0.046	0.805	0.088	0.107
-39	0.122	0.117	0.689	0.075	0.236
-40	0.120	0.155	0.691	0.074	0.245
-41	0.144	0.160	0.648	0.071	0.281
-42	0.171	0.166	0.655	0.072	0.273
-43	0.169	0.127	0.693	0.076	0.232
-44	0.049	0.178	0.697	0.076	0.227
-45	0.074	0.185	0.692	0.076	0.232
-46	0.099	0.186	0.687	0.075	0.238
-47	0.121	0.186	0.674	0.074	0.253
-48	0.146	0.179	0.652	0.071	0.277
-49	0.169	0.187	0.645	0.070	0.285
-50	0.049	0.218	0.681	0.074	0.245
-51	0.073	0.213	0.688	0.075	0.236
-52	0.098	0.224	0.679	0.074	0.247
-53	0.049	0.050	0.808	0.088	0.104
-54	0.075	0.178	0.713	0.078	0.210
-55	0.098	0.183	0.701	0.077	0.223
-56	0.116	0.217	0.672	0.073	0.255

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 20.7 VOL %
 COAL CHAR CONC: 32.4 WT %
 TEMPERATURE : 81. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
223- 1	0.051	0.0	0.766	0.200	0.0
- 2	0.076	0.0	0.765	0.200	0.0
- 3	0.100	0.0	0.770	0.201	0.0
- 4	0.126	0.0	0.766	0.200	0.0
- 5	0.152	0.0	0.770	0.201	0.0
- 6	0.178	0.0	0.766	0.200	0.0
- 7	0.052	0.047	0.718	0.188	0.094
- 8	0.077	0.048	0.711	0.186	0.103
- 9	0.100	0.049	0.700	0.183	0.117
-10	0.126	0.050	0.703	0.184	0.113
-11	0.146	0.051	0.701	0.183	0.116
-12	0.049	0.078	0.699	0.183	0.119
-13	0.063	0.078	0.693	0.181	0.127
-14	0.087	0.079	0.690	0.180	0.130
-15	0.109	0.079	0.674	0.176	0.149
-16	0.133	0.079	0.660	0.172	0.168
-17	0.048	0.101	0.685	0.179	0.136
-18	0.064	0.102	0.683	0.178	0.139
-19	0.088	0.102	0.677	0.177	0.147
-20	0.126	0.108	0.650	0.170	0.180
-21	0.151	0.106	0.644	0.168	0.187
-22	0.051	0.128	0.674	0.176	0.150
-23	0.076	0.126	0.675	0.176	0.148
-24	0.100	0.130	0.668	0.175	0.157
-25	0.126	0.129	0.659	0.172	0.168
-26	0.151	0.128	0.635	0.166	0.199
-27	0.051	0.145	0.662	0.173	0.165
-28	0.076	0.151	0.662	0.173	0.165
-29	0.100	0.156	0.662	0.173	0.165
-30	0.126	0.160	0.659	0.172	0.169

CALCULATED HOLDUPS, RUN 223--DILUTE PHASE

CATALYST : HDS2A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 20.7 VOL %
 COAL CHAR CONC: 32.4 WT %
 TEMPERATURE : 65. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
223-31	0.051	0.173	0.653	0.171	0.176
-32	0.076	0.183	0.656	0.171	0.172
-33	0.100	0.184	0.654	0.171	0.175
-34	0.126	0.185	0.649	0.170	0.181
-35	0.177	0.056	0.707	0.185	0.108
-36	0.052	0.205	0.632	0.165	0.202
-37	0.051	0.203	0.637	0.166	0.197
-38	0.076	0.210	0.643	0.168	0.189
-39	0.100	0.213	0.635	0.166	0.199
-40	0.100	0.0	0.770	0.201	0.0
-41	0.147	0.0	0.760	0.199	0.0
-42	0.100	0.111	0.667	0.174	0.159
-43	0.051	0.0	0.766	0.200	0.0
-44	0.051	0.053	0.722	0.189	0.090
-45	0.126	0.168	0.645	0.169	0.186
-53	0.147	0.158	0.634	0.166	0.201
-54	0.177	0.145	0.622	0.163	0.215
-55	0.177	0.104	0.660	0.172	0.168
-56	0.177	0.085	0.678	0.177	0.144
-57	0.203	0.0	0.771	0.201	0.0
-59	0.199	0.058	0.707	0.185	0.108
-60	0.199	0.072	0.688	0.180	0.133
-61	0.198	0.119	0.647	0.169	0.184
-62	0.198	0.131	0.653	0.171	0.176
-63	0.207	0.144	0.731	0.115	0.154
-64	0.198	0.087	0.679	0.178	0.143
-99	0.459	0.072	0.004	0.001	0.995

CATALYST : AMOCAT1A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 15.4 VOL %
 COAL CHAR CONC: 25.0 WT %
 TEMPERATURE : 68. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
224- 1	0.049	0.0	0.844	0.153	0.0
- 2	0.074	0.0	0.853	0.155	0.0
- 3	0.079	0.0	0.852	0.155	0.0
- 4	0.094	0.0	0.853	0.155	0.0
- 5	0.112	0.0	0.855	0.155	0.0
- 6	0.112	0.057	0.789	0.143	0.068
- 7	0.093	0.046	0.794	0.144	0.062
- 8	0.083	0.040	0.800	0.145	0.055
- 9	0.073	0.041	0.803	0.146	0.051
-10	0.047	0.043	0.818	0.148	0.034
-11	0.048	0.070	0.789	0.143	0.068
-12	0.072	0.069	0.795	0.144	0.061
-13	0.083	0.068	0.780	0.141	0.079
-14	0.094	0.067	0.778	0.141	0.081
-15	0.110	0.067	0.765	0.139	0.096
-16	0.045	0.090	0.770	0.140	0.090
-17	0.074	0.090	0.772	0.140	0.088
-18	0.083	0.090	0.759	0.138	0.103
-19	0.093	0.090	0.746	0.135	0.118
-20	0.112	0.089	0.733	0.133	0.134
-21	0.113	0.102	0.740	0.134	0.125
-22	0.094	0.101	0.764	0.138	0.098
-23	0.083	0.100	0.771	0.140	0.089
-24	0.073	0.100	0.774	0.140	0.086
-25	0.046	0.100	0.769	0.139	0.092
-26	0.046	0.148	0.737	0.134	0.130
-27	0.073	0.151	0.734	0.133	0.132
-28	0.083	0.152	0.733	0.133	0.134
-29	0.141	0.0	0.858	0.156	0.0
-30	0.173	0.0	0.861	0.156	0.0

CALCULATED HOLDUPS, RUN 224--DILUTE PHASE

CATALYST : AMOCAT1A
 GAS : NITROGEN
 LIQUID : KEROSENE
 COAL CHAR CONC: 15.4 VOL %
 COAL CHAR CONC: 25.0 WT %
 TEMPERATURE : 69. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow rate, Ft/Sec	ELG	Efg	EGG
224-32	0.093	0.150	0.738	0.134	0.128
-33	0.112	0.152	0.733	0.133	0.134
34	0.140	0.151	0.720	0.131	0.149
-35	0.140	0.096	0.706	0.128	0.166
-36	0.140	0.084	0.729	0.132	0.139
-37	0.143	0.067	0.757	0.137	0.106
-38	0.140	0.039	0.787	0.143	0.070

APPENDIX B

LIQUID DISPERSION DATA ANALYSIS

APPENDIX B

LIQUID MIXING DATA ANALYSIS PROGRAM

A modified method of moments involving approximation of the RTD curves by a log-normal distribution has been selected to process the liquid mixing data. This method starts with calculations of the total area under the RTD curve and the percentage of cumulated area as a function of time. These percentage-cumulated areas are then plotted against sampling time on logarithmic probability paper and fit by a straight line to smooth the raw, experimental data points. The Peclet number (or the dispersion coefficient) is calculated from the mean (the first moment) and the variance (the second moment) of the RTD curves according to an axial dispersion model.

Axial Dispersion Model

To calculate the liquid Peclet number and the dispersion coefficient, a dispersed plug flow axial mixing model is assumed. In dimensionless form, the basic differential equation representing this dispersion model is

$$\frac{\partial C}{\partial \theta} = \left(\frac{D_1}{U_z L} \right) \frac{\partial^2 C}{\partial Z^2} - \frac{\partial C}{\partial Z} \quad (B-1)$$

where C = Sulfur concentration, ppm.
 θ = Sampling time, $T \cdot U_z / L$, dimensionless.
 D_1 = Liquid dispersion coefficient, cm^2/sec .
 U_z = Average linear liquid velocity, cm/sec .
 L = Distance between measuring points, cm .
 Z = Axial position, z/L , dimensionless.

Solutions to the Dispersion Model

If a perfect input pulse is assumed, the analytical solution to Equation 1 (Levenspiel and Smith, B-4) is

$$C = \frac{Q}{A \sqrt{4\pi\theta D_1/U_z L}} \exp \left[\frac{-(1-\theta)^2}{4\theta D_1/U_z L} \right] \quad (B-2)$$

where Q = Amount of tracer injected into the system.
 A = Cross-sectional area of the reactor.

D_1 can be estimated by least squares using Equation 2 and the concentration-time data.

In practice, it is impossible to introduce an ideal input pulse of the tracer into the system. In order to eliminate the necessity for a perfect input pulse, the technique of detecting tracer concentration at two downstream positions is employed.

As shown by many investigators (Aris [B-1]; Bischoff and Levenspiel [B-2]; Levenspiel and Smith [B-4]; Sater and Levenspiel [B-7]), the axial dispersion coefficient can be calculated from the first and second moments of the RTD curves at two positions. The first and second moments of the RTD curves are given by

$$\mu_i = \frac{\int_0^{\infty} C_i \cdot T \, dT}{\int_0^{\infty} C_i \, dT} \quad (B-3)$$

$$\sigma_{\theta i}^2 = \frac{\int_0^{\infty} C_i \cdot T^2 \, dT}{\int_0^{\infty} C_i \, dT} - \mu_i^2 \quad (B-4)$$

where $i = 1$ and 2 .
 T = Real time, sec.

Since the first moment of the RTD curve is the mean of that curve, the average residence time of the tracer can be calculated by taking the difference of the first moments of the RTD curves:

$$T_m = \mu_2 - \mu_1 \quad (B-5)$$

The second moments of the RTD curves are a measure of the spread of the RTD curves, and the difference of the second moments is a measure of the amount of axial mixing occurring between the two measuring points. This relationship has been derived analytically by Levenspiel and Smith (B-4):

$$(\Delta\sigma_{\theta})^2 = (\sigma_{\theta 2})^2 - (\sigma_{\theta 1})^2 = 2/Pe + 8 (1/Pe)^2 \quad (B-6)$$

Aris (B-1) has shown that for a one-shot injection of tracer with the RTD being measured at two downstream positions, the above equation can be approximated adequately by

$$(\Delta\sigma_{\theta})^2 = 2/Pe \quad (B-7)$$

In this method, however, the tail of the RTD curve can cause significant error in the calculation of Pe . Ostergaard and Michelsen (B-6) introduced a method involving the Laplace transform of the axial dispersion model and evaluation of a linear transfer function. The authors claim that this method gives more consistent results than the method of moments, regardless of the severity of the tailing and the terminal point of the tail.

The transfer function for a linear system is described by

$$F(s) = \frac{C_2(s)}{C_1(s)} = \frac{\int_0^{\infty} C_2(T) \exp(-sT) dt / \int_0^{\infty} C_2(T) dT}{\int_0^{\infty} C_1(T) \exp(-sT) dt / \int_0^{\infty} C_1(T) dT} \quad (B-8)$$

The axial dispersion model after the Laplace transformation is

$$F(s) = \exp \left\{ \frac{Pe}{2} \left[1 - \left(1 + \frac{4s T_m}{Pe} \right)^{1/2} \right] \right\} \quad (B-9)$$

After rearranging, the above equation becomes

$$\left[\log \left(\frac{1}{F(s)} \right) \right]^{-1} = T_m s \left[\log \left(\frac{1}{F(s)} \right) \right]^{-2} - \frac{1}{Pe} \quad (B-10)$$

A plot of $(1/F(s))^{-1}$ versus $S[\log(1/F(s))]^{-2}$ should yield a straight line. The Peclet number and the mean residence time can then be calculated from the intercept and slope, respectively.

This method was evaluated by processing several sets of liquid mixing data, and the results were unsatisfactory. One of the problems associated with using the Laplace transfer function for estimating dispersion parameters is the range of the Laplace operator, s . Hopkins, et al., (B-3) studied this effect and showed that for small values of s , the transfer function becomes insensitive to variation in the parameter $s \cdot T_m$. Therefore, small errors in the transfer function will greatly affect the resulting values of slope and intercept. For large values of s , the initial part of the tail is too heavily weighted to give accurate evaluation of the transfer function.

Selected Method of Solution

Due to unsatisfactory results produced by using the Laplace transfer function, a modified method of the moments was used to process the liquid mixing data. This modified method of moments, involving approximation of the RTD curves by a log-normal distribution, has been selected to process the liquid-phase mixing data. The method requires plotting the cumulative percentage (per cent total area) against the corresponding sampling time (t) on the logarithmic probability paper (Levenspiel and Smith [B-4]). Data smoothing, including determination of the cut-off point of the tail, was done by drawing a straight line which best fit the data points up to about 98% total area on the logarithmic probability paper. An example plot is shown in Figure B-1.

Because the cumulative percentage area is plotted against the corresponding sampling time on logarithmic probability paper, the following conversions are involved in obtaining the first and second moments on the arithmetic scale. The first moment about the origin of sampling time is

$$\mu = \exp [\ln T' + S^2/2] \quad (B-11)$$

where $S = \ln T'' - \ln T'$

$T' =$ Sampling time corresponding to log-normal distribution mean (at 50%).

$T'' =$ Sampling time corresponding to one standard deviation in log-normal distribution (at 84%).

The second moment about origin of sampling time is

$$\sigma = \mu [\exp(s^2) - 1]^{1/2} \quad (\text{B-12})$$

Since the dimensionless sampling time $\theta = t/T_m = t/\Delta\mu$

$$(\sigma_\theta)^2 = \frac{\sigma^2}{T_m^2} = \frac{2}{Pe} \quad (\text{B-13})$$

For liquid mixing experiments using two RTD curves to estimate the variance, the above equation becomes

$$(\Delta\sigma_\theta)^2 = \frac{\Delta\sigma^2}{T_m^2} = \frac{2}{Pe} = 2 \frac{D_1}{U_z L} \quad (\text{B-14})$$

Substituting values calculated from Equations B-11 and B-12 into Equation B-14, the Peclet number and dispersion coefficient can be calculated.

Sensitivity of Pe on Parameters s , μ , and σ

The application of the method of moments to estimate the dispersion coefficient is straightforward, as illustrated by Equation B-14. The value of Pe , however, is greatly influenced by the difference of the second moments. As the values of $\Delta\sigma^2$ approach zero, error associated with the Pe calculation increases rapidly.

In general, the first moments can be estimated with less relative error than the second moments. As can be seen from the logarithmic probability plot (Figure B-1) for the liquid mixing data, the first moment is estimated at a single point (50%) and is relatively insensitive to the slope of the line. The second moment, estimated from the difference between 84% and 50% points (one standard deviation), is very sensitive to the slope of the line.

The slope of the line is determined by the distribution of the cumulative area under the RTD curve, which in turn is affected by the value of the base line for the RTD curve. Based on analysis of the cold-flow liquid mixing data, a guideline was established for selecting a proper base line concentration. In general, if the initial sulfur concentration agrees closely with the average sulfur concentration of the tail, the average of the two is chosen. When this is not the case, the average sulfur concentration of the tail is chosen as the base line.

Variability between tests may also be estimated by comparison of results of experiments performed at similar conditions. Table B-I contains two such comparisons.

NOMENCLATURE FOR APPENDIX B

A	Cross-sectional area of the column.
C	Sulfur concentration, ppm.
C(s)	Laplace transform of C.
D ₁	Dispersion coefficient, cm ² /sec.
F(s)	Laplace transfer function, Equation B-8.
Pe	Peclet number.
Q	Amount of tracer injected into the system.
S	Standard deviation or square root of the variance in log scale.
T	Sampling time corresponding to C, sec.
T'	Sampling time corresponds to log-normal mean.
T''	Sampling time corresponds to one log-normal standard deviation.
T _m	Mean residence time, sec.
U _z	Average linear liquid velocity, cm/sec.
z	Axial position, cm.
z	Axial position, dimensionless (z/L).
<u>Greek</u>	
θ	Sampling time, dimensionless.
σ	Second moment or square root of the variance.
σ _θ	σ/T _m .
μ	First moment or mean of the RTD curve.

REFERENCES FOR APPENDIX B

- B-1) Aris, R., "Notes on the Diffusion-Type Model for Longitudinal Mixing in Flow," Chem Eng Sci, 9, 266 (1959).
- B-2) Bischoff, K. B., and O. Levenspiel, "Fluid Dispersion--Generalization and Comparison of Mathematical Models--I. Generalization of Models," Chem Eng Sci, 17, 245 (1962).
- B-3) Hopkins, M. J., A. J. Sheppard, and P. Eisenklans, "The Use of Transfer Function in Evaluating Residence Time Distribution Curves," Chem Eng Sci, 24, 1131 (1969).
- B-4) Levenspiel, O., and W. K. Smith, "Notes on the Diffusion-Type Model for Longitudinal Mixing of Fluids in Flow," Chem Eng Sci, 6, 227 (1957).
- B-5) Levenspiel, O., Chapter 9 of Chemical Reaction Engineering, Second Edition, John Wiley and Sons, Inc.
- B-6) Ostergaard, K., and M. L. Michelsen, "On the Use of the Imperfect Tracer Pulse Method for Determination of Hold-up and Axial Mixing," Can. J. Chem Eng, 47, 107 (1969).
- B-7) Sater, V. E., and O. Levenspiel, "Two-Phase Flow in Packed Beds," Ind Eng Chem Fund., 5, 86 (1966).

TABLE B-I

VARIABILITY BETWEEN DENSE-PHASE LIQUID MIXING RESULTS

<u>Run No.</u>	<u>U_g (cm/Sec)</u>	<u>U₁ (cm/Sec)</u>	<u>D₁ (cm²/Sec)</u>
490-11	2.80	2.96	76.0
490-20	2.90	2.95	72.2
490-23	2.87	2.90	<u>94.3</u>
Mean			80.83 ± 11.81
Standard Error			14.6%, 2 df
490-8	0	2.96	9.10
490-14	0	2.95	12.50
490-15	0	3.05	7.68
490-16	0	2.96	<u>13.3</u>
Mean			10.65
Standard Error			25.2%, 3 df

Figure B-1

PROBABILITY PLOT FOR RUN 490-08

APPENDIX C

VISCOSITY AND DENSITY
OF H-COAL PDU LIQUID SLURRY SAMPLES:
BATTELLE INSTITUTE FINAL REPORT

FINAL REPORT

on

VISCOSITY OF H-COAL SAMPLES

to

AMOCO OIL COMPANY

June 30, 1981

by

**J. W. Droege, D. R. Taylor,
and S. P. Chauhan**

**BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201**

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	1,
INTRODUCTION	1
RHEOLOGY	2
Viscometer	2
Procedure	5
Results	10
Changes with Time	15
Effect of Pressure	16
Replication	17
Comparison with ORNL Results	17
FUTURE WORK	20
REFERENCES	20

LIST OF TABLES

Table 1. Viscous Properties of AMOCO Samples	11
Table 2. Comparison of Results at 725 K	18

LIST OF FIGURES

Figure 1. Viscometer and Mixing Autoclave	4
Figure 2. Sample Data	6
Figure 3. Viscous Properties of AMOCO 15 at 700 K	19

VISCOSITY OF H-COAL SAMPLES

by

J. W. Droege, D. R. Taylor,
and S. P. Chauhan

June 30, 1981

ABSTRACT

The rheological properties of 14 samples taken from the H-COAL pilot plant have been determined at temperatures up to 725 K (845 F), mostly at a pressure of 17.24 MPa (2500 psia) of hydrogen. The samples showed Bingham-plastic behavior. It was found that the properties were time-dependent in that the yield stress decreased to near zero. The rate of this change was higher at higher temperatures. Most measurements were made without regard to this time dependency. Therefore the results at the highest temperatures are somewhat in doubt for this reason.

INTRODUCTION

On December 11, 1979, Battelle submitted its report to AMOCO on measurements made on a few H-COAL samples. On April 25, 1980, we submitted our proposal for more measurements and the current program got under way on September 18, 1980.

The objective of this research was to determine the rheological properties of 15 samples to be supplied by AMOCO. The samples were to be collected without exposure to air and were to be transferred into our apparatus without exposure to air. Measurements were to be made of a suitable range of shear rates at temperatures between 350 and 450 °C under hydrogen pressures of 2000 to 3000 psi.

It was not expected that the properties would change appreciably with time, but the possibility was recognized. Therefore measurements were begun at lower temperatures and continued at higher temperatures until the series was complete. With some of the samples repeat measurements were made

at lower temperatures after completing the high-temperature work. Some of the samples were maintained for a while at one temperature to check on the constancy of results. It became apparent that the results were indeed time-dependent. Since extensive constant-temperature measurements with extrapolation to zero time would have required large samples and a lot of time, only one such measurement was made in this manner, by way of illustration.

RHEOLOGY

Most of the work on rheology of coal-derived liquids has been done at atmospheric pressure and therefore not far above room temperature. Viscosities of concentrated slurries were found to be non-Newtonian and to be generally higher than predicted for spherical particles.⁽¹⁾ Coal-oil suspensions made with brown coal have been found to increase in viscosity with aging, even at room temperature.⁽²⁾ The viscosity of coal-derived liquids has been related to asphaltene and preasphaltene content.^(3,4) Aging of coal-derived liquids, especially in the presence of oxidizing gases, has been found to increase viscosity.⁽⁵⁾

A few attempts have been made to measure viscosity at elevated temperature and pressure. In a continuing program at Oak Ridge National Laboratory a continuous coal-liquids flow system has been instrumented for measurement of viscosity and density.⁽⁶⁾ The slurry is pumped through a length of heated tubing after which it passes through a constant-temperature section equipped with pressure taps. By varying the flow rate it is possible to vary shear stress and shear rate and thus to study the rheology of non-Newtonian mixtures. Since the residence time in the heated tubing is short, the viscosity of newly made sample can be determined.

Viscometer

A reciprocating concentric-cylinder viscometer was developed at Battelle for measuring the viscous properties of Synthoil liquids at elevated temperature and pressure.⁽⁷⁾ It seemed well suited for the present

measurements. The mechanical system is simple. There is little loss due to friction. The reciprocating action of the inner cylinder, the "bob", tends to keep the sample mixed. Data can be obtained quickly. The bob can be operated over a wide range of shear rates.

The apparatus is shown in Figure 1. It depends on the axial movement of concentric cylinders resulting in a pumping action which forces liquid to flow in the direction opposite to that of the inner cylinder in the space between the cylinders. A mixing autoclave, shown on the right, can be used to hold the sample at a controlled temperature prior to transfer into the viscometer.

A 0.3L autoclave constitutes the outer cylinder of the viscometer. A cylindrical bob (9) has a radius approximately 1-mm less than the inside radius of the autoclave. The bob is attached by tubing through a connector (6) to a large Alnico V bar magnet (5) and, by means of a thin rod, to a second smaller bar magnet (3). The magnets move freely inside tubing extending from the top of the autoclave. The bob is hollow, the space inside communicating with the autoclave atmosphere through the suspension tube. Outside the pressure system are located two large coils (4). When a current is passed through these two coils in opposite directions, an upward or downward force is exerted on the bob, depending on the direction of current flow. A second pair of coils (2) surrounds the smaller magnet. Motion of this magnet induces a current in these coils whereby the direction and rate of motion of the bob are detected. A vibrator helps to keep the bob moving smoothly.

Good temperature control is important. Because of the high heat capacity of the system it is important that the heaters be closely coupled to the metal of the autoclave. A sheathed heater is brazed to the body of the viscometer. It provides the primary temperature control. A control thermocouple is inserted into the bottom of the autoclave where it makes good contact with the metal. A three-action proportional controller is used. Two band heaters are used, one around the flange, the other around the cover, independently controlled to keep these parts at the same temperature as the body of the autoclave. A copper enclosure around the valves below the autoclave (10) is provided with strip heaters to keep the valves

FIGURE 1. VISCOMETER AND MIXING AUTOCLAVE

and tubing at the same temperature. The principal heat leak is through the water-cooled tube extending out the top of the autoclave. Its cooling effect is well compensated by the band heaters. A sheathed thermocouple (8), immersed in the sample just above the bob, shows the sample temperature. A similar temperature-control system keeps the preheater autoclave at constant temperature.

Valves and 1/4-in. tubing are provided, as shown, by which sample can be transferred from one autoclave to the other. Ports for the introduction and withdrawal of gas, for pressure measurement and control and for a safety valve are provided, several of which are shown (1,7). A sensitive back-pressure regulator controls the pressure. The mixing autoclave is stirred with a magnetically driven (13) turbine-type agitator (11). The viscometer autoclave is stirred only by the action of the bob. As an aid in keeping the solids in suspension, a burst of gas is occasionally admitted from below.

Procedure

The viscosity of a fluid in the apparatus can be calculated from the velocity and force acting on the bob. The equations have been worked out for Newtonian liquids. (7,8) The force applied to the bob was derived by calibration from the current passed through the lifting coils. From this calibration and from the dimensions of the apparatus the current scale could be converted to a shear-stress scale. The zero on this shear-stress scale was not easily established, since some force was required to keep the bob just suspended in the liquid. This force was related to the mass of the bob assembly and to the density of the liquid. For liquids not departing greatly from Newtonian behavior, the zero on the shear-stress scale could be estimated from data obtained with both a rising and a falling bob. From this value and from a knowledge of the volume of the bob, the density could be calculated. Shear rate was related to bob velocity through a calibration with a Newtonian standard.

Figure 2 shows a sample plot of shear stress versus shear rate. A force equivalent to about 3.65 on the shear-stress axis was required to overcome gravity in this instance. Two sets of points are shown, one for

FIGURE 2. SAMPLE DATA

a rising, the other for a falling bob. The gap between the two intercepts at zero shear rate is a measure of the yield stress. It will be seen that the data follow the Bingham-plastic model. The viscous behavior is characterized by two constants, as follows:

$$\tau = \tau_0 + \eta_{pl} \dot{\gamma}, \quad (1)$$

where τ_0 is the dynamic stress and η_{pl} is the "plastic viscosity", that is, that is, the slope of the line of Figure 2. The self-centering behavior of the bob works better on the rising stroke, therefore, results were based mostly on the upper line. Most of the data were obtained at shear rates between 10 and 100 s^{-1} .

When good data were obtained both with a rising and a falling bob, the point on the stress axis corresponding to a balanced bob could be determined (3.65 in Figure 2). This corresponds to a determination of the weight of the bob immersed in the liquid. A similar determination for the bob suspended in air and a determination of the volume of the bob are all that are needed for a density determination. This procedure was used for the density determinations shown. The procedure was not very successful at 400 K but appeared to give reliable results at higher temperatures.

The procedure for loading samples into the viscometer was as follows. The AMOCO sample bulb, wrapped with heating tape and insulation, was mounted vertically beside the apparatus shown in Figure 1. The valves at the bottom were connected with 1/4-in. tubing. The valve at the top of the sample bulb was connected to a low-pressure hydrogen supply and to a vent through a bubbler. Both autoclaves were first evacuated and flushed with hydrogen. After warming the sample to about 350 K the pressure in the sample bulb was relieved. Then a stream of hydrogen was passed up through the sample to provide some mixing.

The transfer was begun by applying a small pressure (about 20 psig) to the top of the sample bulb as well as to the mixing autoclave, opening connecting valves, then bleeding gas slowly from overflow tube 7 in the mixing autoclave. When the liquid level reached the overflow tube the flow of gas stopped and the connecting valve below this autoclave was closed. Hydrogen was then passed from the viscometer autoclave into the mixing autoclave to clear the connecting tube. The mixing autoclave now contained about 300 cm^3 of sample, a little more than three times as much

as the required sample. The charge was stirred vigorously for a time to homogenize the sample. Sample was then transferred in a similar way into the viscometer until it rose to the level of overflow tube 7 in this autoclave. Excess sample was blown out this overflow tube, thus leaving a sample of the required volume in the viscometer. Unused sample from the mixing autoclave was then transferred back into the sample bulb where it was kept with an overpressure of about 50 psig of hydrogen.

Before beginning the measurements the pressure in the viscometer was increased to the desired level, then the temperature of the viscometer was raised to the temperature chosen for the first measurements. After making the necessary measurements, the viscometer and contents were raised to the next higher temperature until the work was completed. At the conclusion of the measurement the sample was drained from the autoclave and discarded.

On several occasions we departed from the standard procedure, either intentionally or out of necessity. The exceptions will be described by sample number:

- AMOCO 1, 2, and 3. For these samples the pressure was increased and decreased throughout the run in an attempt to find the effect of pressure. These pressure changes were not made at the highest temperatures for fear of losing volatiles with the venting gas.
- AMOCO 3. Because of the formation of a plug in the transfer line, it was not possible to return sample to the sample bulb. The sample used for the measurements was subsequently removed through the top of the viscometer and, after exposure to air, was returned to the sample bulb.
- AMOCO 7. This was the sample chosen for our first measurements. We developed a plug during the first attempt to transfer. The sample was contaminated with air and water and some of it was evaporated after overheating. After the measurements we had trouble draining the sample and damaged our bob, which had not been properly calibrated. Therefore this run was declared to be a total loss.
- AMOCO 8. At the end of the second run with this sample we judged that no unused sample remained in the mixing autoclave. We therefore transferred the sample used for the measurements back into the sample bulb.

- AMOCO 12. There appeared to be insufficient sample for a full charge to the viscometer although apparently normal data were obtained. The sample was returned to the sample bulb after the measurements.
- AMOCO 13. We could find no sample in this bulb. Therefore, we presume the sample bulb now to be empty.
- AMOCO 15. In order to make one good observation of the effect of time on our results, a second measurement was made with this sample using a modified procedure. The overflow tube 7 in the mixing autoclave was lengthened so that a sample of the required size could be introduced into this autoclave. After making this transfer the autoclaves were both pressurized to 17.24 MPa (2500 psi) and the viscometer (but not the mixing autoclave) was heated to 700 K. The cold sample was then transferred to the preheated autoclave where it was heated rapidly to the measurement temperature. Data were recorded at 700 K for an hour and a half. Subsequently measurements were made at lower temperatures.
- AMOCO 16. We apparently encountered a plug in the line and found transfer difficult. We were unable to return sample to the sample bulb and presume that the sample bulb is now empty.

The viscometer, which had been performing acceptably at higher viscosities, gave data with a lot of scatter with these specimens. Presumably this was due to frictional effects, which represent a much more serious problem at low viscosities than at higher. During January, 1981, we made some vigorous attempts to correct these frictional problems.

For our bob to move up and down within the autoclave without friction requires good alignment. We had been relying on a flexible joint which allowed the bob to center itself in the autoclave even if the magnet assembly was not perfectly aligned. During January we made the following changes:

- (1) A tool was devised which enabled us to align the axis of the magnet housing accurately with the axis of the autoclave
- (2) One of the guides which kept the magnet assembly running true was removed. This eliminated one possible source of friction.

- (3) The flexible joint was replaced with a fixed connector.
- (4) The system was calibrated over the range of 0.3 to 33 mPA·s (cP).

The calibrations, which were based on the known viscosities of five liquids, were in satisfactory agreement. The scatter in the measurements on coal liquids was greatly reduced.

We believe that the older data, while flawed by excessive scatter, were otherwise valid. However the quality of data improved, beginning with AMOCO 4, run in late January, and subsequent determinations will be considered more reliable. Most of the measurements made prior to that time were subsequently repeated.

Results

The results of our measurements are shown in Table 1. Within each set of measurements on a single sample the results are shown in the order in which they were obtained. In most cases the first measurement was made at about 400 K. This routine was established in order to show gross differences from sample to sample, since measurements are more easily made at higher viscosities. The results show that there was the expected relationship between viscosities at lower and at higher temperatures, but there were also many departures from this relationship.

The accuracy of these results is plainly not high. It is difficult to place a numerical estimate on the uncertainty. Perhaps the best assessment would come from the numerous repeat determinations and comparisons with work at ORNL. These comparisons will be discussed in a subsequent section.

In almost all cases the data followed the relationship corresponding to Bingham plastic behavior. At low shear rates, of the order of 10 s^{-1} , shear rates often appeared to be higher than the Bingham-plastic straight line would predict. This may be an artifact of the measurement or it may indicate that the true behavior is somewhere between pseudo-plastic and Bingham plastic.

TABLE 1. VISCOUS PROPERTIES OF AMOCO SAMPLES

Temperature		Pressure		Elapsed Time,	Viscosity	Yield Stress, τ_0	Density,
K	$^{\circ}\text{F}$	MPa	psia	min	η_{pl} mPa·s	N/m ²	kg/m ³
<u>AMOCO 1 (March)</u>							
408.2	275.0	17.24	2500		10.4	.20	1065
625.5	666.1	13.79	2000		2.07	.05	909
624.9	665.1	17.24	2500		2.15	.04	907
625.2	665.6	20.69	3000		2.03	.03	901
648.5	707.5	13.79	2000		1.87	.06	881
649.2	708.8	17.24	2500		1.87	.05	884
648.9	708.3	20.69	3000		1.84	.04	882
673.2	752.0	20.69	3000		1.77	.08	858
673.5	752.5	17.24	2500		1.77	.08	858
673.2	752.0	13.79	2000		1.71	.08	862
699.2	798.8	17.24	2500		1.73	.09	828
724.0	843.4	17.24	2500		1.33	.13	766
673.2	752.0	17.24	2500		1.84	.06	832
<u>AMOCO 1 (November)</u>							
413.4	284.4	17.24	2500		8.8	.13	1033
623.5	662.5	13.79	2000		1.76	.06	900
624.3	664.0	17.24	2500		1.72	.02	899
624.8	664.9	20.69	3000		1.72	.01	897
645.3	701.8	13.79	2000		1.45	.12	869
645.7	702.5	17.24	2500		1.63	.13	870
646.2	703.4	20.69	3000		1.46	.11	870
669.2	744.8	13.79	2000		1.81	.12	856
698.2	797.0	17.24	2500		1.55	.08	818
723.2	842.0	17.24	2500	4	1.28	.16	762
722.7	841.1	17.24	2500	18	1.68	.24	755
723.1	841.8	17.24	2500	26	1.87	.32	750
723.2	842.0	17.24	2500	34	2.03	.33	755
<u>AMOCO 2 (May)</u>							
406.6	272.3	17.24	2500		16.6	.26	1080
626.2	667.4				2.10	.10	913
648.2	707.0				1.92	.13	895
672.4	750.7				2.01	.15	844
672.2	750.2			37	1.92	.13	849
697.4	795.6			67	1.81	.20	804
725.0	845.2			97	1.76	.20	763
<u>AMOCO 2 (December)</u>							
405.2	269.6	17.24	2500		14.0	.31	1107
627.0	668.8	13.79	2000		2.18	.07	925
627.2	669.2	17.24	2500		2.07	.05	932
626.6	668.3	20.69	3000		1.98	.09	926
646.0	703.2	13.79	2000		1.57	.11	903
646.6	704.3	17.24	2500		1.82	.13	899
648.2	707.0	20.69	3000		1.91	.09	900
672.6	751.1	17.24	2500		1.38	.17	865
696.2	793.4	17.24	2500		1.60	.18	836
719.6	835.7	17.24	2500		1.53	.23	798
<u>AMOCO 3 (March)</u>							
409.0	276.4	17.24	2500		80.2	2.0	
627.0	668.8	13.79	2000		5.81	.42	995
627.3	669.4	17.24	2500		5.53	.42	996
627.2	669.2	20.69	3000		5.60	.42	1000
627.4	669.6	17.24	2500		5.23	.35	985
622.7	661.1	13.79	2000		5.22	.37	993
649.5	709.3	13.79	2000		5.00	.39	979
650.2	710.6	17.24	2500		4.97	.38	977

TABLE 1. (Continued)

-249

Temperature		Pressure		Elapsed	Viscosity	Yield	
K	°F	MPa	psia	Time, min	η_{pl} mPa·s	Stress, τ_0 N/m ²	Density, kg/m ³
<u>AMOCO 3 (March)</u> (Continued)							
650.0	710.2	20.69	3000		4.94	.35	973
673.4	752.4	20.69	3000		3.58	.32	944
673.2	752.0	17.24	2500		3.52	.29	941
672.8	751.3	13.79	2000		3.70	.28	945
699.0	798.4	13.79	2000		2.95	.22	911
699.0	798.4	17.24	2500		2.65	.20	905
699.0	798.4	20.69	3000		2.77	.18	900
723.2	842.0	17.24	2500		2.20	.14	875
699.1	798.6	17.24	2500		2.11	.07	902
409.6	277.5	17.24	2500		46.3	.00	1093
<u>AMOCO 4 (May)</u>							
407.6	274.1	17.24	2500	0	44.5	1.52	1069
622.2	660.2			80	3.46	.22	968
646.6	704.3			120	3.13	.22	952
646.2	703.4			190	3.11	.18	950
672.2	750.2			230	2.78	.15	925
698.2	797.0			250	1.85	.16	896
722.7	841.2			280	1.72	.08	863
722.8	841.5			288	1.60	.07	865
722.6	841.1			316	1.31	.00	859
722.6	840.9			345	1.32	.01	861
722.6	840.9			381	1.31	.01	860
<u>AMOCO 4 (January)</u>							
407.2	273.2	13.79	2000		31.8		
626.2	667.6				3.65	.18	958
649.2	708.8				3.51	.18	942
673.6	752.7				2.78	.20	922
697.4	795.7				2.12	.20	896
722.4	840.7				1.74	.09	887
<u>AMOCO 5 (February)</u>							
408.2	275.0	12.41	1800		73.0		
623.6	662.9	13.10	1900		3.49	.10	981
647.4	705.6				2.66	.11	971
673.2	752.0				2.62	.07	946
698.7	797.9				2.38	.06	924
722.7	841.1				2.19	.06	897
<u>AMOCO 6 (February)</u>							
408.5	275.5	17.24	2500		197		
623.7	662.9				5.26	.42	1032
647.2	705.2				4.93	.40	1021
670.5	747.1				4.46	.32	995
697.4	795.6				3.42	.26	966
723.2	842.0				2.30	.08	938
<u>AMOCO 8 (April)</u>							
407.2	273.2	17.24	2500		20.8	.60	1071
621.6	659.3				4.20	.15	960
648.2	707.0				3.96	.14	938
672.2	750.2				3.78	.18	917
699.2	798.8				3.67	.15	877
724.2	843.8				2.59	.08	844
698.6	797.9			0	2.32	.02	864
698.2	797.0			50	2.16	.02	855
697.6	796.1			80	2.40	.00	865
698.2	797.0			120	2.33	.00	849

TABLE 1. (Continued)

Temperature		Pressure		Elapsed Time, min	Viscosity η_{pl} , mPa·s	Yield Stress, τ_0 , N/m ²	Density, kg/m ³
K	°F	MPa	psia				
<u>AMOCO 8 (December)</u>							
352.2	174.2	17.24	2500		147		
416.6	290.3				17.1	.53	1092
625.4	666.0				3.30	.25	969
649.2	709.0				3.77	.22	953
672.2	750.2				2.58	.21	923
696.6	794.1				2.41	.15	910
725.0	845.4				2.20	.16	883
<u>AMOCO 9 (April)</u>							
407.8	274.3	17.24	2500		19.9	.38	1076
624.0	663.4				3.25	.14	956
648.2	707.0				2.60	.14	940
674.4	754.2				2.30	.12	916
699.2	798.8				1.82	.09	881
725.2	845.6				1.81	.04	844
700.2	800.6			0	1.19	.00	850
700.6	801.5			25	1.10	.00	848
701.2	802.4			40	1.15	.00	844
701.4	802.8			55	1.15	-.01	843
701.2	802.4			70	1.09	-.01	841
<u>AMOCO 9 (December)</u>							
407.4	273.6	17.24	2500		24.0	.48	1120
626.2	667.4				2.71	.25	930
648.8	708.3				2.15	.28	919
650.0	710.4				3.35	.31	912
672.6	750.9				2.33	.34	895
695.6	792.3				2.55	.37	800
695.8	792.7				3.24	.36	881
717.0	830.8				1.80		
<u>AMOCO 10 (April)</u>							
408.4	273.5	17.24	2500		37.2	1.03	1121
625.6	666.5				3.61	.18	963
648.6	707.9				3.25	.16	937
677.0	758.8				3.01	.13	915
700.6	801.5				1.92	.12	877
724.6	844.7				1.74	.10	838
699.4	799.3			0	1.42	.01	852
699.6	799.7			34	1.31	.01	866
699.2	798.8			63	1.20	.00	864
698.2	797.0			91	1.22	-.01	865
698.8	798.3			137	1.24	.00	863
<u>AMOCO 10 (January)</u>							
407.0	273.0	17.24	2500		31.0	.53	1122
625.0	665.4				2.08	.18	965
649.2	708.8				1.92	.16	935
673.6	752.7				1.60	.13	917
697.0	794.8				1.64	.12	884
725.4	846.0				1.10	.17	870
<u>AMOCO 11 (February)</u>							
409.7	277.7	17.24	2500		31.0	.23	1244
620.2	656.6				4.92	.48	1028
649.4	709.2				4.43	.47	1011
672.7	751.1				3.88	.46	990
697.9	796.9				2.95	.41	965
721.4	838.8				2.23	.23	938

TABLE 1. (Continued)

Temperature		Pressure		Elapsed Time,	Viscosity	Yield Stress, τ_0	Density,
K	$^{\circ}\text{F}$	MPa	psia	min	η_{pl} mPa·s	N/m ²	kg/m ³
<u>AMOCO 12 (February)</u>							
408.2	275.0	17.24	2500		18.4	.49	1067
723.7	662.9	↓	↓		2.81	.08	950
647.7	706.1				2.25	.10	931
672.7	751.1				1.94	—	(906)
698.5	797.5				(2.05)	.11	890
723.5	842.5	↓	↓		1.58	.06	873
<u>AMOCO 14 (March)</u>							
411.2	280.4	17.24	2500		24.7	1.41	1096
624.0	663.4	↓	↓		1.77	.08	978
647.8	706.3				1.47	.08	949
673.4	752.4				1.18	.05	928
698.0	796.6	↓	↓		.89	.01	897
724.3	844.0	↓	↓		.74	.00	864
<u>AMOCO 15 (March)</u>							
410.0	278.2	17.24	2500		96.3	.76	1190
626.1	667.2	↓	↓		6.74	.38	1031
650.0	710.2				5.24	.26	1002
674.3	754.0				3.53	.21	975
697.6	795.9	↓	↓		2.88	.18	947
723.3	842.2	↓	↓		2.13	.06	928
<u>AMOCO 15 (June)</u>							
353.2	176.0	17.24	2500	0			
702.0	805.0	↓	↓	8	4.66	.54	989
694.0	790.0			11	3.74	.51	986
702.6	805.1			18	3.85	.41	978
700.2	800.8			28	3.52	.29	972
702.6	804.9			36	3.24	.24	969
701.2	802.4			60	2.77	.14	955
701.6	803.1			75	2.73	.10	950
702.8	805.3			85	2.73	.08	948
704.0	807.4			100	2.81	.06	941
677.6	760.1			135	2.84	.04	955
649.0	708.6	↓	↓	220	3.12	.04	976
623.4	662.5	↓	↓	257	3.61	.05	988
<u>AMOCO 16 (March)</u>							
408.7	275.9	17.24	2500		59.9	.88	1220
623.7	662.9	↓	↓		4.92	.11	1028
648.2	707.0				4.14	.11	1012
673.5	752.5				3.49	.11	994
698.5	797.5	↓	↓		2.54	.05	966
724.0	843.4	↓	↓		2.11	.04	956

The plastic viscosity was found to decrease as the temperature increased, as expected. The relationship between yield stress and temperature was not so clear. As will be shown in the next section, the yield stress at any high temperature was found to decrease with time. Therefore a decrease in yield stress with increasing temperature, as was generally observed, may have been due in part to change with time. Nevertheless, there did seem to be some decrease of yield stress with increasing temperature. This effect was relatively small, however. The determination of yield stress at 400 K was not very reliable because of the higher viscosities and the uncertainty in the density.

Changes with Time

The program was devised with the assumption that the samples would not change appreciably with time. It was recognized, however, that changes were possible. Therefore measurements were made as rapidly as possible, starting at lower temperatures and continuing at higher temperatures. In several cases (1, 3, 8, 9, 10) measurements at a lower temperature were repeated after completing the high-temperature work to determine whether any changes had occurred. It was found that both lower plastic viscosities and lower yield stress values were measured after holding at a higher temperature.

On several occasions we held the specimen for a while at constant temperature to determine whether the properties were constant. In general (8, 9, 10) we found a slight decrease in plastic viscosity and a decrease in yield stress. The yield stress at 700 K after previous measurements at 725 K were already low, but they appeared to decrease essentially to zero. AMOCO 1 appeared to be an exception to this behavior. After half an hour at the highest temperature, both plastic viscosity and yield stress were increasing.

At the end of the program, with the opportunity for one more experiment, it was determined to devote this one experiment to a determination of the changes with time at a single temperature. AMOCO 15, previously measured in March, was chosen for this experiment. The sample

was transferred from an autoclave at 353 K to the viscometer at about 700 K. Measurements began immediately but it took about 8 minutes to bring the sample to this temperature. The behavior at 400 K is shown in Figure 3.

The decrease in plastic viscosity seems to be clearly indicated, though the apparent upturn at the end is not so certain. The yield stress changed in a much more pronounced way, decreasing nearly to zero in less than 2 hours. At 425 K the change would, no doubt, be more rapid. The change in density was unexpected. The change was about 5 percent, which is a substantial change. The data seem to indicate this change clearly. We are not sure what to make of it.

The data obtained during sample heatup, while not sufficient for treatment in the usual way, indicate a thickening of the sample, whether caused by an increase in plastic viscosity or an increase in yield stress is not clear. This time period was difficult to interpret because rising temperatures were changing density and therefore buoyancy of the bob. Therefore we cannot be sure about this apparent transitory phenomenon.

One more peculiarity in the data should be pointed out. For AMOCO Samples 1 and 2 the trend with time and for yield stress even with temperature seems to be the reverse of that generally observed. Each sample was run twice, and in each of the four runs there appears to be an upward trend in yield stress.

Effect of Pressure

For several samples measurements were made at pressures of 13.8, 17.2 and 20.7 MPa of hydrogen. No discernible trend was detected. Previous experience with similar but much more viscous materials indicates that the pressure effect might have been expected to be about 5 percent for a 1000 psi change -- higher viscosity for the higher pressure. The scatter in our data was often greater than that.

Replication

In all, six of the determinations were repeated. The scatter was great, even after the January corrections. Furthermore, in most cases the earlier results in plastic viscosity were consistently lower than the later. The average difference between the earlier and later determinations ranged from 11 percent for AMOCO 1 to 36 percent for AMOCO 10. Sample 1 is said to be the same as Sample 2; similarly Samples 8 and 9 were duplicates. Each of these four was run twice. The deviations between the pairs in each case were of the same order of magnitude as deviations between repeat runs.

A similar comparison between repeat determinations of yield stress shows a similar level of agreement. At higher temperatures the agreement is worse, presumably as the yield stress decreased with time for each measurement.

In general we would have to say that the agreement between repeat determinations was less good than we had hoped. This is only partly due to uncertainties regarding the condition of the sample, such as the possible separation of solids before transferring the sample to the viscometer, uncertainties in the extent of change brought about by standing at high temperature and the like. A large part of the uncertainty seems to be due to instrumental problems such as the lack of alignment.

Comparison with ORNL Results

It is our understanding that the correspondence of samples used by ourselves and ORNL is as shown in Table 2. Because of the changes with time we have shown also our yield stress results at 700 K, which we judge to be closer to the value at zero time than the value determined at 725 K.

Although the order of magnitude of the results was the same as determined by the two methods, the agreement is not very close. Especially in the case of AMOCO 11 and Sample 43A the results are in serious disagreement, even in density. Because of the rapidity of changes with time, which is no doubt greater at 725 K than shown in Figure 3 at 700 K, and because of the differences in residence time for the two methods, the differences

TABLE 2. COMPARISON OF RESULTS AT 725 K

<u>ORNL Results</u>				
Sample Number	27B	34A	42A	43A
η_{pl} , mPa·s	1.68	3.03	1.73	1.07
τ_o , N/m ²	.126	.307	.232	.192
ρ , kg/m ³	908	985	882	851
<u>This Research</u>				
Sample Number	5	6	9	11
η_{pl} , mPa·s	2.19	2.30	1.81	2.23
τ_o at 725, N/m ²	.06	.08	.04	.23
τ_o at 700, N/m ²	.06	.26	.09	.41
ρ , kg/m ³	897	938	844	938

FIGURE 3 VISCOSUS PROPERTIES OF AMOCO 15 AT 700 °F

in results were perhaps no greater than should be expected. Even so, the differences between 11 and 43A seem to be excessive.

FUTURE WORK

Further determinations of rheological properties of samples such as those examined in this program would have to be based on an experimental design that deals with the changes in properties with time. Experiments similar to the one illustrated in Figure 3 could be used to extrapolate results back to zero time. With a little practice we believe that usable results could be obtained within 2 or 3 minutes of the transfer. Similar measurements at other temperatures would aid in interpretation of the results.

Further development of the instrument, together with some stable samples with which to make comparisons, should lead to greater confidence in the results.

REFERENCES

- (1) Castillo, C. and Williams, M. C., "Rheology of Very Concentrated Coal Suspensions", Chem. Eng. Commun., (3), 529-547 (1979).
- (2) Bhattacharya, S. N. and Barro, L., "Rheology of Aging of Coal-Oil Suspensions", Rheology (Proceedings of 8th International Congress), 1980, 2, 615-621.
- (3) Krzyzanowska, T. and Marzec, A., "Coal-Derived Group Component Effects on Viscosity", Fuel, 57, 804-805 (1978).
- (4) Bockrath, B. C., Lacount, R. B., and Noceti, R. P., "Coal-Derived Asphaltenes: Effect of Phenol Content and Molecular Weight on Viscosity of Solutions", Fuel, 59, 621-626 (1980).
- (5) Brown, F. R. and Karn, F. S., "Stability Studies of Coal Liquids", PETC/TR-79/5.
- (6) Oswald, G. E., Youngblood, E. L., and Hightower, J. R., Jr., "Rheological Characterization of Coal-Solvent Slurry at High Temperature and Pressure", AIChE Meeting, Philadelphia, Pennsylvania, June 8-12, 1980.

REFERENCES
(Continued)

- (7) Droege, J. W., Hassell, J. A., and Chauhan, S. P., "Viscosity at Elevated Temperature and Pressure of Coal-Derived Liquids", Proc. 7th Symp. Thermophysical Prop., Gaithersburg, Maryland, May 10-12, 1977, ASME.
- (8) Smith, T. L., Ferry, J. D., and Schremp, F. W., J. Appl. Phys., 20, 144-153 (1949).

Battelle

Columbus Laboratories

505 King Avenue

Columbus, Ohio 43201

Telephone (614) 424-6424

APPENDIX D

VISCOSITY AND DENSITY
OF H-COAL PDU LIQUID SLURRY SAMPLES:
OAK RIDGE NATIONAL LABORATORIES FINAL REPORT

OAK RIDGE NATIONAL LABORATORY

OPERATED BY
UNION CARBIDE CORPORATION
NUCLEAR DIVISION

POST OFFICE BOX X
OAK RIDGE, TENNESSEE 37830

-261

July 8, 1981

Mr. Robert J. Schaefer
Amoco Oil Company, H-6
P. O. Box 400
Naperville, Illinois 60566

Dear Bob:

Enclosed is our final report on viscosity and density measurements of the H-Coal samples. This will be published soon.

If you have any questions, do not hesitate to call.

Sincerely,

A handwritten signature in dark ink, appearing to read "Bob Hightower". The signature is fluid and cursive, with a long, sweeping underline.

J. R. Hightower, Jr., Head
Advanced Technology Section
Chemical Technology Division

JRH:jts

Enclosure

cc: D. D. Lee
B. R. Rodgers
J. H. Wilson

-262

Contract No. W-7405-eng-26

CHEMICAL TECHNOLOGY DIVISION

FINAL REPORT - H-COAL PRODUCT PHYSICAL
PROPERTIES MEASUREMENT

D. D. Lee

Date Published:

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
operated by
UNION CARBIDE CORPORATION
for the
DEPARTMENT OF ENERGY

TABLE OF CONTENTS

-263

	PAGE
ABSTRACT	1
1. INTRODUCTION	1
2. EXPERIMENTAL APPARATUS	3
3. EXPERIMENTAL PROCEDURE	7
4. DATA ANALYSIS AND DISCUSSION	11
5. REFERENCES	18

FINAL REPORT - H-COAL PRODUCT PHYSICALPROPERTIES MEASUREMENT

D. D. Lee

- ABSTRACT

Rheological and density measurements have been made on the H-Coal PDU reactor effluent samples from run PDU-10, periods 27B, 34A, 42A, and 43A, which used Wyodak coal. The characterization was done in the ORNL Coal Liquids Flow System (CLFS) at 719 K and 19.3 MPa, which closely approximated the conditions in the H-Coal PDU reactor at the time the samples were taken. Under these conditions, the apparent viscosities of the samples at a shear rate of 300 s^{-1} were 2.1, 4.0, 2.5, and 1.5 mPa·s (cP), respectively. The corresponding densities were 0.908, 0.985, 0.882, and 0.851 g/cm³. The rheological data were fitted to both power law and Bingham plastic models. Samples 27B and 34A were described almost equally well by both models, while samples 42A and 43A were better described by the Bingham plastic model. The apparent viscosities appeared to correlate to the amount of solids in the samples.

1. INTRODUCTION.

The density and rheology of H-Coal PDU reactor effluent collected in high-pressure sample bombs during PDU operation have been measured at the temperatures and pressures of reactor operation. Such information is needed for the design and operation of the ebullated-bed reactor. The viscosity determines the minimum liquid recirculation rate necessary for fluidization of the catalyst bed, and the maximum rate above which catalyst particles

are carried out of the reactor. The samples 27B, 34A, 42A, and 43A were taken during PDU run No. PDU-10. The sample information is shown in Table 1. The measurements were made in the ORNL Coal Liquids Flow System (CLFS), which is designed to operate at up to 810 K and 31 MPa. The CLFS consists of a slurry feed system (1.2 g/s capacity), slurry preheater, reactor, high-pressure hydrogen or inert gas supply, vapor-liquid separator, and liquid and gas pressure letdown systems. The liquid stream from the preheater exit is passed through the physical property instruments at the desired process conditions for measurement.

Table 1. Data for H-Coal PDU effluent samples

Data Component	Sample 27B	Sample 34A	Sample 42A	Sample 43A
PDU run No.	PDU-10	PDU-10	PDU-10	PDU-10
PDU period No.	27B	34A	42A	43A
Feed rates				
Coal, kg/h	111	106	107	105
Liquid, kg/h	179	186	207	207
Gas, m ³ /h	203	207	209	272
Liquid/coal weight ratio	1.61	1.75	1.92	1.96
Coal type	Wyodak	Wyodak	Wyodak	Wyodak
Reactor temperature, K	719	719	716	718
Reactor pressure, MPa	19.3	19.6	19.3	19.3

2. EXPERIMENTAL APPARATUS

The Coal Liquids Flow System (CLFS), shown schematically in Fig. 1, was designed to permit study of both rheological properties and density of coal liquefaction process streams. For measurement of H-Coal samples, the basic CLFS was modified by adding heating tapes and insulation to the outside of the feed tank and fitting the tank for operation under slight helium pressure (<100 kPa). The H-Coal slurry was heated to 400 K in the feed tank to permit easy flow to the measurement system. A Bran-Lubbe positive displacement pump was used to feed the sample to the preheater, which is a 12-m-long serpentine coil of 2.8-mm-ID stainless steel tubing wrapped with high-temperature electrical resistance heating tape capable of supplying 2.2×10^4 J/s·m² heat flux. The pipeline viscometer (PLV) (0.61 m long) of the same internal diameter as the preheater is located immediately downstream from the preheater. Figure 2 is a schematic diagram of the viscometer. The viscometer is equipped with a differential pressure cell for measuring the pressure drop across the viscometer and two chromel-alumel thermocouples for measuring the inlet and outlet slurry temperatures.

Located downstream of the viscometer is the gamma-radiation absorption instrument, which is used for measuring the density of coal liquids and slurries. Figure 3 is a schematic of the experimental assembly, which consists of the commercially available gamma-radiation absorption instrument* and a 0.030-m-ID x 0.042-m-OD pressure vessel, which was constructed at ORNL. Density is determined by the attenuation of 662-keV gamma rays from the ¹³⁷Cs source as they pass through the fluids. The attenuation by

*Kay-Ray 3600F Liquid-Slurry Density System manufactured by Kay-Ray, Inc., 516 W. Campus Dr., Arlington Heights, Ill.

ORNL-DWG 60-66R

COAL LIQUIDS FLOW SYSTEM
FOR H-COAL TESTS

ORNL DWG 78-18277R

PIPELINE VISCOMETER

ORNL DWG. 78-1128

GAMMA ABSORPTION LIQUID DENSITY GAUGE

the pressure vessel and the correction for the pressure vessel expansion with temperature are taken into account.

3. EXPERIMENTAL PROCEDURE

In preparation for the rheological characterization and density measurements, the slurry was removed from each of the sample bombs in turn (7-liter capacity each) by first melting the slurry by heating the surface of the bomb to ~ 435 K. The slurry was discharged from the bombs directly into the feed tank by applying helium overpressure. About 3 to 5 kg of material was recovered from each bomb. The material was placed in a stirred and heated feed tank. The slurry mass feed rate to the system was determined by continuously monitoring the feed tank weight with an electronic load cell. Feed tank temperature was controlled at 400 K. The slurry displayed almost no tendency to settle out. Therefore, the feed to the Bran-Lubbe pump was maintained by a slight positive gas pressure (< 5 psig) on the feed tank rather than by a recirculating pump as in normal operations. The feed pump and the lines were heat traced and controlled at 400-450 K to maintain fluidity. Samples 27B and 43A were easily deformable at room temperature, while 34A and 42A were slightly firmer. The preheater was operated with constant heat flux along its length; heat input was controlled such that the slurry just reached the measurement temperature at the preheater exit-viscometer entrance. Thus, slurry residence time at the measurement temperature before it entered the viscometer was zero. The viscometer was maintained at the measurement temperature by an independent trace heat system.

The slurry then passed into the gamma-radiation absorption density instrument at the viscometer discharge. The density measurements were made at the same time as the rheology tests. The slurry feed rate was varied and density was measured at each feed rate for samples 27B, 34A, and 42A and measured at the first rate for sample 43A. The slurry was kept at the measurement temperature by trace heating on the density instrument feed lines and by a separate control circuit on the pressure vessel wall, which was maintained at 719 K. The output voltage of the density instrument was recorded by the on-line digital computation system. The gamma-density instrument calibration was checked before and after each run and calibrated as described previously.¹ For the first two samples, the H-Coal slurry from the CLFS was collected in high-pressure sample bombs so it could be reused if needed. However, a line between the bomb and the off-gas letdown valve plugged on the run with the third sample, and the slurry was diverted to the regular product tank. The slurry was also caught in the regular product tank in the fourth sample run because of solvent purging of the dp cell lines on the PLV, which diluted the H-Coal.

The viscometer differential pressure, inlet and outlet temperatures, and the slurry mass flow rate (varied from 0.1 g/s to 1.4 g/s) were recorded by the on-line digital computer data acquisition and analysis system. Each sample run lasted about 1-1/2 h while data were being taken, and the total time including heatup, cool-down, and calibration lasted about 10 h. Data are shown in Tables 2 and 3. There was no evidence of additional slurry liquefaction reactions as the data were being taken.

Table 2. Results of CLFS runs on II-Coal PDU-10: samples 27B, 34A, and 42A

Sample	Feed rate (g/s)	Average P.I.V AP (kPa)	Average P.I.V temperature (K)	Densitometer temperature (K)	Density (g/cm ³)	8V/D (s ⁻¹)	τ_w (N/m ²)	Apparent viscosity $\tau_w/\dot{\gamma}_w$ (mPa·s)
27B	1.162	1.00	715	728	0.931	595	1.132	1.76
	1.128	1.00	711	728	0.937	575	1.132	1.77
	0.689	0.70	718	729	0.921	353	0.792	2.03
	0.750	0.75	717	728	0.921	384	0.849	1.98
	0.469	0.52	712	727	0.907	239	0.594	2.26
	0.232	0.35	706	729	0.897	118	0.396	2.75
	0.155	0.22	697	724	0.872	78	0.255	3.08
	0.340	0.37	701	722	0.876	172	0.424	2.48
34A	0.449	0.92	706	720	0.981	217	1.047	4.38
	0.441	0.90	710	734	0.952	214	1.019	4.41
	0.832	1.40	721	727	0.987	405	1.585	3.78
	0.827	1.42	721	718	0.987	402	1.613	3.72
	1.048	1.72	719	717	1.002	509	1.952	3.50
	1.025	1.72	721	717	0.994	499	1.952	3.52
	0.658	1.24	721	720	1.001	320	1.415	3.96
	0.710	1.27	721	722	0.980	345	1.443	3.88
	0.686	1.27	722	724	0.980	334	1.443	3.81
	0.266	0.75	714	716	0.986	129	0.849	5.04
42A	0.650	0.87	710	718	0.899	348	0.990	2.61
	1.324	1.40	721	723	0.882	716	1.585	2.03
	0.921	1.15	719	720	0.894	497	1.302	2.40
	1.217	1.20	710	718	0.876	652	1.358	1.91
	0.523	0.67	705	718	0.891	279	0.764	2.51
	0.702	0.77	710	721	0.872	376	0.877	2.14
	0.405	0.77	709	720	0.874	217	0.877	3.71
	0.251	0.72	705	720	0.874	134	0.821	5.63
	0.158	0.55	697	722	0.874	84	0.622	6.82

Table 3. Results of CLFS run on H-Coal PDU-10, sample 43A, reactor effluent slurry

Time	Feed rate (g/s)	PLV Φ (kPa)	Avg PLV (K)	Density ^a (g/cm ³)	8V/D (s ⁻¹)	τ_w (N/m ²)	n $\left(\frac{d \ln \tau_w}{d \ln 8V/D} \right)$	$\dot{S}_w$ $\left(\frac{8V}{D} \cdot \frac{3n+1}{4n} \right)$	$\tau_w = k \dot{S}_w^n$ (N/m ²)	Apparent viscosity $\tau_w / \dot{S}_w$ (mPa·s)
1238	0.665	0.750	716	0.851	374	0.608	0.838	392	0.612	1.56
1304 ^b	0.880	0.949	711		493	0.770	0.838	517	0.771	1.49
1312	1.377	1.395	716		785	1.132	0.838	823	1.138	1.38
1324 ^b	0.450	0.617	696		249	0.501	0.473	318	0.501	1.58
1332	0.329	0.562	699		183	0.456	0.244	324	0.461	1.42
1342 ^b	0.174	0.488	691		96	0.396	0.244	170	0.394	2.32
1349	0.109	0.433	692		60	0.351	0.244	106	0.351	3.30
1403 ^b	1.025 ^c	0.102	708		574	0.894	0.838	602	0.876	1.45
1411	0.665 ^c	0.757	710		373	0.614	0.838	391	0.610	1.56

^aDensity was measured only during the first data point.

^bPipeline viscometer pressure taps purged before measurement.

^cCorrected readings from weigh cell recalibration tests.

4. DATA ANALYSIS AND DISCUSSION

To produce a rheogram from the viscometer differential pressure vs flow rate data at constant temperature, the Rabinowitsch-Mooney (R-M) analysis was used. For steady, time-independent laminar flow, a log-log plot of the R-M parameter, $8V/D$, vs the wall shear stress is made. Flow rate measurements were used to determine the slurry superficial velocity (V), and the viscometer internal diameter (D) was precisely measured. The wall shear stress (τ_w) is determined from a force balance over the length (L) of the viscometer,

$$\tau_w = \frac{D\Delta P}{4L}, \quad (1)$$

where ΔP is the pressure drop across the viscometer. Figure 4 shows the flow curves developed for the H-Coal samples.

To construct a rheogram, values of wall shear rate are determined over the range of wall shear stress achieved during each series of measurements at constant temperature and pressure. This is accomplished by determining the slope (n) of the flow curve at various values of wall shear stress (τ_w). The slopes are then used in the Rabinowitsch-Mooney relation to convert the corresponding $8V/D$ value to the wall shear rate ($\dot{S}_w$):

$$\dot{S}_w = 8V/D \left(\frac{3n+1}{4n} \right). \quad (2)$$

If the power law model is assumed (linear behavior of the flow curve) to describe the flow behavior,

$$\tau_w = k\dot{S}_w^n, \quad (3)$$

where k is the consistency factor determined from the flow curve by

$$k = \tau_w \left(\frac{3n + 1}{4n} \right)^{-n} \quad (4)$$

at $8V/D = 1$ and n is called the flow behavior index. The slurry is Newtonian if n is equal to 1, pseudoplastic if the slope is less than 1, and dilatant if the slope is greater than 1.

The data can also be fitted to the Bingham plastic model using the analytical solution for the Rabinowitsch-Mooney analysis assuming Bingham plastic behavior.² Thus,

$$8V/D = \frac{\tau_w}{\eta} - \frac{\tau_y^4}{3\eta} + \frac{\tau_y^4}{3\eta\tau_w^3} \quad (5)$$

where τ_y is yield stress and η is plastic viscosity in the Bingham plastic constitutive equation

$$\tau_w = \tau_y + \eta \dot{\gamma}_w \quad (6)$$

The plots on rectangular coordinates of τ_w vs $8V/D$ for the H-Coal samples are shown in Fig. 5.

The results of the rheological analyses are presented in Table 4, and the chemical analyses of the samples are shown in Table 5. From Table 4 and Figure 5, the data for all of the H-Coal samples fit the Bingham plastic model quite well. From Table 4 and Figure 4, the data for samples 27B and 34A also fit the power law model. However, using the power law model, the data for samples 42A and 43A produced two different slopes for each sample which depended on the shear rate. Below $8V/D = 300 \text{ s}^{-1}$, the slopes are

Table 4. Rheological analyses results^a

Sample No.	Power law model parameters				Bingham plastic model				Density (g/cm ³)
	Slope	k	r ²	μ_a	τ_y	η	r ²	μ_a	
PDU-10-27B	0.724	10.51	0.989	2.18	127.0	1.68	0.990	2.10	0.908
PDU-10-34A	0.724	18.80	0.993	4.12	306.7	3.03	0.993	4.05	0.985
PDU-10-42A	0.734 ^b	11.75	0.927	2.58	232.4 ^c	1.73	0.919	2.51	0.882
PDU-10-42A	0.215 ^d	225.3	0.632	2.56					
PDU-10-43A	0.838 ^e	4.11	0.998	1.63	191.1 ^e	1.07	0.983	1.71	0.838
PDU-10-43A	0.244 ^f	112.3	0.997	1.51					

^ak = consistency factor (mN·sⁿ/m²).

r² = correlation coefficient for least squares analysis.

μ_a = apparent viscosity at $\dot{\gamma} = 300 \text{ s}^{-1}$ (mPa·s).

τ_y = yield stress (mN/m²).

η = plastic viscosity (mN·s/m²).

^bCalculations based on $8V/D > 200 \text{ s}^{-1}$.

^cCalculations based on $8V/D = 50 \text{ to } 800 \text{ s}^{-1}$.

^dCalculations based on $8V/D < 400 \text{ s}^{-1}$.

^eCalculations based on $8V/D > 300 \text{ s}^{-1}$.

^fCalculations based on $8V/D < 300 \text{ s}^{-1}$.

Table 5. Chemical analysis of H-Coal PDU samples

Component	Sample 27B (wt %)	Sample 34A (wt %)	Sample 42A (wt %)	Sample 43A (wt %)
Carbon	77.9	78.7	78.4	80.3
Hydrogen	6.3	6.2	6.7	7.2
Nitrogen	0.7	0.8	0.8	0.7
Sulfur	0.3	0.4	0.5	0.4
Oxygen (by difference)	2.5	1.4	1.5	1.6
Ash	12.3	12.5	12.1	9.8
Density at 294 K, g/cm ³	1.204	1.238	1.201	1.171
Soxhlet extraction				
Pentane sol and benzene sol (oils)	54.5	52.9	59.4	67.8
Pentane insol and benzene sol (asphaltenes)	4.5	8.6	7.2	9.0
Pyridine sol and benzene insol (preasphaltenes)	4.9	5.6	4.7	0.7
Pyridine insol and benzene insol (residue)	36.1	32.9	28.7	22.5

very low (~ 0.2), while above that value the slopes are comparable to or higher than those of 27B and 34A. However, there is some question about the data below $8V/D = 300 \text{ s}^{-1}$ for 42A because of possible viscometer pressure tap plugging.

As the reaction conditions under which the four H-Coal samples were produced were very similar, the same rheological behavior would be expected.

-280

From Fig. 4, samples 27B and 34A have points at the lower shear rates, which could indicate a change in slope of the power law model. Sample 34A, for example, has a data point at approximately 130 s^{-1} , which lies significantly above the line with the 0.734 slope. Also from Fig. 4, if the data point at 78 s^{-1} were eliminated, sample 27B appears to exhibit a change in slope at the lower shear rates. Additional data would be desirable in order to determine if samples 27B and 34A actually do behave like samples 42A and 34A in the low shear-rate region.

As seen in Table 1, the liquid/coal weight ratio was the only processing parameter that showed significant variation for the four H-Coal samples. The gas feed rate was relatively high for sample 43A, but was essentially the same for the other three samples. There appears to be no correlation of the viscosity of the samples with the given processing conditions.

From Table 4, the chemical analyses demonstrated variations between the H-Coal samples primarily in density and the Soxhlet extraction results. The ash level of sample 43A was lower than that of the other three samples. The elemental compositions of all samples were very similar. Using the results from Table 4, there does appear to be a correlation between the apparent viscosity and density; i.e., the higher the density, the higher the viscosity. As density should be related to the amount of solids (ash plus unconverted coal) in the samples, such a correlation would appear reasonable. However, as pyridine insolubles should also be a measure of solids, the apparent viscosity would also be expected to correlate with this parameter, which does not seem to be the case. If, though, the pyridine insolubles result from sample 27B was in error and

was actually close to that of sample 42A, then a correlation would exist. It is quite conceivable that this analytical result for sample 27B could be high, as indicated by the ash level, which should also be a measure of solids. Even though there is not much variation in each level between samples 27B, 34A, and 42A, the apparent viscosities do appear to increase with increasing ash level.

5. REFERENCES

1. *Fossil Energy Program Quarterly Progress Report for the Period Ending September 30, 1979*, ORNL-5612 (January 1980).
2. A. H. P. Skelland, *Non-Newtonian Flow and Heat Transfer*, Wiley, New York, 1967, pp. 28-38.

APPENDIX E

CALCULATION:

FORCE TO SUSPEND A CATALYST PARTICLE

APPENDIX E

CALCULATION:
FORCE TO SUSPEND A CATALYST PARTICLE

Extreme Case: No Impact from Other Particles:

By a Force Balance Shear Force + Buoyancy Force = Weight.

$$\tau_w \cdot \pi d L + 2 \frac{\pi d^2}{4} + \frac{\pi d^2 L}{4} \rho_{sl} g = \frac{\pi d^2 L}{4} \rho_p g$$

Values used for calculation are listed in Table E-I.

$$\begin{aligned} \tau_w &= 20.41 \frac{\text{gm-cm}}{\text{sec}^2} / \text{cm}^2 \\ &= 20.41 \text{ dyne/cm}^2 \\ &= 2.04 \text{ N/m}^2 \end{aligned}$$

By contrast, yield stresses measured by Battelle and ORNL are ~ 0.2 .

The shear stresses predicted using the Bingham plastic model are expressed:

$$\tau_w = \tau_o + \dot{\gamma} \eta_{pl}$$

For the slurries tested:

$$\begin{aligned} \tau_o &= 0.2 \text{ N/m}^2 \\ \eta_{pl} &\approx 2.5 \text{ cp} \\ &= 2.5 \cdot 10^{-2} \text{ gm/cm-sec} = 2.5 \cdot 10^{-3} \text{ kg/m-sec} \\ &= 2.5 \cdot 10^{-3} \frac{\text{N-sec}}{\text{m}^2} \end{aligned}$$

$$\text{Then } 2.0 \text{ N/m}^2 \approx 0.2 \text{ N/m}^2 + 2.5 \times 10^{-3} \dot{\gamma}$$

$$\dot{\gamma} = 720 \text{ sec}^{-1}$$

For the PDU tests, this value was reduced to 450 sec^{-1} to account for the buoyant effect of the gas. A pseudo-viscosity was then calculated:

$$\eta^* \equiv \frac{\tau}{\dot{\gamma}} = \eta_{pl} + \frac{\tau_0}{\dot{\gamma}}$$

TABLE E-I

d	Particle diameter (1)	0.140 cm
L	Particle length (1)	0.332 cm
ρ_{sl}	Slurry density (2)	0.93 g/cm ³
ρ_p	Soaked Particle Density (2)	1.65 g/cm ³
τ_w	Shear stress, N/m ²	--
g	Gravitational acceleration	980.7 cm/sec ²
$\dot{\gamma}$	Shear rate, sec ⁻¹	
τ_o	Yield stress, N/m ²	
η_{pl}	Plastic viscosity, cp	
η^*	Pseudo-viscosity, cp	

(1) Table 28, Reference 2.

(2) Nominal value--Table XIX.

(3) Nominal value--Table XVIII.

APPENDIX F

I/O DATA FOR PDU-10 FLUID DYNAMIC TESTS

FIGURE F - 1

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 01

FIGURE F - 2

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 02

FIGURE F - 3

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 03

FIGURE F - 4.

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 04

FIGURE F - 5

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 05

FIGURE F - 6

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 06

FIGURE F - 7

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 07

FIGURE F - 6

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 08

FIGURE F - 9

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 09

FIGURE F -10

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 10

FIGURE F -11

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 11

FIGURE F -12

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 12

FIGURE F -13

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 13

FIGURE F -14

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 14.

FIGURE F -15

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 15

FIGURE F -16

PDU-10 GAMMA RAY SCAN I/I0 RATIO, TEST NUMBER 16

APPENDIX G

PREDICTION OF RICHARDSON-ZAKI PARAMETERS
FOR PDU EXPERIMENTAL MODELING

APPENDIX G

PREDICTION OF RICHARDSON-ZAKI PARAMETERS FOR PDU EXPERIMENTAL MODELING

BED EXPANSION OF CYLINDRICAL CATALYSTS IN LIQUID FLUIDIZATION

ABSTRACT

Fluidization of cylindrical particles by pure fluids and by coal slurries has been studied. It is shown that bed expansion data of cylindrical-shaped particles in liquid fluidization are adequately correlated using the Richardson-Zaki method. Particle terminal velocities are shown to be well predicted using the drag coefficient--Reynolds number relationship for infinite cylinders, with the characteristic dimension given by the cylinder diameter. The original correlations suggested by Richardson and Zaki for their index, n , in terms of particle Reynolds number are shown to be satisfactory.

INTRODUCTION

Nearly all gas-solid and liquid-solid fluidization data have been obtained in beds of spherical or near-spherical particles. Fluidization data for cylindrically shaped pellets are scarce. Table I lists only four sources where these kinds of data are available. In view of the recent interest in the catalytic hydroliquefaction of coal (the H-Coal process) and the hydroprocessing of petroleum resids, a better understanding of the fundamentals of ebullated (or three-phase fluidized) beds is essential for successful scale-up. The commercial catalysts used in these processes are nearly all in the form of cylindrical extrudates. The prediction of the hydrodynamic behavior of a three-phase fluidized bed from basic fluid and particle mechanics is not possible at present. This applied even to basic parameters such as bed expansion and phase holdup. At least two phenomenological models have been proposed: namely, those of Bhatia and Epstein (1), and

Darton and Harrison (5). However, their correlations for gas holdups were borrowed entirely from the two-phase literature, and a more definitive analysis seems necessary. As for the treatment of the particulate phase (i.e., phase excluding the bubble and its associated wake), both models suggest the use of the Richardson-Zaki correlation (8). Thus it is crucial to determine how well the Richardson-Zaki correlation can predict liquid fluidized bed behavior of cylindrical particles. For such a study, Vasalos et al (12) found that the data could be correlated by the Richardson-Zaki type of correlation, but the two parameters (i.e., terminal velocity and index n) in the Richardson-Zaki correlation were fit empirically using the data. The present study is an extension of Vasalos' work. In addition, methods are suggested for determining the two parameters in the Richardson-Zaki correlation.

DISCUSSION

The Richardson-Zaki correlation (8) has been widely used to correlate gas-solid and liquid-solid fluidization data. Basically, the model relates the liquid holdup, ϵ_1 , to the superficial liquid velocity, j_1 , and the terminal velocity of the solid, U_t :

$$\epsilon_1^n = j_1/U_t \quad (1)$$

where n is the so-called Richardson-Zaki index. Richardson and Zaki used the drag-coefficient relationship to estimate U_t and proposed to correlate n as a function of particle Reynolds number.

Usually the settled bed porosity, ϵ_{10} , and bed height, H_0 , are known, and the material balance on the solids yields:

$$(1 - \epsilon_1)H = (1 - \epsilon_{10})H_0 \quad (2)$$

Thus, bed expansion can be predicted once the liquid holdup inside the bed is found using Equation 1.

Terminal Velocity of a Cylindrical Particle

The terminal velocity of a particle falling in a fluid is determined by balancing the gravitational and drag forces. For spherical particles of diameter, d , we have:

$$\pi d^3 g (\rho_s - \rho_1)/6 = C_D (\pi d^2/4) (\rho_1 U_t^2/2) \quad (3)$$

where the nomenclature has the usual meaning. The drag coefficient, C_D , is a function of the particle Reynolds number, Re_t , defined by:

$$Re_t = \rho_1 U_t d / \mu_1 \quad (4)$$

For example, the drag coefficients for spheres, disks, and cylinders can be found in Perry (10) and are shown in Figure 1. In order to calculate

the terminal velocity, one has to resort to trial and error using Figure 1. However, one can simplify Equation 3 and replace U_t with Re_t , thus obtaining:

$$C_D Re_t^2 = 4/3 \left[d^3 \rho_s g (\rho_s - \rho_l) / \mu_l^2 \right] = (4/3) Ga, \text{ for spheres} \quad (5)$$

where Ga is the Galileo number and can be evaluated solely from known quantities. Since C_D is a function of Re_t , $C_D Re_t^2$ can be expressed analytically or graphically as a function of Re_t . The resulting graphical relationship based on Figure 1 is shown in Figure 2 for all three shapes, and therefore the Reynolds number, Re_t , and hence terminal velocity, U_t , can be derived.

Terminal velocity measurements were made by dropping single HDS-2A (American Cyanamid) catalyst extrudates in various fluids. Since the catalyst is porous in nature, it was thoroughly soaked in the particular fluid studied before testing. The soaked catalyst density was then determined by simple volume displacement and weighing methods. The results are summarized in Table II. Experimentally, the catalyst was observed to fall with its axis horizontal, even in cases where it was released from the vertical position. The following methods were used to correlate the data.

Method A--The pellet diameter, d , was used as the characteristic length. Here, a similar relationship can be derived between $C_D Re_t^2$ and the Galileo number; namely:

$$C_D Re_t^2 = (\pi/2) Ga, \text{ for cylinders} \quad (6)$$

The experimental data are plotted in Figure 3, where the data exhibited good agreement with the infinite cylinder C_D vs. Re_t relationship.

Method B--For this calculation of the dimensionless groups (Re_t , Ga), the equivalent particle diameter, d_s , (defined as the diameter of a sphere having the same volume) is used. For a cylindrical pellet:

$$d_s = (3d^2 \ell / 2)^{1/3} \quad (7)$$

The results shown in Figure 4 indicate that the data lie significantly below the sphere relationship; hence, it is concluded that d_s is not a suitable dimension to be used. Note that this is the characteristic diameter used by Vasalos et al (12) and their proposed correlation is also shown in Figure 4 for comparison. It is not surprising that the Vasalos et al correlation fit the data in this form quite well because d_s was used to calculate these data points.

Method C--Heywood (7) suggested using the projected diameter, d_h , equal to the diameter of the circle, having an area equal to the projected area of the particle when placed in the most stable position. In the present case, d_h is given by:

$$d_h = (4d \ell / \pi)^{1/2} \quad (8)$$

The terminal velocity data reduced by Heywood's approach are presented in Figure 5. The agreement with the sphere C_D vs Re_t relation is poor.

Method D--In packed column studies, the usual mean particle diameter, d_m , (see Bird, et al, 1960) is given by:

$$d_m = 6/a_v \quad (9)$$

where the specific surface, a_v , is the ratio of particle surface area to particle volume. It can easily be shown that d_m is related to d_s via sphericity, ϕ_s ; i.e.:

$$d_m = \phi_s d_s \quad (10)$$

where the sphericity is defined as:

$$\phi_s = (\text{surface area of sphere/surface area of particle, both of the same volume})$$

and hence for a cylindrical-shaped particle

$$\phi_s = (3/2 d^2 L)^{2/3} / [(d^2/2) + dL] \quad (11)$$

The terminal velocity data using d_m as the characteristic length are shown in Figure 6. As a whole, the drag relationship for a sphere correlates the data very well at Reynolds numbers less than 100, but tends to overestimate the terminal velocity at higher Reynolds numbers.

Among the four methods tried, the best correlation is obtained by using the pellet diameter as the characteristic length in conjunction with the C_D vs Re relationship for infinite cylinders. This analysis demonstrates that the drag coefficient for finite cylinders of L/d equal to 3 approaches closely to that for infinite cylinders.

Bed Expansion

According to the Richardson-Zaki correlation, bed expansion data can be reduced on a plot of $\log j_1$ versus $\log \epsilon_1$. A straight line can be drawn through the data:

$$\log j_1 = \log U_t + n \log \epsilon_1 \quad (12)$$

The intercept at $\epsilon_1 = 1$ yields $\log U_t$ and the slope of the line gives the value for n . All the available data were reduced in this fashion. Liquid holdup was evaluated based on the expanded bed height. In the case of H-Coal AU-77H data (11), the kerosene/coal fines slurry holdup was obtained from bed height measurements using a travelling gamma-ray densitometer. All of the results are summarized in Table III, and the corresponding Richardson-Zaki plots can be found in Appendix A. As a whole, the data were found to be well correlated by a straight line on these plots, thus confirming the validity of the form of the correlation proposed by Richardson and Zaki.

Viscosity

For pure liquids such as kerosene and mineral oil, the liquid viscosity, μ_o , can be easily measured or obtained from physical property tables. However, for the kerosene/coal fines slurry employed in the H-Coal modeling study, the mixture (effective) viscosity, μ_m , is dependent on the solid content. In general, it was found (6) that the relative viscosity (μ_m/μ_o) is a function of the solid volume fraction. The mixture viscosity data (11) for kerosene slurry were so reduced, as shown in Figure 7, and a best linear fit was found:

$$\mu_m = \mu_o (1 + 26.8 \epsilon_s) \quad (13)$$

The dimensionless numbers in Table III for the kerosene/coal fines slurry fluidization data were all evaluated using Equation 13 for the viscosity values.

Terminal Velocity

The terminal velocity data extrapolated using the Richardson-Zaki method were plotted as terminal velocity Reynolds number versus the Galileo number. Figure 8 shows that on a plot of Re_t versus Ga using d as the characteristic length, the terminal velocity data can be well correlated by the drag coefficient relationship for infinite cylinders. However, the mineral oil data from the H-Coal AU-77H unit were found to lie significantly lower than other data in Figure 8. One possible explanation is that the catalyst was not completely soaked before the mineral oil experiments were conducted. Table IV compares the predicted terminal velocities with the experimental terminal velocities from Richardson-Zaki plots. The overall mean ratio of predicted value to observed value is 0.967, although the mean ratio for mineral oil data is only 0.737. This analysis demonstrates that the terminal velocities for cylindrical pellets in a fluidized bed can be predicted using the C_D vs Re_t relationship for infinite cylinders with the characteristic dimension given by the pellet diameter.

Correlation of Index, n

Richardson and Zaki found the index, n , to be a function of the particle Reynolds number as follows:

$$\begin{aligned} Re < 0.2 \quad n &= 4.65 \\ 0.2 < Re < 1 \quad n &= 4.35 Re^{-0.03} \\ 1 < Re < 500 \quad n &= 4.45 Re^{-0.1} \\ 500 < Re \quad n &= 2.39 \end{aligned} \quad (14)$$

However, this relationship was obtained for spherical particles only. Values of n for the present data are plotted in Figure 9 as a function of Galileo number. Since a unique relationship exists between Reynolds number and Galileo number for spherical particles, Equation 14 can easily

be transformed to yield n as a function of Ga . The resulting curve as shown in Figure 9 is seen to slightly overpredict the value of n . Values of n are also presented as a function of particle Reynolds number as shown in Figure 10. The original Richardson-Zaki correlation for n (Equation 14) is seen to correlate the data slightly better in terms of Reynolds number. However, more data are needed to confirm the validity of this correlation for cylindrical particles, particularly in the laminar ($Re < 1$) and turbulent ($Re > 1000$) regimes.

Richardson and Jeronimo (9) have also recently investigated methods of estimating n . One method that appears quite promising is to assume Equation 1 to be valid at both the minimum fluidization point and the fully expanded bed of voidage unity. The minimum fluidization velocity, U_{mf} , was estimated using the Ergun equation (2) for packed beds. The value of n is hence given by:

$$n = \frac{\log U_{mf} / U_t}{\log \epsilon_{mf}} \quad (15)$$

where ϵ_{mf} is the voidage at minimum fluidization. Their results indicate that values of n calculated using Equation 15 and the Ergun equation are considerably higher than the original Richardson-Zaki values, i.e., Equation 14. Richardson and Jeronimo attributed the discrepancy to the validity of using Ergun's equation for a fluidized bed and the uncertainty of choosing the value for ϵ_{mf} . We feel that the Richardson-Zaki correlation (Equation 1) does not apply near the minimum fluidization point and the original experimental data of Richardson and Zaki tend to support this observation. Richardson and Jeronimo's data also show that the Richardson-Zaki approach does not apply at the minimum fluidization point, which resulted in the larger values of n being obtained. Thus, Equation 15 is not recommended as a method for evaluation of n . Equation 14 is the best available at the present time.

CONCLUSIONS

- 1) The Richardson-Zaki type correlation can be used to analyze bed expansion data of cylindrical-shaped particles in both pure liquid and coal slurry fluidization.
- 2) The terminal velocity values obtained from both single cylindrical catalyst drop experiments and liquid fluidized bed experiments can be adequately correlated using the C_D versus Re_t relationship for infinite cylinders with the characteristic dimension given by the pellet diameter. Three other characteristic dimensions were tried in attempting to correlate the single pellet terminal velocity data, but none was found to be satisfactory.
- 3) The Richardson-Zaki index, n , obtained from data used in this study is presented both as a function of the Galileo number and the Reynolds number. The original Richardson-Zaki correlation for index n is found to correlate present data better in terms of Reynolds number. More data are required, however, to confirm the validity of this correlation at both low and high Reynolds number.

REFERENCES FOR APPENDIX G

- G-1) Bhatia, V. K., and N. Epstein, "Three-Phase Fluidization: A Generalized Wake Model," Proc Euro Symp on Fluidization, 380 (1974).
- G-2) Bird, R. B., et al., Transport Phenomena, John Wiley and Sons (New York) (1960).
- G-3) Chemical Systems, Incorporated, "Liquid Phase Methanation," R&D NPT No. 78, Interim Report No. 1 (1973).
- G-4) Darton, R. C., and D. Harrison, "Gas and Liquid Holdup in Three-Phase Fluidization," Chem Eng Sci, 30, 581 (1975).
- G-5) Happel, J., and H. Brenner, Low Reynolds Number Hydrodynamics with Special Applications to Particulate Media, Chapter 9, Prentice-Hall (New Jersey) (1965).
- G-6) Heywood, H., "Uniform and Non-uniform Motion of Particles in Fluids," Sym Interaction Between Fluids and Particles, Inst Chem Eng (London) (1962).
- G-7) Richardson, J. F., and W. N. Zaki, "Sedimentation and Fluidization: Part I," Trans Inst Chem Eng, 32, 35 (1954).
- G-8) Richardson, J. F., and M. Jeronimo, "Velocity-Voidage Relations for Sedimentation and Fluidization," Chem Eng Sci, 34, 1419 (1979).
- G-9) Perry, J. H., Chemical Engineers' Handbook, Fourth Edition, McGraw-Hill (New York) (1963).
- G-10) Vasalos, I. A., et al., "Study of Ebullated Bed Fluid Dynamics for H-Coal," Final Report, M80-21 (1980).
- G-11) Vasalos, I. A., et al., "Holdup Correlations in Slurry Solid Fluidized Beds," AIChE J, 28, 346 (1982).
- G-12) Wolk, R., "Gas-Liquid-Solid Fluidization," MS Thesis, Polytechnic Institute of Brooklyn (1962).

TABLE I

LIQUID FLUIDIZATION DATA FOR CYLINDRICAL CATALYSTS

<u>Source</u>	<u>Catalyst</u>	<u>Fluid</u>	<u>Test Column Diameter (cm)</u>
Amoco Oil (Vasalos, 1980)	HDS-2A d = 0.16 cm l = 0.51 cm	Kerosene/Coal Fines Slurry Mineral Oil (Amoco 9 NF)	15.2
HRI (Wolk, 1962)	Nalcomo 471 d = 0.079 cm l = 0.48 cm	2-Propanol	2.5
Chem Systems, Inc. (1973)	Methanation Catalyst d = 0.16 cm l = 0.32 cm	Hexane	2.2

JCL/ml
9/28/81

TABLE II

SUMMARY OF TERMINAL VELOCITY MEASUREMENTS OF INDIVIDUAL CYLINDRICAL PELLET

<u>Pellet</u>	<u>Particle Diameter, cm</u>	<u>Particle Length, cm (1)</u>	<u>Fluid</u>	<u>Temp, °C</u>	<u>Fluid Density g/cm³</u>	<u>Viscosity, cP</u>	<u>Soaked Density, g/cm³</u>	<u>Measured (2) Terminal Velocity, cm/s</u>
HDS-2A	0.16	0.5	Water	20	1.0	1.0	1.79	14.4
HDS-2A	0.16	0.5	Mineral Oil	66	0.82	6.2	1.61	11.9
HDS-2A	0.16	0.5	Mineral Oil	79	0.81	4.2	1.61	13.0
HDS-2A	0.16	0.5	Kerosene	20	0.79	1.39	1.68	16.4, 17.0
HDS-2A	0.16	0.5	Ethylene Glycol	20	1.11	19.9	1.76	6.0
Glass Rod	0.20	0.5	Ethylene Glycol	20	1.11	19.9	2.23	10.8

(1) All Lengths are nominal values.

(2) At least 20 particles were dropped and the velocities reported are statistical averages.

JCL/ml
9/28/81

TABLE III

TABULATION OF RICHARDSON-ZAKI PARAMETERS FROM LIQUID FLUIDIZATION DATA

Source	Catalyst	Extrudate Diameter (cm)	L/d	Run No.	Fluid	Coal Fines Conc (Vol%)	Temp °C	Soaked ρ (Cat.) g/cm ³	ρ (Slry) g/cm ³	Viscosity (cP)	U_t (cm/s)	Index n	Re_t	Ga
Amoco Oil (Vasalos, 1980)	HDS-2A	0.16	3.2	201 (1)	Kerosene	0	21	1.64	0.79	1.39	14.0	2.66	127	13,970
				301	Kerosene	0	22	1.64	0.79	1.39	15.0	2.64	136	13,970
				204	Kerosene	5.1	21	1.64	0.84	3.34	13.8	3.0	55	2,420
				205	Kerosene	10.4	19	1.64	0.89	5.28	12.5	3.0	34	960
				210	Kerosene	11.9	22	1.64	0.90	5.84	10.7	2.89	26	790
				209	Kerosene	15.5	36	1.64	0.93	6.40	10.2	3.05	24	650
				208,	Kerosene	15.5	66	1.63	0.91	4.58	11.0	2.81	35	1,250
				214										
				420,	Mineral Oil	0	37	1.68	0.85	14.6	7.1	3.58	6.6	130
				427										
				421,	Mineral Oil	0	52	1.67	0.83	9.5	8.2	3.28	12	310
				428										
				422,	Mineral Oil	0	61	1.66	0.82	6.0	9.0	2.94	20	800
				426										
				423,	Mineral Oil	0	79	1.66	0.81	4.2	10.3	2.83	32	1,570
				429										
Chem Sys, Inc.	Standard Methanation Catalyst	0.16	2.0	46-1	Hexane	0	20	2.25	0.66	0.3	25.0	2.6	868	3.67×10^5
HRI (Wolk, 1962)	Nalcomo 471	0.079	6.0	--	2-Propanol	0	20	1.42	0.79	2.5	9.6	2.81	24	391

(1) Run number corresponding to original report.

(2) This run was conducted in a 15.2 cm ID column.

TABLE IV

TERMINAL VELOCITY PREDICTIONS FOR EED EXPANSION DATA

-314

<u>Source</u>	<u>Fluid</u>	<u>Run ID</u>	<u>Ga</u>	<u>Expt U_t</u> (cm/s)	<u>Pred U_t</u> (cm/s)	<u>Pred U_t /</u> <u>Expt U_t</u>	
Amoco Oil (Vasalos)	Kerosene Slurry	201	13,970	14.0	15.3	0.915	} $\bar{x} = 0.966$ (1) $\sigma = 0.041$
		301	13,970	15.0	15.3	0.980	
		204	2,420	13.8	13.4	1.030	
		205	960	12.5	12.4	1.008	
		210	790	10.7	11.4	0.939	
		209	650	10.2	10.8	0.944	
		208,214	1,250	11.0	11.6	0.948	
	Mineral Oil	420,427	130	7.1	9.5	0.747	} $\bar{x} = 0.737$ $\sigma = 0.024$
		421,428	310	8.2	11.1	0.739	
		422,426	800	9.0	12.8	0.703	
		423,429	1,570	10.3	13.6	0.757	
Chem Sys	Hexane	46-1	3.67×10^5	25.0	24.8	1.008	
HRI (Wolk)	n-Proparol	--	391	9.6	7.2	1.333	

(1) $\bar{x}$ = mean; σ = standard deviation.

Figure 1

DRAW COEFFICIENTS FOR SPHERES, DISKS AND CYLINDERS (from Perry (10))

Figure 2

$C_D Re_t^2$ CORRELATION FOR SPHERES, DISKS AND CYLINDERS

Figure 7

SLURRY VISCOSITY CORRELATION

46 1513

HDS-2A Cat

Resid Cat

● Kerosene slurry

□ Kerosene

△ Mineral oil

Figure 9

EXPERIMENTAL VALUES OF n AS A FUNCTION OF
GALILEO NUMBER

LEGEND
● Kerosene slurry } Alkyl (Naphthalene)
△ Mineral oil } Alkyl (Naphthalene)
□ Kerosene } Alkyl (Naphthalene)
○ Kerosene } Alkyl (Naphthalene)
* Kerosene } Alkyl (Naphthalene)

Richardson-Zahl correlation for
spheres

46 1513

Figure 10
EXPERIMENTAL VALUES OF n AS A FUNCTION OF
REYNOLDS NUMBER

LEGEND

- KENNEDY, STAINING, AMMONIA (1954)
- △ MURPHY, OIL, AMMONIA (1954)
- KENNEDY, AMMONIA (1954)
- HENNING, CHEM. SYS. INT.
- M. MURPHY, AIR (1954)

Richardson-Zaki correlation (1954)
for spheres

APPENDIX

RICHARDSON-ZAKI PLOTS OF BED EXPANSION DATA

The Richardson-Zaki plots for the bed expansion data are presented in this appendix.

u_t	n	PAN
15	2.64	310
14	2.66	201
13.8	3.0	204
12.5	3.0	205

RUN	FINE	
• 201	0.7	72%
x 310	0.7	74
○ 203	1.7	68
<hr/>		
Δ 204	5.1%	70
□ 205	10.4%	67

▲ RUN 208

○ RUN 214
15.5% 150°F

u_t n
11.0 2.81

VCL 9/12/81

u_t 25 cm/s
 n 2.6

$$u_t = 7.6 \frac{\text{cm}}{\text{s}}$$

$$n = 2.31$$