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SOLVENT REFINED COAL (SRC) PROCESS

Monthly Report for the
Period of October 1980

The Pittsburg & Midway Coal Mining Co.
Englewood, Colorado 80111

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ABSTRACT

This report summarizes the progress of the Solvent Refined Coal (SRC) project by The Pittsburg & Midway Coal Mining Co. at the SRC Pilot Plant in Fort Lewis, Washington and the Gulf Science and Technology Company Process Development Unit (P-99) in Harmarville, Pennsylvania, for the Department of Energy during the month of October, 1980. The Fort Lewis Pilot Plant was shut down the entire month of October, 1980 for inspection and maintenance. PDU P-99 completed two runs during October investigating potential start-up modes for the Demonstration Plant.

I. Plant Operation

The Fort Lewis SRC Pilot Plant was shut down from September 26 to October 28 for the first half of the 1980 annual turnaround. The remaining half of the annual turnaround was rescheduled for March, 1981. The primary objectives for this current turnaround were to (1) inspect all areas of the plant, (2) reinsulate the lower 30% of the coil in Preheater B, (3) install the 1 1/2" corrosion/erosion spool in the outlet line of Preheater B, (4) install the 1 1/4" corrosion/erosion loop in the outlet line of Dissolver A, and (5) test the process and storm drain sewers.

Inspections were made in all areas of the plant but the main emphasis was placed on equipment in Areas 01, 02, 04 and 09.5. These inspections, along with associated repairs, included:

The Coal Dust Collector (Area 01). Using an ultraviolet light sensitive tracer, it was determined that 42 of the 130 bags were leaking. Most of the leaks were in the seam folds at the tube sheet. Discussions with the equipment vendors indicate that some leaking is normal but that it can be reduced if necessary. The leaking bags were replaced. Six holes in the baghouse vessel walls were sealed with silicone.

The Fully Lined Lawrence Pump (Area 01). Extreme wear in the cutwater section of the liner prompted replacement of the liner, the suction hub disc, the hub disc and the impellor.

The Vortex Mixer (Area 01). Wear on the upper propeller was substantially different from that of the lower propeller. This difference may indicate that the upper propeller is running in the eye of the vortex and not affecting mixing.

The Willis Isolation Valves (Area 02). Two of the valve bodies were showing signs of erosion; one of these valves was replaced.

The Hydrogen/Dowtherm Heat Exchanger (Area 02). This exchanger was partially plugged with slurry but could not be hydroblasted because of limited access. Removal of the unit required cutting the tube sheet. After hydroblasting, this unit was sent to Alaska Cooper and Brass (Seattle) for reassembly. Fortunately, cracks in the weld zone between the adapter and the tube sheet were discovered during weld preparation. Attempts to repair these cracks were unsuccessful. Repairs were eventually completed by redesigning the tube sheet. Installation of the new tube sheet required several changes in the exchanger and repairs were quite time consuming.

The Filter Feed Flash Vessel Exchanger (Area 03). In the past year, the U-bends in this double pipe exchanger had lost 65% of nominal wall thickness; the straight sections, 48%. The exchanger was rebuilt with 2 1/2" schedule 80 carbon steel tubes, but they will be replaced with 300 series stainless steel in March, 1981.

The Vacuum Flash Preheaters (Area 04). Radiographs of both these units indicate decoking will be required in the immediate future.

The Light Ends Column and the Wash Solvent Column (Area 04). The walls and the reboiler bundles of each of these units were in good condition. Some corrosion was observed on trays 15 through 17 in the Wash Solvent Column. Approximately 90% of the valves in tray 16 were corroded.

The Fresh Hydrogen Compressor Intercooler (Area 05). The carbon steel tube bundles were deeply pitted. Repairs consisted of retubing an older spare bundle with 316 stainless steel and substituting it for the pitted cooler. The spare cooler was modified instead of the pitted cooler to avoid scheduling problems. The newer carbon steel bundles will be retained as spares.

The Inert Gas Compressor Intercooler (Area 09.5). This cooler was retubed using 304 stainless steel in place of carbon steel.

Inert Gas Unit Flue Gas Separator (Area 09.5). The carbon steel internals of the separator were severely corroded. A new separator was ordered and will be installed in March, 1981.

The DEA Unit (Area 09.5). The DEA reboiler was retubed using 304 stainless in place of carbon steel.

The Process Drain Sewers. Testing of the process drain sewer system was completed in compliance with an agreement with the Washington State Department of Ecology. With one exception, repairs were required in all sections of the system. One section of the main line was repaired by installing 6" carbon steel pipe inside the 8" vitreous clay pipe. Other sections of drain were excavated and replaced. The lateral drains in Areas 03 and 04 and at the Flare Knockout Drum were replaced with surface drain trenches. These trenches drain into a new lateral drain field which delivers the waste to an existing main. This work delayed startup approximately one week.

Solvent circulation was initiated October 28. Gas metering tests, solvent metering tests, vortex mixer tests, Lawrence pump tests and dissolver mixing tests were either completed or underway within the month. Completion of these tests is expected early in November. Plant operation with coal feed

will then resume to complete a series of high heat flux runs using Slurry Preheater B. The high heat flux run tests consist of 4 solvent baseline runs and 6 slurry runs. These runs are duplicates of previous runs completed in January-February, 1980. They are being repeated because of coil skin temperature measurement problems during the original test runs.

II. Experimental Programs

A) Dissolver Mixing Tests

Five dissolver mixing tests designed to study the mixing patterns extant in the Fort Lewis reactor were completed. Actual conditions maintained during each run period are shown in Table 1.

The first two runs (DA-1 and DA-2) were designed as calibration checks to identify any instrumentation problems. These runs were conducted with only solvent and process gas in the reaction system. The observed temperature profiles revealed an error in the bottom wall-side temperature reading. Otherwise, temperature measurements were in good agreement, especially considering the possibility of a small heat loss near the top of the vessel.

Slurry test runs DB-1, DB-2 and DB-3 were designed to study dissolver flow profiles when feeding coal slurry and process gas. Preceding each DB test, the slurry preheater temperature was reduced to the minimum level necessary to maintain an 860°F dissolver outlet temperature without introducing quench gas to the dissolver. Relatively stable operation was maintained for short periods during each of the DB series runs.

Test run DB-1 produced a relatively flat temperature profile indicating a fair degree of back mixing. During this run, the inner core of the reactor appeared to be somewhat cooler than the outer edges. Some temperature loss near the top of the dissolver was probably the result of heat loss from the uninsulated dissolver top head and outlet piping.

Runs DB-2 and DB-3 were designed to study dissolver flow patterns with different gas and liquid flow rates. The resulting temperature profiles showed only slight differences, indicating a fairly well back mixed system.

B) Preheater Baseline Tests

Two slurry preheater baseline tests were completed during the month. These tests, R-8 and R-9, were repeats of previous tests conducted before the improved skin thermocouples were installed on the preheater coil. Run average operating conditions for the completed tests are shown in

Table 2. Preceding these tests, the bottom 30% of the heater coil was insulated, producing higher overall heat flux in the active section. In both tests the fluid temperature rose quite slowly in the insulated section, indicating good insulation. In the uninsulated section, the fluid temperature increased in a nearly linear fashion with coil distance.

Run R-9 had higher inlet and outlet temperatures and a greater temperature increase than run R-8. Both runs showed uniform bulk to skin differentials in the uninsulated portion of the coil indicating uniform heat flux and heat transfer. A slightly larger differential was indicated during run R-9, which reflects the higher fluid throughput during this test.

Overall coil pressure drops in each run were very low (approximately 10-12 psi). Both tests showed a uniform pressure drop with increasing distance from the coil inlet.

C) SRC-I Liquid Inventory

Weekly plant inventory measurements of liquid products were made in accordance with standard procedure during the recent SRC-I run period. Figure 1 shows a plot of weekly oil production data and coal consumption. During the 16 week period shown, a total of 1,440 tons of Kentucky coal was consumed. Wash solvent was the major product accumulated, netting a production rate of approximately 0.6 barrels per ton of raw coal feed. Process solvent production showed a net increase of approximately 0.3 barrels per ton of raw coal. Light oil production figures during the period were inconclusive due to the use of this material in recycle service and numerous transfers and shipments.

D) Rheological Test Program

The rheological program continued with characterization of vacuum flash feed material at a hold-up temperature of 770°F. Samples of the synthetic feed were poured into 1/2" o.d. stainless steel sample bombs that were fitted with Swageloc caps. Each sample was inerted with nitrogen and sealed, and the tubes were lowered vertically into the furnace. A thermocouple was wrapped around each tube so the temperature of each sample could be monitored.

All samples reached 770°F in 20 minutes. Samples were held in the furnace for 1, 2, 4 and 8 hours after having reached the target temperature, and were then removed and allowed to cool. Upon opening each sample bomb, it was found that gas had been created during the test and solids had stratified in the lower half of the sample bomb forming a hard plug. The solids were drilled out

and the remaining sample was melted out. The solids were crushed to -60 mesh and combined with the liquid.

Viscosity measurements were made using the vacuum flash material which had soaked for 1, 2, 4 and 8 hours. Measurements were made at 450°F, 500°F, 550°F and 600°F. Higher temperature measurements were not attempted since the synthetic feed had an initial boiling point of 642°F. Viscosity measurements made at several test temperatures are shown in Figure 2.

Since it was impossible to directly measure the viscosity at the target (770°F) temperature, results expected at this temperature were obtained by extrapolation. The predicted vacuum flash feed viscosity calculated in this manner is shown in Figure 3.

Attempts at measuring the viscosity of samples that were held at 770°F for 24 and 48 hours failed, since the solid and liquid portions of the sample could not be recombined to produce a homogeneous mixture. Wide fluctuations were observed when viscosity measurements were attempted (± 50 cp). It is recommended that further testing of vacuum flash feed be done in a stirred autoclave. Testing of coal feed slurry will begin in November.

III. SRC-II Operation of Process Development Unit P-99

A. P-99 Operation

During October, two runs (Runs 75 and 76) were completed. Operating conditions for these runs, which were a continuation of the low coal concentration run series, are shown in Table 3. Both these runs were made with a feed slurry containing 22.5% coal and 11.3% recycled solids. Run 75 was made at a slurry residence time of 1.33 hr, thus representing a coal feed rate about half the standard rate. Run 76 was made at a slurry residence time of 1 hr and represents a coal feed rate three quarters the standard rate.

At the beginning of Run 77, a number of difficulties were experienced with the fractionation system. First, a pressure build-up began to occur across the vacuum column. It was decided to flush the column with process solvent in an attempt to overcome the pressure drop problem. Some plugging was experienced during the flushing operation, but after about a day and a half, the vacuum column was successfully put back into operation. However, after a short period of operation, the pressure drop across the column again began to build up. It was thought that leaking valves were contributing to this problem, and while attempting to replace the bottom valve, the vacuum column plugged again. At the same time several incidents of plugging of the atmospheric flash column level control valve occurred. Then, one of the Calrod heaters on the fractio-

nator reboiler burned out and caused a leak in the reboiler. Rather than try to continue operations, it was decided to come down for a general unit turnaround.

Upon opening the dissolver, it was found to be in very good condition with only about 1% of its volume being occupied by coke. The hot separator contained some dry, powdery solids, but was otherwise in good shape. The bottom of the vacuum column was full of solidified material which had accumulated when the bottom drain plugged, but otherwise was found to be cleaner than expected. Most internal surfaces were covered with a thin coating of tar. The Koch demister was coated with tar but was not plugged. This raises some questions as to the cause of the pressure drop observed across the vacuum column.

Data on Runs 67 to 71 are now essentially complete, and yields for these runs are presented in Table 4.

B. Future Work

Studies during November and December will evaluate 11% ash Ireland Mine coal at two slurry compositions and a longer (1.33 hr.) slurry residence time.

IV. SRC-II R&D Support Programs

A. SRC-II Prepilot Plant Development

Five slurry recycle runs were successfully completed during the month of October. These runs (13) constitute the entire experimental compliment of the Feed Coal Effect on Kinetics task. As several product analyses have not yet been completed, no conclusions based on the dataset generated under this project can be made at this time.

B. SRC-II Reactor Modeling and Simulation

Efforts were primarily directed toward developing concepts in the area of SRC-II reactor thermal behavior.

New data on the thermal effects in the Ft. Lewis reactor have been obtained and analyzed. These results show that the heat loss relative to heat generated is significant. Also, the calculated heat of reaction is consistent with the new value obtained from certain P-99 data.

C. Process Cost Model

Work has begun on developing the equation systems for the model, which is the central task of the project. The cost equation system will revolve around the flow matrix, F , which is a 30 by 30 array of flow rates between the process areas. A yield matrix, y , will also be set up to relate the flow rate out of a process area as a function of the input flow rates.

This yield matrix will operate on the initial flow matrix to give a new flow matrix, which will be checked for consistency and material balances. An iterative process will be employed to achieve a solution for the flow and yield systems. From the final flow matrix, the investments and operating costs can be easily determined.

D. Coking of SRC-II Process Streams

A study of a Fort Lewis preheater outlet sample is being carried out to characterize its coking tendency using techniques developed for P-99 streams. The thermogravimetric analysis of the preheater sample showed that it had a higher content of light volatile matter and a lower content of fixed carbon plus heavy volatile matter compared to those found in feed slurry. A solubility study showed that parts of coal have been dissolved or liquefied under the preheater operating conditions. The X-ray diffraction analysis of low temperature ash indicated that pyrite and pyrrhotite coexist in the preheater outlet sample.

E. Corrosion, Erosion and Materials

An evaluation of the Argonne National Laboratory high temperature ultrasonic method for measuring wall thickness indicates that it should be acceptable for on-stream monitoring of wall thickness where corrosion or erosion is expected.

V. Schedule of Operations

Activities and milestones scheduled for the period of November through February, 1981 are indicated in Figure 4.

TABLE 1
Dissolver Mixing Test
Operating Conditions

Run	DA-1	DA-2	DB-1	DB-2	DB-3
Date	8/24/80	8/25/80	9/15/80	9/22/80	9/19/80
Time	1700-1800	0400-0600	1000-1800	2100-2300	0400-0600
Raw Coal Feed (lb/hr)	---	---	2160.0	2190.0	1440.0
Moisture Free Coal Feed (lb/hr)	---	---	2083.5	2131.1	1393.4
Recycle Slurry or Solvent (lb/hr)	7192.0	7072.0	4127.9	4415.0	2651.5
Pump Seal Flush (lb/hr)	---	---	728.4	416.1	493.7
Total Slurry or Solvent (lb/hr)	7192	7072	6940	6962	4538
Gas to Dissolver (lb/hr)	623.2	798.4	1120.4	837.5	818.1
Pulsation Damper Purge (lb/hr)	10	10	10	10	10
Preheater dP Taps Purge (lb/hr)	10	10	10	10	10
Total Gas to Dissolver (lb/hr)	643	818	1140	857	828
Dissolver Pressure (psig)	1985.6	2013.6	1812.9	1985.1	2013.6

TABLE 2
Preheater B SRC-II Baseline Runs
Operating Conditions

Run No.	R-8	R-9
Date	10/31/80	11/01/80
Time	0000-0200	1600-1800
Solvent Feed, FT-1152 (lb/hr)	6848	7412
Total Gas to Preheater (lb/hr)	372	375
Gas to Dissolver, FT-85 (lb/hr)	353	363
Preheater Inlet Temperature (°F)	176	349
Preheater Outlet Temperature (°F)	516	801
Preheater Inlet Pressure (psig)	2038	2041
Preheater Outlet Pressure (psig)	1995	1995
Pressure Drop, by difference (psid)	43	56
Preheater Pressure Drop, dP 985 (psid)	11	14
Fuel Gas Rate (lb/hr)	213	253
Ambient Temperature (°F)	50	52
Stack Gas Temperature (°F)	1100	1334
Firebox Temperature (°F)	986	1262

Table 3

OPERATING CONDITIONS FOR OCTOBER PDU P-99 RUNS

<u>Run No., P99-</u>	<u>75</u>	<u>76</u>
Dissolver Outlet Temp., °F	833	842
Dissolver Avg. Temp., °F	831	837
Preheater Outlet Temp., °F	790	763
Pressure, psig	2000	2000
Slurry Residence Time, Hr	1.33	1.0
Feed Slurry Composition, Wt%		
Coal	22.5	22.5
Recycled Solids	11	11
Dissolver Gas		
Rate, 10 ³ SCF H ₂ /T Coal	92	68
Purity, Vol%	94	94

Table 4
YIELDS FROM
POWHATAN NO. 6 AND IRELAND MINE COAL RUNS MADE ON PDU P-99

<u>Run No., P99-</u>	<u>67</u>	<u>68</u>	<u>69</u>	<u>70</u>	<u>71</u>
Hydrogen	-4.0	-3.8	-4.0	-3.9	-4.0
C ₁ -C ₄	14.4	13.6	15.5	14.9	14.8
Other Gases	4.6	4.3	4.6	4.5	4.7
Water	4.9	5.4	4.8	4.7	4.8
C ₅ -380°F	8.9	7.8	8.9	8.4	7.9
380-550°F	16.2	13.2	15.2	14.3	15.2
550-900°F	<u>15.4</u>	<u>17.2</u>	<u>15.5</u>	<u>13.2</u>	<u>16.0</u>
Total C ₅ -900°F	40.5	38.2	39.6	35.9	39.1
900°F+ Pyridine Sol.	22.6	26.4	23.2	23.9	20.4
IOM	5.6	4.5	4.9	3.8	4.0
Ash	<u>11.4</u>	<u>11.4</u>	<u>11.4</u>	<u>16.2</u>	<u>16.2</u>
Total 900°F+	39.6	42.3	39.5	43.9	40.6

FIGURE I

FIGURE 2

RHEOLOGICAL BEHAVIOR OF VACUUM FLASH FEED

FIGURE 3

PREDICTED RHEOLOGICAL BEHAVIOR OF
VACUUM FLASH FEED AT 770 °F

FIGURE 4

SCHEDULE OF OPERATIONS AND EVENTS**DEAC-0179-ET10104**

TASK NO.	TASK	NOV 1980	DEC 1980	JAN 1981	FEB 1981
1	SRC-I PROCESS STUDIES	NO ACTIVITY PLANNED DURING PERIOD			
2	LUMBUS ANTISOLVENT DEASHING	△ ^A		▽ ^B	
3	SRC-II PROCESS STUDIES	▽ ^A		▽ ^B	
4	EROSION / CORROSION STUDIES			▽ ^A	
5	SLURRY FLOW METER TESTS	▽ ^A			
6	PREHEATER PROGRAM - PHASE III	▽ ^A		▽ ^B	▽ ^C
7	COARSE COAL FEEDING	▽ ^A	▽ ^B		
8	PUGMILL EXTRUDER	▽ ^A			
9	ENVIRONMENTAL & TOXICOLOGICAL PROB.		▽ ^A		
10	PROCESS DEVELOPMENT UNIT (PDU) OPERATION	▽ ^A	▽ ^B	▽ ^C	▽ ^D

Milestone IdentificationTask 2

A. Complete maintenance and settler modifications for December 1980 operation. B. Complete operability demonstration run with underflow modifications.

Task 3

A. Complete data collection for "normal" startup. B. Complete 2nd startup study, 100% design.

Task 4

A. Complete erosion/corrosion topical report.

Task 5

A. Demonstrate modified ANL Doppler flowmeter in slurry service.

Task 6

A. Complete SRC-II, 2" coil high heat flux recheck runs. B. Complete installation of 1 1/2" preheater coil.
C. Complete SRC-II preheater survey runs, 1 1/2" coil.

Task 7

A. Complete coarse coal runs with vortex mixer. B. Report results of vortex mix tank tests on "coarse" and "fine" coal.

Task 8

A. Begin sustained operation of full scale pug mill - low priority.

Task 9

A. Begin acute testing on SRC-II materials.

Task 10

A. Begin Ireland Mine and L/D studies. B. Winter turnaround. C. Complete L/D Studies. D. Solvent Boiling Range Studies.