Energy

FOSSIL

150
19/9/84
mjt
① DR-0468-5

DOE/OR/03054-5-Suppl.
(DE84015103)

SRC-I QUARTERLY TECHNICAL REPORT

Supplement for the Period July–September 1981

Work Performed Under Contract No. AC05-78OR03054

International Coal Refining Company
Allentown, Pennsylvania

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 A03
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.

DOE/OR/03054-5-Suppl.
(DE84015103)
Distribution Category UC-89

SRC-I
QUARTERLY TECHNICAL REPORT
SUPPLEMENT
JULY—SEPTEMBER 1981

International Coal Refining Company
Allentown, Pennsylvania

TABLE OF CONTENTS

	<u>Page no.</u>
EFFECT OF FEED COAL VARIATION ON DEMONSTRATION PLANT PERFORMANCE D. S. Hoover, D. Kang, E. N. Givens, A. Bland, B. Davis, J. Hower, D. Koppelaar, and D. Pollock	1
LARGE-SCALE DISSOLVER COLD-FLOW MODELLING W. T. McDermott and D. H. Ying	21
IMPACT OF SRC RECYCLE ON COMMERCIAL DESIGN D. Kang and E. N. Givens	37
WILSONVILLE TECHNICAL SUPPORT PLAN S. C. Weiner	55

Note

Articles in this Supplement were previously withheld from publication to avoid premature disclosure of inventions. One article was published prior to June 1984 and is not republished here. The article on page 1 was included in the January-March 1982 Quarterly Technical Report (DOE/OR/03054-7). Pagination has not been altered, since this could invalidate any cross-references.

LARGE-SCALE DISSOLVER COLD-FLOW MODELING

W. T. McDermott and D. H. Ying*

The coal liquefaction dissolver column will be designed so that the gas/liquid mass-transfer rate will not control the rate of hydrogen consumption. Experimental evidence from the Wilsonville Pilot Plant and theoretical predictions developed from cold-flow-derived correlations suggest that hydrogen consumption will not be controlled by the gas/liquid transfer rate. In addition, the mass transfer coefficient should be measured directly in a large vessel designed comparably to the dissolver, which will considerably increase confidence in the scale-up design. APCI will test the design for solids removal to control solids loading in the laboratory reactor. In addition, the demonstration plant dissolver design includes a large, (2-ft-diameter) single inlet that will be tested for the first time.

For this simulation study, a 1-ft-diameter inlet to a 6-ft diameter x 25-ft-tall metal column has been constructed. Seven prospective column internal configurations to disperse the gas phase and to generate fine bubbles will be tested.

The piping layout simulates the interstage from the first dissolver to the second dissolver in series. A transparent section will be installed in this section in order to see the gas/slurry flow pattern. Also a U-shaped, 180-degree turn incorporating a transparent horizontal pipe section will be included so that this section can be observed.

During this reporting period, researchers at APCI constructed a 3-in.-i.d. Plexiglas piping system to investigate the dynamic characteristics of flow through the inlet piping leading to the column. To ensure a structurally safe design, researchers determined the approxi-

*Air Products and Chemicals, Inc., APCI.

mate intensity of impulses resulting from liquid slugs as they impacted upon bends in the inlet to the column piping system.

The objective of studying the rate of gas/liquid mass transfer and solids removal in a cold-flow tubular column is to provide data for designing the coal dissolver in the SRC-I Demonstration Plant. Other areas investigated included: studying the rate of gas/liquid mass transfer for several internal designs by using a two-phase system (air/water); examining the effect of solids on the rate of gas/liquid mass transfer; and testing the design of a solids removal system.

The dissolver plays a major role in coal dissolution for any liquefaction plant. Although most liquefaction occurs in the preheater, many necessary chemical changes occur in the dissolver, such as sulfur removal, oil and distillate formation, and solvent rehydrogenation.

To evaluate specific performance of a commercial-scale dissolver column, a large-scale (6-ft-diameter), cold-flow study is necessary. Currently, APCI is researching smaller scale (1-ft-diameter), gas/slurry flow in coal liquefaction processes under a different program (Ying, et al., 1980). The large-scale, cold-flow modeling program addresses specific questions of dissolver scale-up that are directly related to the SRC-I Demonstration Plant but are not covered under the smaller-scale program.

The coefficient for gas/liquid mass transfer, $k_L a$, should be examined in an industrial-size dissolver column. Results from studies at the Wilsonville Pilot Plant show that chemical reaction rather than gas/liquid mass transfer is the rate-limiting step for hydrogen consumption. Results from the APCI research program have correlated with those reported in literature for a two-phase system. Predictions from the correlation indicate that the demonstration plant dissolver should not be limited by gas/liquid mass transfer, even when the transfer coefficient is substantially reduced in the presence of solid phase. Recently, Kataska et al. (1979) studied gas/liquid mass transfer in an 18-ft-diameter vessel; their results confirm the correlation for two-phase systems.

However, the demonstration-scale dissolver design uses a 2-ft-diameter inlet that is more than 20 times larger than any inlet opening

tested. Conceivably, large initial bubbles may occur. To what extent the large entrance will influence the overall mass transfer coefficient is an important question in the dissolver design. Directly measuring $k_L a$ in a large-scale column with a large inlet will provide data necessary for designing the dissolver column.

Efficient solids removal is also essential to ensure an effective design for the demonstration plant dissolver. Tremendous solids accumulation has occurred in the dissolver at Wilsonville; therefore, a solids removal system is required to control solids loading in the demonstration-scale dissolver. Large particles should be removed, but the fines should remain, because they may be catalytically active. Because the amount of accumulation varies with column dimensions as well as with rates of gas and slurry flow, evaluating the removal system in a large-scale dissolver column will provide necessary data for confirming or improving the design.

PROGRAM OF EXPERIMENTATION

To ensure successful scale-up to an 11-ft-diameter column--as proposed for the Demonstration Plant--APCI will test the performance of a column 6 ft in diameter and 25 ft high. The results should verify the design for the SRC-I dissolver to extrapolate currently available data to a much larger column.

Equipment Preparation

To execute the entire experiment, equipment and piping layout are being modified for the 6-ft-diameter, cold-flow simulator. Sampling equipment has been designed to extract one- and two-phase samples from several depths in the column.

APCI researchers will also examine the performance of a distributor plate, part of the internal design for the dissolver, that was fabricated according to an ICRC design. For two-phase flow separation, extended risers were constructed and attached on site to the distributor plate. Other internals have been designed so that, with minor on-site modifications, the equipment will simulate all proposed design con-

figurations. This equipment includes gas spargers, a bubble cap, and 1- and 2-ft-diameter target plates to disperse large bubbles.

Other equipment provided for these studies included a slurry-pumping, gas-distribution system and a holding tank for air/slurry experiments. Currently available equipment requires extensive modifications. For example, completely new interconnecting piping must be installed to simulate the interstage from the first to the second dissolver in series. In addition, an air compressor with suitable capacity has been obtained for the experiments.

Gas Liquid Contact

Experiments to determine the extent of gas/liquid contacting will be performed without any internals in the column and with previously mentioned design configurations. Each set of experiments with a particular design configuration will be followed by sufficient time to modify equipment for the next design.

Superficial gas velocities of up to 0.5 ft/sec and liquid velocities of up to 0.05 ft/sec will be investigated to explore the full range of operating velocities within the SRC dissolver column.

Air/Water Measurements (Two-Phase System)

Gas-phase holdup. Liquid displacement method will be used to measure the difference between liquid levels with and without air bubbling through the system. The ratio of this difference to the aerated liquid height represents the overall gas holdup.

Gas/liquid contact. At the entrance and throughout the column, gas/liquid contact will be observed and photographed when desirable.

Liquid surface. To estimate bubble size and the frequency of slug formation, liquid surface will be observed. If necessary, high-speed cinefilm will be used.

Gas/liquid, mass-transfer coefficient. Mass-transfer will be measured with oxygen probes in desaturated water to plot the rate at which oxygen saturates the column contents. Nitrogen sparged initially into the column at low flow rates will desaturate the water of oxygen. To determine the degree of uniformity in oxygen saturation throughout

the column, researchers will record simultaneous oxygen concentrations at several vertical levels in the column.

Liquid axial-dispersion coefficient. After injecting a saline solution into the column, researchers will plot liquid conductivity as a function of time at a fixed level of the column. The resulting conductivity versus time curves are used to calculate the liquid axial-dispersion coefficient.

Ying, et al. (1980) experimented with 5-in. and 12-in.-diameter tubular columns and found that, within the range of velocities investigated, liquid velocity only slightly affects gas-phase holdup, gas/liquid mass transfer, and liquid axial-dispersion coefficient. Consequently, the best design configuration will be determined based upon two-phase studies using batch operation and zero liquid velocity.

For a three-phase flow system (air/water/sand), gas holdup and mass transfer will be measured as in the two-phase system. In this system, overall mixing at the column bottom will be studied using fiber optics. A series of solids concentration measurements taken along the length of the column will determine solids dispersion throughout the column. Stagnation zones will also be investigated.

Solids Removal Test

Under a different program, limited examination of solids removal will be made. Based on results from this study and the system designed by ICRC, a solids removal system will be installed in the 6-ft, cold-flow column. While formulating this program, researchers prepared a list of specific data requirements and the sequence of experimentation.

The experiments are designed to determine the effect of the rates of solids and slurry removal upon system stability and wt % of solids withdrawn. Operating conditions are: batch operation and internal liquid flow; 8 and 16 wt % solids concentrations of sand; superficial gas velocities up to 0.5 ft/sec; and varied slurry withdrawal rates.

TECHNICAL PROGRESS

Designing the System

The piping has been designed so that gas/liquid contact can be studied when only gas and liquids are flowing or while gas, liquid, and solids are flowing simultaneously. The system is designed for batch operation and for internal liquid flow. Air is distributed through a manifold to instrument, operating, and to process lines. One of the air process lines that simulates the gas feed into the dissolver joins with the water/sand slurry line in a 12-in.-diameter pipe before entering the column. The other air supply line goes directly into the column through a gas sparger system. Nitrogen can be sparged through the system to desaturate the water of oxygen, so that the uptake rate of oxygen from air into water can be measured. This rate is used to calculate the gas/liquid mass-transfer rate.

The design of the 12-in.-diameter column feed pipe simulates the interstage from the first to the second dissolver in series and originates from an elevated section where the slurry exits the column. The pipe turns 90 degrees, dropping to where gas/slurry flows through a 15-ft horizontal section before it enters the column at the bottom. Part of this section is made of a 12-in.-diameter Plexiglas pipe, so that the gas/slurry flow pattern can be observed before entering the column. Part of the feed pipe to the lower level is transparent so that the downflow pattern can be studied.

Preliminary Investigations

To understand the dynamic characteristics of flow through the inlet pipe to the column, a separate 3-in.-i.d., transparent piping system was arranged (Figure 1). This system uses approximately 55 ft of piping to recirculate liquid, incorporating 90- and 180-degree bends. In addition, this piping separately regulates and meters air and liquid flow rates. Air is introduced to the flow stream at the beginning of the loop through a tee section. A rotameter measures the air velocity; a Doppler flowmeter measures the liquid velocity.

These studies are designed to identify possible slug flow in the column inlet piping. Slug flow is characterized by large, periodic gas bubbles that can significantly affect dissolver performance. In addition, this study will determine the frequency and approximate intensity of rapidly moving liquid slugs as they impact upon bends in the piping system. Consequently, these data will possibly improve the design for interconnecting piping and structural support.

Experimental variables in this study include liquid viscosity, pipe inclination, and gas (V_G) and liquid (V_L) superficial velocities, which will vary widely and encompass values present in the column inlet piping. Experimental data will include pipe holdup measurements, photographic documentation of the character of the flow, and the approximate frequency, length, and velocity of any slugs.

The gas holdup data generated in APCI laboratory correlate very well with a dimensionless parameter (X) developed by Lockhart and Martinelli (1949). In the 3-in.-diameter piping, gas holdup was determined by simultaneously closing both the entrance and exit valves; the retained liquid was then drained and measured. In Figure 2, experimental values of gas holdup are plotted versus the dimensionless parameter (X). This parameter represents the ratio of superficial liquid pressure drop to superficial gas pressure drop. The data obtained collapse well into a single curve with slight deviation at the highest liquid viscosity tested: 400 centipoise (cP).

Researchers at APCI have found that the liquid slug velocity (V_S) can be correlated with the gas superficial velocity (V_G) and gas holdup (ϵ) through the relation $V_S = V_G/\epsilon$. Slug velocity is found by measuring the time required for the slug front to traverse a known distance. Although APCI results agree well with the work of Griffith and Wallis (1961); these results show that $V_S = V_G/\epsilon$ describes the data well only up to a V_G/ϵ value of 20 ft/sec. Nevertheless, by combining the preceding relation with the gas holdup correlation, researchers can define slug flow characteristics up to a slug velocity of 20 ft/sec (Figure 3).

One of the objectives of this program is to correlate the slug frequency with various dimensionless groups to extrapolate the data

obtained for the flow conditions and physical dimensions of a dissolver inlet line. The slug frequency can be presented in a dimensionless form uD/V_L , where u is the slug frequency and D is the pipe inside diameter. APCI found that dimensionless frequency closely depends upon the superficial liquid Reynolds number, Re , and the gas superficial Froude number, F_{RG} .

For one-degree pipe inclinations, plots of frequency parameters versus gas Froude numbers and frequency parameters versus Reynolds numbers are presented in Figures 4 and 5, respectively. In Figure 4 the distinct minimum agrees qualitatively with the theoretical results of Taitel and Dukler (1977). However, the subsequent drop of the curves is unpredicted. Researchers believe the drop results in transition from an intermittent to a stratified flow (Figure 4). Figure 5 also illustrates this drop by the sharp decline in the lower curve, which corresponds to a slightly increased gas Froude number. Nevertheless, in Figure 5, data are insufficient between Reynolds numbers 100 and 10,000 in order to confirm the three curves. Future experiments will clarify this behavior.

Figure 6 shows that, in experiments with zero-degree pipe inclination, the transition between slug (intermittent) and stratified (separated) flow agrees well with the correlation of Weisman et al. (1979) in which F_R represents the gas Froude number, $V_G/(gD)^{1/2}$. The data points were taken for 1 cP liquid viscosity. In contrast, no stratified flow was observed for one-degree pipe inclination within the range of conditions examined, although encroachment in the transition zone is suspected. Results indicate that stratified flow should not occur under anticipated fired-heater conditions. Furthermore, both dispersed bubbles and annularly dispersed liquid flow are unlikely to exist within the demonstration flow conditions.

LITERATURE CITED

Griffith, P. R. and G. B. Wallis. 1961. Two-phase slug flow. Heat Transfer. 307.

- Kataska, H., H. Takeuchi, K. NaKao, H. Hagi, T. Tadaki, T. Otake, T. Miyauchi, K. Washimi, K. Watanabe, and F. Yoshida. 1979. Mass transfer in a large bubble column. *J. Chem. Eng. Jpn.* 12(2):105.
- Lockhart, R. W. and R. C. Martinelli. 1949. Proposed correlation of data for isothermal, two-phase, two-component flow in pipes. *Chem. Eng. Prog.* 45(1):39.
- Taitel, Y. and A. E. Dukler. 1977. A model for slug frequency during gas-liquid flow in horizontal and nearly horizontal pipes. *Int. J. Multi-Phase Flow.* 3:585.
- Weisman, J., D. Duncan, J. Gibson, and T. Crawford. 1979. Effects of fluid properties and pipe diameter on two-phase flow patterns in horizontal lines. *Int. J. Multi-Phase Flow.* 5:437-462.
- Ying, D. H., R. Sivasubramanian, and E. N. Givens. 1980. Gas slurry in coal liquefaction processes. Prepared for the U.S. DOE by APCI. DOE/FE/14801/3.

Figure 1
Flow System for the Preliminary Investigations

Figure 2
Nondimensional Gas Holdup Correlation

Figure 3
Proposed Slug Velocity Correlation

Figure 4
Nondimensional Slug Frequency vs. Gas Froude Number

Figure 5
Nondimensional Slug Frequency vs. Liquid Reynolds Number

Figure 6
Points of Flow-Regime Transition for Horizontal Pipe Flow

IMPACT OF SRC RECYCLE ON COMMERCIAL DESIGN

D. Kang* and E. N. Givens*

Studies were initiated to explore the effect of SRC recycle on the SRC-I plant design to determine whether or not recycling SRC could increase solvent yield. Mixtures of Kentucky #9 coal, SRC, and solvent were processed in Air Products' coal process development unit (CPDU) under simulated demonstration plant conditions of 840°F dissolver temperature, 2,000 psig pressure, 40 min residence time, and 20,000 standard cubic feet (scf) hydrogen gas rate per ton of coal. SRC materials used in this study included both light and heavy SRC (LSRC and HSRC) that were produced from the Kerr-McGee solvent deashing unit in the Wilsonville Pilot Plant.

Results, based on a single-pass run through the CPDU, show that adding either LSRC or HSRC to the coal feed enhances oil yield from coal at the expense of SRC yield. Moreover, the presence of SRC effectively decreased the optimal process temperature below normal operating conditions. At the same process severity (oil make) during SRC recycle, hydrogen consumption decreased substantially due to suppressed light hydrocarbon gas formation. Under these milder conditions, sulfur content in the SRC products increased slightly.

After using a batch vacuum distillation still to regenerate the process-derived solvents, passing a mixture of process solvent/LSRC through the CPDU reactor simulated continuous recycle, successfully. The purpose of simulating continuous recycle was to determine whether or not single-pass-run data differ from steady-state recycle run data. When LSRC-recycle, process-derived solvents were batch recycled, ulti-

*Air Products and Chemicals, Inc. (APCI).

mate oil yields were significantly higher than those from the single-pass run. These results demonstrate the necessity for studying SRC in continuous recycle to obtain better quantitative data. In addition, sulfur content in the SRC increased slightly, compared to that in the single-pass run. As the recycle run progressed, conversion, hydrogen consumption, and light hydrocarbon gas yield remained the same.

Heavy and light SRC are strictly process-defined materials that are produced in the second-and third-stage settlers of the Kerr-McGee critical solvent deashing unit at Wilsonville. Attempts to analytically define these materials through distillation and solvent separation have been unsuccessful.

Consequently, we had hoped that recycling process-derived solvent would improve the certainty of quantitative data. Unfortunately, the data were extremely complicated due to the poorly defined composition of LSRC. Because LSRC is a mixture of distillable oils and nondistillable, molecular-weight SRC, analyses of LSRC were extremely sensitive to variations in component separation methods for determining distillable oils and other components. Therefore, better recycling techniques and separation methods are necessary to obtain a data base sufficiently accurate to model essential process variables in the ultimate process.

PROGRAM OF EXPERIMENTATION

Kentucky #9 coal from the Lafayette mine was used in process runs simulating demonstration plant conditions. The starting solvents were SRC-I from the Ft. Lewis Pilot Plant blended with 15% light SRC. After the initial charge of this mixture into the CPDU, process solvents were recovered by using a 7-gal, batch still at vacuum distillation. Recovered solvents from each pass were recycled to the slurry mixer for five subsequent passes. Recycle solvents were cut at 450°F initial boiling point (IBP) under atmospheric pressure. In contrast, the final boiling points (fbp) were maintained below 600°F pot temperature at minimum attainable vacuum, usually less than a few millimeters of mercury. Final boiling points were adjusted to cut the solvents at 900°F and 1 atm based on a pressure-temperature correlation chart.

Detailed below, process conditions for LSRC-recycle runs were similar to demonstration design conditions:

	<u>Base run</u>	<u>LSRC-recycle run</u>
Run no.	BCL-34	BCL-35
Coal type	Ky #9	Ky #9
Coal concn (wt %)	30	30
Temp (°F)	840	840
Pressure (psig)	2,000	2,000
H ₂ feed rate (M scf/ton of coal)	33	33
Feed slurry (wt %)	2.5	2.5
Nominal residence time (min)	40	40
Space rate (lb/ft ³ -hr)	106	106
Coal space rate (lb/ft ³ -hr)	32	32
LSRC-to-solvent ratio	0:100	15:85

Appendices A-1 and A-2 summarize the operating data and are available upon request. Because of time constraints, however, the base run was incomplete. Further data on feed materials and product slurry, distillation, and gas chromatography are also available upon request.

FEED MATERIALS

Coal

Used exclusively in both recycle runs (BCL34, BCL35), Kentucky #9 coal was dried, and sized to -150 mesh. Table 1 provides typical proximate and ultimate analyses of this coal.

Solvent

SRC-I recycle solvent from the pilot plant in Ft. Lewis was used during start-up. As Table 2 indicates, elemental compositions and other properties of this solvent were very similar to those in Wilsonville recycle solvent (WRS).

Ft. Lewis solvent contained, by nuclear magnetic resonance (NMR) analysis, 8.3% hydrogen (¹H), which was comprised of 32.3% aromatic

protons (H_{ar}), 33.5% alpha protons (H_{α}), and 34.2% other protons (H_o). Data for simulated distillation of this solvent showed an initial boiling point of 400°F and a final boiling point of 934°F.

Light SRC

During the material balance periods at Wilsonville in run 167, LSRC was produced from Kentucky #9 coal. Process conditions for this run were 825°F dissolver exit temperature, 38 lb/hr-ft³ of coal space rate, 43 min nominal residence time, 2,100 psig dissolver pressure, and 39,000 scf/ton of coal hydrogen gas rate. Approximately 4% LSRC was recycled to the front end of the feed/solvent stream. Table 3 shows that Wilsonville LSRC contained 6.9% hydrogen, which is typical of LSRC, but was 1.4% lower than hydrogen in Ft. Lewis solvent.

LIGHT AND HEAVY SRC

Mass spectra were based on detecting samples of 100% solvent and 76% HSRC that were volatilized in the mass spectrometer. Figure 1 describes molecular-weight distribution spectra for WRS determined at Stanford Research Institute using field ionization mass spectroscopy (FIMS). Molecular weight distribution of WRS, LSRC, and HSRC was continuous and distributed diffusely, ranging from 100 to 500 mass units for the recycle solvent (Figure 1) to 800 for the major constituents in light and heavy SRC. The peak mass distribution decreased progressively--550 for HSRC to 180 for WRS--suggesting some sequential conversion between each solvent. Molecular weight distributions for HSRC, LSRC, and WRS overlap significantly. Although all three materials share the same molecular species and homologs, the concentrations of their molecular species differ.

For these materials, the number average molecular weights measured by FIMS differed dramatically from those measured by vapor-phase osmometry (VPO). For example, although the FIMS number average molecular weight for HSRC was 526 mass units, the same parameter measured by VPO in pyridine solutions was 1,294 mass units (Table 3).

Despite its experimental difficulties, FIMS allows absolute molecular weight distribution to be measured directly. In contrast, VPO permits only indirect measurement, because it relies on the accuracy of calibration curves. Moreover, VPO assumes that the solute, or SRC, is dissolved in an infinitely diluted solvent in which the solute molecules do not physically interact. Apparently, this assumption is incorrect, for pyridine is an insufficient solvent for peptizing HSRC aggregates. Although in VPO strong physical interactions between SRC molecules lead to erroneously high absolute values for number average molecular weight, this technique correctly ranks these values for all three SRC materials.

Table 3 compares LSRC characterization data with those of HSRC produced during the same or similar conditions. Except for significantly higher amounts of hydrogen in LSRC, light and heavy SRC were grossly similar in elemental composition. For example, LSRC from run 167 contained 6.9% hydrogen, whereas HSRC from the same run contained 6.0% hydrogen. Lower amounts of hydrogen in HSRC were balanced with higher amounts of oxygen, nitrogen, and sulfur: 4.5%, 2.0%, and 1%, respectively. Corresponding amounts of heteroatoms in LSRC were 3.9%, 1.4%, and 0.9%.

For both LSRC and HSRC, NMR data showed that about one-third of the hydrogen was aromatic, and remaining hydrogen was naphthenic or aliphatic. Heavy SRC, however, contained consistently more aromatic protons than LSRC. In contrast to LSRC, HSRC contained more heteroatoms and fewer hydroxyl groups such as OH and NH, indicating relatively abundant stable ethers in HSRC.

Solvent separation data showed that LSRC is predominantly composed of oils, asphaltenes, and minor amounts of preasphaltenes. Heavy SRC was comprised mostly of asphaltenes and preasphaltenes in nearly equal amounts. Again, the major components in both LSRC and HSRC were asphaltenes; the softening point for HSRC was higher than for LSRC.

SOLVENT LSRC MIXTURE

Table 4 compares laboratory distillation data (15 plates, 10:1 reflux ratio) for the recycle solvent, LSRC, and 85% solvent/15% LSRC

mixture. When the components of solvent and LSRC were mathematically blended to a ratio of 85 to 15, the distillate portion was 84.2%, which is 6.4% less than the measured value of 90.6% for the mixture. This result indicates that fractionation achieved by distillation depends strongly upon the amount and nature of the residual bottoms material. Probably this dependency arises, because the major compounds in LSRC do not fractionate cleanly into either distillate or bottoms material. Before distillation if solvent and LSRC are separated rather than mixed, after distillation the separated fractions distribute between the distillate and bottoms fractions quite differently from the mixed fractions.

Assuming that the solvent was 100% distillable, the mixture would be calculated as 89.0% distillable, which is similar to the actually measured value: 90.6%.

RESULTS AND DISCUSSION

Results

Kentucky #9 coal was liquefied in Ft. Lewis recycle solvent containing 15% Kerr-McGee LSRC. The resulting solvent was batch recycled successively five times through the reactor in order to simulate steady-state recycle conditions to determine whether any significant changes would occur in the yield distribution. Table 5 summarizes the yield data from selected grab samples and compares these data with base-line data. Oils were determined by distillation; SRC was separated using hot-pyridine separation and filtration.

In Table 5, for both the base-line and LSRC-recycle runs through the first to the fifth pass, conversion measured as pyridine solubles remained the same within experimental error. Moreover, the product yield distribution became more favorable as recycling progressed. These results suggest the importance of studying multiple-pass recycle runs further to obtain more reliable yield data.

Discussion

Oil yields showed a net increase of 13% after a 10% loss in the first pass and a 3% yield in the fifth pass. SRC yields decreased from

85% in the first pass to 74% in the fifth pass. For the base-line run and the first four passes of the LSRC-recycle run, the oil yields and solvent hydrogen consumption indicate that the CPDU was operating in solvent imbalance (Table 5). Compared to base-line data, LSRC-recycle data showed that the presence of LSRC increased oil yields at the expense of SRC yields. Moreover, LSRC is significantly more effective in the fully developed recycle passes than in the first pass.

Sulfur content in SRC increased from 1.0% in the first pass to 1.2% in the fifth pass, confirming that the amount of sulfur (0.9%) shown in data from the base-line run without LSRC was less than in the passes with LSRC.

Light hydrocarbon gas yields decreased slightly as the number of recycle passes increased. In addition, the gas hydrogen take-up with LSRC increased from 0.4% in the first pass to 1.3% in the fifth pass, as the number of recycle passes increased. After the fourth pass of recycle solvent, the amount of solvent hydrogen stabilized at 7.8%.

Trends in data for total hydrogen consumption were not definitive due to variations in data associated with determining elemental hydrogen and other experimental uncertainties. However, data for light hydrocarbon gas formation, which could be more accurately determined than data for hydrogen consumption, suggested that differences in hydrogen consumptions were small and indistinguishable between single-pass and multiple-pass data. In contrast to previous results, data from the base case without LSRC showed light hydrocarbon gas yields were slightly higher than those with LSRC recycle. Although the cause of this discrepancy is currently unknown, data for hydrogen consumption after the first pass showed no apparent difference between the base case and LSRC recycle.

Table 6 compares data for simulated distillation of the oils produced during the base-line run and the LSRC-recycle run. These data indicate that recycling LSRC apparently increased the heavier components in the recycle solvent.

Table 7 summarizes data for distillable oil yield determined from the inventory of process solvents from the 7-gal batch distillation still. Data for gas yields and solvent hydrogen were based on average values obtained throughout these studies. Data for oil yield from the

7-gal still differ greatly from oil-yield data from laboratory analytical distillation, though the general trends in these data appear similar.

We assumed that the 7-gal distillation system could provide better data, because larger amounts of materials could be distilled. However, the still had fundamental design problems. For example, the only way to dispose of solids residue was to rinse the bottom of the still with creosote oil through a $\frac{1}{2}$ -in. discharge line. This operation not only plugged the discharge line, but also it introduced foreign material: wash solvent. Consequently, there was no way to completely dispose of bottoms sludge or to be certain of the status of the remaining materials.

Solids residue not only interfered with the cut points of distillate yields, but also it retained wash solvents in the system, even when the still was heated and purged with nitrogen for prolonged periods. Although small amounts of remaining wash solvent could greatly vary distillate yields in the material balance calculation, the amounts would be insufficient to disturb the properties of solvent beyond its detectable limit.

In spite of the apparent trend in the data, these observations suggest that distillation with the 7-gal still provided oil yield data no more reliable than data from the smaller, laboratory analytical distillation. Nevertheless, the still successfully performed its primary function: to recover process solvents.

CONCLUSIONS

Multiple-pass recycle runs should be studied further to determine meaningful data for studying SRC materials, especially data for oils, SRC yields, and sulfur contained in SRC. Recycle-run data for hydrogen consumption and hydrocarbon gas yields did not differ significantly from single-pass run data. Recycling LSRC: increased oil yields; decreased SRC yields; increased sulfur in SRC; had little effect on hydrogen consumption, gas yields, or conversion at demonstration-plant conditions; and changed the distribution of recycle-solvent components.

RECOMMENDATIONS

Data from the closed-loop, batch-recycle run suggest that continuously recycling process-derived materials may significantly affect quantitative yields in the SRC unit. In principle, recycle runs are desirable to better simulate actual plant design and operating conditions. However, the complexity of batch recycle in a nonintegrated process unit and the problems associated with the conventional still were phenomenal. To secure more accurate data and more reliable operating conditions, these problems should be overcome before the program is expanded further. In addition, the still should be improved to more precisely correlate data for line distillation and laboratory distillation. The problem of handling bottoms residue should be resolved by developing an effective still, batch or continuous, that can discharge solids residue precisely at the desired conditions. To close the material balance around the distillation loop, the capability to sample bottoms residue should be established.

Conventional American Society of Testing Materials (ASTM) distillation methods were unsatisfactory to sufficiently characterize the mixtures of distillates and nondistillates such as LSRC and solvent/ LSRC. More efficient and accurate separation methods should be developed to improve classification techniques. Simulated gas chromatographic distillation should be explored to adopt better quantitative characterization of coal-derived distillates related to LSRC recycle. To eliminate errors, the gas chromatograph should have the capacity to handle slurry samples without pretreatment. Techniques that incorporate proper internal or external standards should be developed to quantitate data more precisely.

The fundamental nature of Kerr-McGee separation must be understood to more accurately simulate SRC-recycle runs with laboratory solvent separation. Because achieving simulated continuous SRC recycling is currently unrealistic, a wide range of SRC must be recycled to provide a broad data base for developing mathematical simulations and to model continuous SRC recycle.

Table 1

Kentucky #9 Coal from Lafayette Mine

Proximate analysis	Wt %
Moisture	1.34
Volatile matter	40.38
Fixed carbon	48.92
Dry ash	10.23
Ultimate analysis, MF ^a	Wt %
Carbon	73.19
Hydrogen	4.95
Oxygen	7.42
Nitrogen	1.59
Sulfur	2.59
Dry ash	10.62

^aMF, moisture free.

Table 2

Comparison of Ft. Lewis and Wilsonville Recycle Solvents

	Ft. Lewis	Wilsonville	
	#1967	Run no. 211	Run no. 205
Specific gravity (wt %)	1.05	1.02	
Carbon (wt %)	87.27	86.90	87.29
Hydrogen (wt %)	8.29	8.56	8.61
Oxygen (wt %)	3.00	3.20	2.92
Nitrogen (wt %)	0.84	0.86	0.89
Sulfur (wt %)	0.60	0.48	0.44
Mol wt by VPO ^a	235	210	185
¹ H distributions (%)			
H _{ar}	32.3	32.3	32.7
H _α	33.5	30.4	28.2
H _β	34.2	37.2	39.0
SDGC ^b (°F), % off ^c			
IBP	400	369	421
10	455	440	455
20	490	470	478
30	522	490	489
40	552	518	510
50	588	545	537
60	615	581	570
70	655	614	606
80	701	658	652
90	769	718	718
95	823	768	773
FBP	934	859	877
RI ^d	1.5892	1.3050	-
SQIS ^e	N.D.	73.2	-

^aVPO, vapor-phase osmometry.^bSDGC, simulated distillation by gas chromatography.^cThis percentage approximates volumetric % off the column.^dRI, refractive index.^eSQIS, the short test for the solvent quality index.

Table 3

Compositions of Light and Heavy SRC Used for SRC Recycle Solvents^a

	Solvent (Wilsonville run no.)			
	WRS (205)	LSRC (167)	HSRC (167) (203)	
Carbon (wt %)	87.3	86.7	86.9	87.0
Hydrogen (wt %)	8.6	6.9	6.0	6.3
Oxygen (wt %)	2.9	3.9	4.5	3.6
Nitrogen (wt %)	0.9	1.4	2.0	2.1
Sulfur (wt %)	0.4	0.9	1.0	1.1
Ash (wt %)		0.02	0.1	0.3
¹ H (%)				
H _{ar}	32.7		35.2	31.8
H _α	28.2		37.2	30.3
H _o	39.0		27.5	37.8
Distillables (wt %) (1,000°F cut)	98.0	26.3		
Pentane-soluble oils	98.5		1.0	1.2
Asphaltenes	1.0		44.4	58.6
Preasphaltenes	0.5		54.6	40.2
Mol wt by VPO in pyridine	185		641	1,294
$\bar{M}_w$ by FIMS	234		-	526 ^b
$\bar{M}_w$ by FIMS	265		-	563 ^b
Softening point (°F)			380	
OH in asphaltenes as O (wt %)	1.29 ^c		1.41	1.35
NH in asphaltenes as N (wt %)	0.10 ^c		0.31	0.26
Process conditions				
	Run 167		Run 203	
Coal type	Ky #9, Lafayette		Ky #9, Dotkiki	
Slurry concentration (wt %)	37.8		36.5	
Temp (°F)	825		825	
Pressure (psig)	2,100		1,700	
Space rate (lb coal/hr-ft ³)	38		37	
Residence time (min)	43		42	
Gas rate (M scf/ton of coal)	38.8		41	
LSRC recycle rate (% recycle solvent)	4		3	

^aAbbreviations used are: VPO, vapor-phase osmometry; FIMS, field ionization mass spectroscopy; $\bar{M}_w$, number average molecular weight; $\bar{M}_w$, weight average molecular weight; H_{ar}, aromatic protons; H_α, alpha protons; H_o, protons other than aromatic and alpha.

^bThese values represent 76% of the sample detected.

^cThese values are for the oils.

Table 4

Distillation Data for Feed Materials

Feed materials	Distillable (wt %) (1,000°F cut)
1. Start-up solvent Ft. Lewis, FOB 29	94.4
2. LSRC	26.3
3. 85% solvent/15% LSRC	
Measured (after mixing) ^a	90.6
Calculated from 1 and 2 (before mixing) ^b	84.2
Calculated from 1 and 2, ^a but assumed that solvent was 100% distillables	89.0

^a Measured by vacuum distillation (methods D86 and D1160, ASTM).

^b Measured by component analysis.

Table 5

Summary of Yield Data for Base-Line and LSRC-Recycle Runs^a

	Base line		LSRC recycle		
	1st ^b	2nd ^b	1st ^b	4th ^b	5th ^b
Sample no.	34-24	34-73	35-12/36	35-163	35-183
Temp (°F)	838	843	838	832	830
Conversion	90	86	89	86	89
C ₁ -C ₄	10.2	9.3	7.7	6.3	6.7
CO, CO ₂	1.0	1.1	1.0	1.0	1.0
H ₂ S, NH ₃	1.8	1.4	1.5	1.7	1.6
H ₂ O	4.8	4.3	3.3	3.3	3.0
Distillable oils	-16	-20	-10	-6	3
SRC	88	90	85	80	74
Sulfur in SRC	0.94	0.93	1.04	1.23	1.24
Hydrogen consumption					
Total	1.6	1.2	1.1	0.9	1.2
Gas	1.2	0.2	0.7	1.1	1.3
Solvent	0.4	1.0	0.4	-0.1	0.0
Hydrogen in solvent					
Start	8.39	8.24	8.16	7.80	7.85
Finish	8.23	7.84	8.05	7.84	7.86

^aAll values are expressed in wt %. To achieve internal consistency of the data, the basis for calculation was 89.0 wt % from Table 4.

^bPass number.

Table 6

Redistribution of Distillate Components in LSRC
Recycle-Derived Solvent^a

Cut (°F)	Composition (wt %)	
	Base line (2nd) ^b	LSRC recycle (4th) ^b
450-550	41.1	37.3
550-650	33.4	33.8
650-750	18.7	21.4
750-850	6.9	7.6

^a Calculations were determined by SGDC, using ASTM 2887.

^b Pass number.

Table 7

Oil Yield Data Based on 7-Gallon Distillation Still^a

	Base line		LSRC recycle				
	1st ^b	2nd ^b	1st ^b	2nd ^b	3rd ^b	4th ^b	5th ^b
Distillable oil yield	-34	-39	-14	13	6	19	14
Hydrocarbon gases (C ₁ -C ₄)	9.8	7.9	7.0	7.6	5.4	6.1	6.2
CO, CO ₂	1.0	1.0	1.0	1.0	0.9	0.9	0.9
H ₂ S, NH ₃	1.5	1.5	1.5	1.5	1.5	1.9	1.6
H ₂ O	4.4	4.1	3.3	3.3	2.6	3.1	3.2
Solvent hydrogen							
Start	8.4	8.2	8.2	8.1	7.9	7.8	7.9
Finish	8.2	8.3	8.0	7.9	7.7	7.8	7.9

^aAll values are expressed in wt %.^bPass number.

Figure 1
Mass Spectrum for the Wilsonville Recycle Solvent
from Run 205

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

WILSONVILLE TECHNICAL SUPPORT PLAN

S. C. Weiner*

INTRODUCTION

Operation of the Wilsonville SRC Pilot Plant serves the vital role of technical support required to ensure the success of the SRC-I Demonstration Plant project. To guarantee that future needs are proposed, reviewed, approved, and implemented, a Wilsonville Technical Support Plan has been formulated by ICRC Engineering Technology.

The plan itself contains four elements: process runs, projects, process variable studies, and samples. Process runs, generally 4 to 6 weeks each, are needed to verify demonstration plant design parameters, or to test potential changes in such parameters. The projects area includes equipment additions, changes, and testing programs. Process variable studies for pilot plant operation investigate the specific effects of an independent variable on a dependent variable. The samples area includes a wastewater sampling program, as well as projections of intermediate and product stream samples required to support the R&D and product demonstration programs of ICRC Engineering Technology and Business Management, respectively.

Most of the programs included in the plan are discussed in the main volume of the SRC-I Quarterly Technical Report, July-September 1981. Three of the programs were believed to be proprietary, but have now been released for publication, and are discussed below.

*International Coal Refining Company (ICRC)

PROJECTS

KERR-MCGEE FIRST-STAGE, HEAVY-PHASE HANDLING

Classification: Evaluation of process improvement

Priority: low

This project addresses the control of the Kerr-McGee ash concentrate particle size by changing the letdown technique from the first-stage settler to either multiple valves or an extruder. Increase in the particle size of the solids in a controlled manner would greatly simplify the handling and storage of the solids.

The scope of this project has not yet been defined.

CHLORIDE MASS BALANCE AND TREATMENT PROGRAM

Classification: Direct design support

Priority: high

Background and Justification

Petroleum refinery experience indicates that, in general, chlorides and other corrosive species are easier to handle and eliminate once they become part of process water streams. Accordingly, this concept will be used in the SRC-I Demonstration Plant design philosophy. It is therefore necessary to determine which process streams contain chlorides, how they flow through the plant, and how they can be removed from hydrocarbon streams and transferred to water streams.

Studies at Oak Ridge National Laboratory (ORNL) and the Institute for Mining and Mineral Research (IMMR) suggest that the constituents necessary for corrosion to occur are phenols, basic nitrogen compounds, and water-soluble or water-extractable chlorides. Removing the basic nitrogen compounds reduces the corrosion rate to base levels of those of phenols. Removing the phenols from the system reduces the corrosion rate even further. It has been found that the most severe corrosion

occurs in the 400-500°F temperature range, and is usually found only in the fractionation tower where chemical as well as physical reactions are probably taking place. This 400-500°F range is generally not considered corrosive to 300 series stainless steel piping and other components in the hot oil system.

To determine the flow patterns of chlorides, phenols, and basic nitrogen compounds in the Wilsonville Pilot Plant, a run was completed in August 1980, during which the plant processed high-chlorine Dotiki coal without sodium carbonate addition. Approximately 17 sets of 20 samples were taken at various locations. Three sets were shipped to ORNL and 14 were sent to IMMR for analysis. The results from both laboratories suggest that large amounts of phenols (up to 20 wt %) are present in most oil streams. Phenols tend to concentrate in the fractionation tower, reaching levels as high as 50 wt % in some locations. Basic nitrogen compounds constitute approximately 2-4 wt % of the streams analyzed. The amounts of phenols and basic nitrogen compounds are fairly stable, regardless of whether or not sodium carbonate is added to the feed coal.

Of two types of chlorides identified in the various oil streams, one is organic and not water-extractable. The other type, sometimes referred to as inorganic chlorides, is water-extractable and probably associated with basic nitrogen compounds. Streams containing only organic chlorides are essentially noncorrosive to the 300 series stainless steels. However, those containing water-extractable chlorides are severely corrosive to stainless steel under certain circumstances.

Two theories have been presented concerning the way in which chlorides are transported to the fractionation column. It is thought that the chlorides from the overhead system, which are washed out in a phase separator, contribute most of the chlorides to the stream entering the fractionation tower, a result of poor phase separation or slugging from this vessel. Another theory, presented by IMMR, suggests that the predominant continuous source entering the fractionation tower comes from the high-pressure separator bottoms flowing through the vacuum tower and into the feed of the atmospheric tower. This theory is supported by numerous chloride material balances around a number of vessels

and by iron analyses and balances around the fractionation tower. ICRC tends to accept IMMR's flow path theory.

Studies have been conducted to determine why the chlorides flow in this path and why severe corrosion is not experienced in the vacuum tower, but in the fractionation tower at Wilsonville, since the same boiling range of components appear in both towers. Interpretation of the data suggest some patentable ideas that, when applied to the process, will remove chlorides from the oil streams. These ideas are being incorporated into the demonstration plant design.

All the above work has essentially been completed and has provided a good understanding of the flow of chlorides, phenols, and basic nitrogen compounds in the Wilsonville Pilot Plant. To better understand how this data can be correlated with the demonstration plant design, additional samples are being collected at other locations. ICRC has been conducting some retrograde reaction tests in the Conoco laboratories. This laboratory pilot plant has three liquid vapor separation vessels that operate under conditions similar to those of the demonstration plant. For example, at Wilsonville, 40 wt % of the chlorides went overhead when the high-pressure separator was operated at 650°F. At Conoco, the high-pressure separator operated at 830°F, which is within 10°F of the demonstration plant design temperature. During one of the recent runs at Conoco, several samples (including gas) were collected around the high-, medium-, and low-pressure separators, and sent to IMMR laboratories for analysis. Results of these analyses are not yet available.

A second study to be conducted within several months involves the collection of samples around the front end of the Ft. Lewis pilot plant. Sample points have been identified upstream of the fractionation area that will provide a phenol, basic nitrogen compound, and chloride balance around this portion of the plant. Since the high-pressure separator at Ft. Lewis operates at a higher temperature than that at Wilsonville, but lower than the Conoco laboratory unit, sampling around the front end of this plant should help verify the distribution coefficients from the Wilsonville and Conoco facilities. This study is being organized by ORNL.

The results from all the above studies will provide engineering data for the demonstration plant design. Interpretation of the data will allow ICRC to design and incorporate cleanup techniques in the demonstration plant. Before finalizing the design, these techniques should be tested at Wilsonville.

Objectives

- ° Reduce or mitigate corrosion in the solvent tower.
- ° Ensure that corrosion is not moved elsewhere in the plant.

Schedule

The following schedule lists completed programs, and provides an estimate of ICRC's future needs:

1. Determine conditions for corrosion to occur - complete.
2. Determine flow of chlorides in the Wilsonville Pilot Plant - complete.
3. Interpret the data to determine why chlorides flow in certain paths - in progress.
4. Collect and analyze samples from Conoco PDU - samples are collected; analyses are underway; additional work is to be prepared.
5. Collect and analyze samples from Ft. Lewis - planned for third quarter, CY 1981.
6. Extrapolate above data for demonstration plant design purposes - in progress.
7. Design cleanup methods - in progress.

8. Test modifications at Wilsonville - proposed for fourth quarter, 1981 to first quarter, CY 1982.
9. Incorporate modifications into demonstration plant design - CY 1982.

PROCESS VARIABLE STUDIES

DEASHING SOLVENT INJECTION PROGRAM

Classification: Direct design support

Priority: high

Justification

The critical solvent deashing (CSD) unit at Wilsonville is started up at a high, first-stage temperature to prevent the heavy-phase underflow from plugging the letdown valve. As the unit starts to line out, the first stage temperature is slowly decreased to normal operating levels. At the high initial temperatures, the ash concentrate will occasionally contain clumps or form a hard, extruded mass instead of a dry, free-flowing powder. During this period, the ash concentrate flow is diverted to a 55-gallon drum instead of the normal ash concentrator. Once the temperature of the first stage is brought down to normal levels, a dry, free-flowing powder is formed and the ash concentrate flow is switched to the ash concentrator.

For the demonstration plant, a system is being proposed to reconstitute the heavy phase from the first-stage with SRC and to then send the reconstituted material to the solidifier. The process will operate in this fashion at start-up until an acceptable, free-flowing powder is formed. The equipment required for this start-up system costs \$1 MM and has not yet been demonstrated.

In this proposal, a suggested alternate approach is based on the theory that a dry, free-flowing powder is not formed at start-up for one of two reasons: (1) the temperature of the ash concentrate is not cool enough to completely solidify the fine ash concentrate droplets formed

at letdown, thereby promoting the agglomeration of particles into clumps; or (2) insufficient deashing solvent (DAS) is present to "explode" the heavy liquid phase into fine droplets, resulting in an extruded solid mass.

Increasing temperature in the first stage causes the ash concentrate to change from a dry, free-flowing powder into the form defined under (1) above and then into the form defined under (2) above. This occurs because the temperature increase brings about three simultaneous detrimental effects. First, the higher temperature of the heavy phase results in a higher flash temperature of the ash concentrate, and consequently incomplete solidification. Second, the higher first-stage temperature reduces the density of the DAS which increases the momentum of the solvent to pass from the heavy phase into the light phase. This results in even less cooling and breakup of the heavy phase upon letdown. Third, the higher first-stage temperature reduces SRC recovery and causes more SRC to be rejected to the heavy phase. This further decreases the softening temperature and hinders complete solidification of the ash concentrate.

This proposal suggests that the formation of clumps in the ash concentrate can be eliminated by injecting DAS upstream of the heavy-phase letdown valve, while maintaining a maximum temperature of operation in the first-stage settler. The injection of DAS upstream of the letdown valve during start-up, normal operation, and shutdown serves three functions: (1) it improves flowability of the heavy phase and prevents plugging at the valve and just downstream of the valve; (2) it provides cooling by evaporation to completely solidify ash concentrate droplets, therefore eliminating the tendency to clump; and (3) it provides sufficient explosion force by evaporation to form the fine particles.

This project proposes controlled experiments to inject DAS at various first-stage temperatures, while carefully monitoring the consistency of the ash concentrate, to determine if DAS injection can assure a dry, free-flowing powder.

Objective

- ° Determine if injection of deashing solvent upstream of the first stage, heavy-phase letdown valve provides adequate explosion force and cooling to assure the formation of a dry, free-flowing ash concentrate at all operating conditions.

Statement of Work

This project is divided into four phases, each successive phase run only after successful demonstration of the preceding phase. Both Phases I and II are to be carried out with the ash concentrate drum.

Phase I (Demonstration plant conditions in front end). The objective of Phase I is to determine the effect of DAS injection and first-stage temperature on the consistency of the ash concentrate. Four temperatures will be tested, with and without DAS injection, for a total of eight tests. The test at the lowest temperature without DAS injection will be run first, followed by a test at the same temperature with DAS injection. Each successively higher temperature will then be run both without and with the DAS injection. A sample of the ash concentrate will be collected 1 hour after the start of each test. The following information will be collected for each test: (1) operability of the letdown valve, (2) consistency of ash concentrate, (3) particle size of ash concentrate (APCI microtrac technique), and (4) composition of ash concentrate (% DAS, % ash). If possible, the DAS passing to the drum during each test is to be collected and measured.

Phase II (Demonstration plant conditions in front end). The objective of Phase II is to determine the effect of DAS injection during start-up and shutdown periods. The same information collected during Phase I on the letdown valve and ash concentrate will be determined. For the first two of six tests in Phase II, the CSD unit will be started up as normally done, first without DAS injection and then with DAS injection. For the next two tests, the CSD unit will be started up with a lower first-stage temperature both without and with DAS injection. The last two tests will involve collecting samples during the normal CSD shutdown period, both without and with DAS injection.

Phase III (Demonstration plant conditions in front end). If the first two phases demonstrate that a dry, powdery ash concentrate can be formed with DAS injection during start-up and shutdown periods and at temperatures normally expected in the first stage, then Phase III will involve running the heavy phase to the ash concentrator vessel during start-up, normal operation, and shutdown with DAS injection to confirm its operability.

Phase IV. If the first three phases are successful, the quality of the CSD feed will be lowered by shifting to less severe liquefaction conditions and checking the applicability of DAS injection when SRC recovery is lower. These tests will be run using the drum with DAS injection at all times, including start-up and shutdown. If the tests are successful, they will be repeated with the ash concentrator vessel.

Schedule

This program should be completed in the third quarter of CY 1981.

☆ U.S. GOVERNMENT PRINTING OFFICE: 1984-746-081/10132