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SOLVENT REFINED COAL (SRC) PROCESS

Monthly Report for the
Period August 1977

The Pittsburg & Midway Coal Mining Co.
Merriam, Kansas 66202

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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 at Fort Lewis, Washington, and the P&M Laboratory in Merriam, Kansas, for the Energy Research and Development Administration for the month of August, 1977. During August, the Fort Lewis Pilot Plant processed a blend of Kentucky Nos. 9 and 14 coal during 31 days of operation in the SRC II mode. Four SRC II material balance runs were completed during this period. Eight exploratory SRC II runs on coals other than the Kentucky Nos. 9 and 14 to determine their suitability for processing in the SRC Pilot Plant were made at the Merriam Laboratory.

I. Summary

Four material balance runs were completed in August. Data from these runs indicate that increasing coal concentration results in somewhat lower distillate yield and higher SRC yield. The effect of coal concentration is non-linear; at least partly due to the fact that some solvent must be added to the slurry to permit steady operation at higher coal concentrations. Results also show tendencies toward increased distillate yield and decreased SRC yield with increased dissolver residence time.

Operating problems were concentrated in the coal-slurry mixing systems, especially at higher coal concentrations. Frequent plugs occurred in the eductor system usually as a result of slurry recirculation pumping problems. The new mix tank system performed well during the first part of the month but then began to have problems with plugging and suffered a broken shaft. Modifications are now underway to improve this system's reliability.

The old vacuum flash heater was unplugged and solvent circulation was established throughout the system. The new vacuum flash system operated continuously throughout the month with no major problems.

Both of the new 1300 barrel storage tanks have been completed and are being commissioned for SRC II liquid storage.

Engineering projects for the month included modification of the slurry mix tank system, temporary insulation of the slurry recycle air cooler to permit higher slurry blend tank temperatures, inspection of "B" dissolver and the mineral residue dryer, and a procedure for reclaiming the Illinois coal with no possibility that it might be mixed with Kentucky coal.

The Johns-Manville filter arrived in good shape August 16. Engineering is proceeding on schedule and the total estimated cost of installation is $1,146,000. Engineering is also proceeding on the Lummus Deashing System. Purchase orders have been issued for all pumps and all major vessels (except the settler). A letter stating the Total Installed Cost is expected to be $3.2 to $3.5 million, was received from Lummus August 11.

Eight exploratory SRC II trials were made at Merriam to determine the suitability of Pittsburgh Seam and Illinois No. 6 coals for processing at Fort Lewis. Six runs were made with an Illinois No. 6 coal from the River King Mine, and one each with the Pittsburgh Seam coal from the Ireland and Blacksville No. 2 Mines. The River King and Ireland Mine coals gave yields quite similar to the Kentucky Nos. 9 and 14 coal while the Blacksville No. 2 Mine coal was somewhat less reactive.

II. Pilot Plant Operations, Engineering, and Maintenance

A. Coal Receiving and Preparation - (Area 01)

During August, coal was processed 31 days resulting in a coal consumption of 864 tons. All operation was in an SRC II mode with Kentucky coal. This month 359 tons of Kentucky coal and 914 tons of Illinois coal were received. Extra precautions were taken to avoid mixing the two types of coal. In addition to matching car numbers with coal type, the shuttle conveyor reversible motor was rewired for single direction rotation. The wiring change eliminated the possibility of accidentally dumping Illinois coal into the Kentucky coal storage bin.

Coal slurrying was a major problem this month. The new slurry mix system was faultless the first nine days of August, but operation after this date was very poor. It appears that a coal-solvent mud builds up on the mix tank walls, plugging the coal inlet chute. Chunks of mud fall off, binding the agitator and plugging the mix tank outlet lines.

The slurry circulating pumps frequently lost suction. To regain suction, the pumps had to be solvent flushed, resulting in slight process upsets. Circulating pump problems were very frequent during MBR 77SR-4 and 6.

B. Slurry Preheating and Dissolving - (Area 02)

Area 02 processed coal slurry every day of August. All operation was in the SRC II mode will a full dissolver. Target conditions were 860-865°F dissolver temperature and 1900 psig dissolver pressure. The slurry preheater outlet temperature was held at 800°F, except for upsets and two short periods of 750°F operation.

Slurry preheater problems caused three process upsets. It took one hour to relight the heater after a power dip August 21. This resulted in a 5 1/4 hour coal outage. A heater flame out August 23 and a partial flame out August 26 caused a half hour production loss. Both occurred when nearby piping was X-rayed. Apparently the X-rays interfered with system electronics.

The slurry preheater was solvent flushed August 26 when the pressure drop became excessive. The recycle hydrogen temperature dropped below 100°F cooling the feed slurry. The increased viscosity of the feed slurry caused the pressure drop problems.

On August 27 & 28, the slurry preheater pressure drop oscillated and the outlet temperature varied uncontrollably. A false signal from recycle slurry flowmeter FT-2482 caused low recycle slurry flow. The resulting high coal concentration slurry with a low total flow caused the preheater upset.

Dissolver B was inspected as well as could be done without entering the vessel. Several cracks were found in the 347 weld overlay material. GS&TC metallurgists believe that these cracks were formed during manufacture of the vessel because they only occur at weld arc starts, and the bottoms of the cracks are rounded rather than sharp edged.

C. Mineral Separation and Drying - (Area 03)

Circulation of filter "B" gas loop at operating temperature was discontinued on August 25. This was done in anticipation of engineering modifications to the filter.

The high pressure water booster pumps were again responsible for a single coal feed curtailment during the month. This occurred on August 30 and resulted from a packing failure on "A" pump and an electrical failure on "B". The total curtailment attributable to the 03 area was 0.20% for the month.

A 3"x3" sample was removed from the 304 SS mineral residue dryer drum and sent to GS&TC to determine if a badly carburized section of the drum would have to be replaced. The sample was found to be carburized to a depth of 25% of the 3/8" wall thickness. The metallurgists advise that the dryer can be safely operated at normal operating conditions, however, every effort must be made to avoid high temperature excursions. Also, special precautions must be taken when rewelding the area where the sample was removed to avoid cracking in the carburized area of the metal. Engineering is studying various means to make the dryer operation more reliable.

D. Solvent Recovery - (Area 04)

The new vacuum flash drum operated without major problems during the month. There was no production curtailment attributable to this system.

The old vacuum flash heater was finally unplugged on August 12. Attempts to clear the coil by spalling were unsuccessful and it was finally necessary to initiate an air burn. This in conjunction with a quick-reversing steam decoking manifold, fabricated for this problem, resulting in dislodging the plug. A pressure drop survey of the heater coil and downstream piping was then conducted per procedure established January 19, 1976. The pressure drop through the piping from the preheater coil inlet to the vacuum flash drum shows some improvement over the 1976 pressure survey.

E. Gas Recompression and Naphtha Unit - (Area 05)

Gas Recompression

Hydrogen recycle compressor "A" was taken out of service August 7 to replace the packing and scored rod. No major upset was encountered as "B" recycle compressor carried the plant load. A forty-five

minute coal outage was taken August 8 to check "B" recycle compressor valves. No other compressor problems were encountered.

Naphtha Unit

The naphtha unit was out of service August 8 and 9 due to a piping weld leak. Plant operation continued at reduced recycle hydrogen purity, but no major plant problems were encountered. No other significant problems arose.

F. Product Solidification and Storage - (Area 08)

The Sandvik cooling belt vapor removal system failed on August 21 and was non-repairable. The Engineering Department is working to provide a new and more efficient system. With the above noted exception, the area operated without major upset during the reporting period.

G. Waste Treatment - (Area 09.1)

The unit continues to operate at less than normal efficiency. A comprehensive program is underway to correct the many problems in this area.

H. Tank Farm - (Area 09.2)

Storage tank 008 was emptied and a new manway pressure vent was installed. The new 1300 barrel storage tanks are mechanically complete. They are being filled with pure nitrogen by water displacement in preparation for SRC II liquid storage.

I. Boiler System - (Area 09.4)

Boiler "A" was in service with "B" on standby. A production curtailment of 0.03% was attributed to the area during August. During record high ambient temperatures the feedwater pump tripped from overheating, resulting in low steam heating pressure and a momentary curtailment of coal feedstock.

J. Gas Systems - (Area 05)

Hydrogen Unit

The hydrogen unit developed three small leaks this month. Repairs will not be made until the plant is shut down. Reformer process gas flow tripped out several times. The outages were short and no plant upsets were encountered. The cause of the process gas dropout has not been pinpointed.

Stretford Unit

The Stretford unit was on line all month except for two short periods. The unit was bypassed a few hours while the melter charge pump was being repaired, and again for a short time while a solution chemical imbalance was corrected.

K. Hot Oil Unit - (Area 09.8)

On August 12 continuing problems with the Hot Oil heater were traced to an erroneous level transmitter on the surge tank. This resulted in insufficient pump suction head which tripped the fired heater on low flow. Sufficient fresh Hot Oil was pumped to the surge tank to establish the required NPSH.

In order to correct excessive polymer levels in the system, the reclaimer unit was returned to operation on August 15.

III. Process Analytical Data

A. Coal Receiving and Preparation (Area 01) and Preheating and Dissolving (Area 02)

The average analysis of raw coal was 8.75% ash and 6.65% moisture. The average analysis of the dehumidified pulverized coal during the month was:

Carbon	71.40%	-40 mesh	99.50%
Hydrogen	5.22%	-100 mesh	98.73%
Nitrogen	1.41%	-140 mesh	95.55%
Sulfur	3.22%	-200 mesh	79.42%
Oxygen (by difference)	8.47%		
Moisture	0.31%		
Ash	9.97%	Total Iron	1.53%

The analysis of forms of sulfur done on weekly composites of pulverized coal was

	8-6-77	8-13-77	8-20-77	8-28-77
Pyritic Sulfur	1.46%	1.44%	1.55%	1.48%
Sulfate Sulfur	0.15%	0.34%	0.16%	0.03%
Organic Sulfur	1.48%	1.47%	1.32%	1.76%
Total Sulfur	3.09%	3.25%	3.03%	3.27%

The slurry from the slurry blend tank averaged 10.05% ash.

The average analysis of the recycle stripper bottoms was:

Water	0.13%
Naphtha	0.15%
Middle Distillate	12.64%
Heavy Distillate	33.15%
Vacuum Bottoms (PI included)	53.93%
Pyridine Insolubles (as rec'd)	19.23%
Ash in Pyridine Insolubles	63.24%
Total Iron (as rec'd)	1.86%

B. Solvent Recovery - (Area 04)

Typical analyses of liquid products based on ASTM D-86 distillation data were:

Laboratory Distillation Fractions	Pilot Plant Products		
	Naphtha	Middle Distillate	Heavy Distillate
Naphtha up to 380°F	96%	10%	0%
Middle Distillate 380 to 550°F	4%	90%	10%
Heavy Distillate 550 to 850°F	0%	0%	90%

C. Product Solidification - (Area 08)

The plant vacuum bottoms produced this month averaged 22.65% ash with a fusion point of 370°F. Elemental analyses typically were: 69.40% carbon, 4.15% hydrogen, 1.08% nitrogen, and 2.46% sulfur.

D. Process Waste Disposal - (Area 09.1)

During the month, average analyses of waste water units were:

	Bio Unit Feed	Bio Unit Effluent	Plant Effluent (Composite)
pH	7.2	7.3	7.2
Total Suspended Solids, ppm	123	75	2
Phenol, ppm	109	16	0.6
Chemical Oxygen Demand, ppm	1132	343	41
Biological Oxygen Demand, ppm	134	54	13

IV. Process Evaluation

A. Material Balance Runs

Four SRC II material balance runs (77SR-4 through 7) were completed during August using Western Kentucky coal feed. Actual conditions achieved during these runs and product yields obtained are summarized in Table I. In three of the runs (77SR-4, 77SR-5, and 77SR-6) coal feed rates exceeded 25 lbs dehumidified coal/hr ft^3 dissolver volume. All four runs continued to completion but at the highest coal concentration levels it was felt that the mechanical limitations of the existing coal-slurry mixing system had been reached. A run at 35% coal concentration and 0.8 hours nominal dissolver slurry residence time was attempted, but frequent slurry circulation pump flushing was required, and the run was aborted. During material balance runs 77SR-5 and 77SR-6, problems were encountered with process gas chromatography. As a result, the hydrogen consumption in both runs and the hydrocarbon gas yields in run 77SR-5

are suspected to be in error. Modifications to the gas chromatograph system were completed during run 77SR-7 and, therefore, the reported hydrogen consumption and hydrocarbon gas yields for this run are reliable.

B. Comparison of Fort Lewis SRC II Results - Western Kentucky Coal

In order to compare the results of the runs shown in Table I, it was necessary to convert the SRC produced in each run to a common fusion point. (The variability in fusion points is shown as the bottom row in Table I.) To convert these results to a common basis, a fusion point of 400°F was chosen, and the SRC yield for each run was converted, based on the correlation presented in the July Progress Report. The corrected SRC and solvent yields are shown in parentheses in Table I.

Analysis of these data shows that increased coal concentration in the feed slurry results in increased yields of SRC and insoluble organic matter and decreased distillate yield. The dependence of SRC and distillate yields on coal concentration is non-linear. This non-linearity is at least partly due to the fact that in the Fort Lewis system, complete recycle has not been attained to date at coal concentrations in excess of 30%. Some solvent must be added to the coal slurry to permit steady operation at these conditions. Addition of solvent has the effect of reducing the recycle ratio which can be represented by the concentration of iron in the feed slurry. Actually, the recycle ratio could also have been expressed in terms of other variables but iron was chosen because it is thought to be a catalytic species. Nevertheless, the results show that at higher iron concentration in the feed slurry (higher recycle ratio), increased distillate yields and decreased SRC yields result. A further conclusion from the results of these runs is that increasing the dissolver residence time results in an increase in distillate yield and a decrease in SRC yield.

Additional studies are planned to investigate the effects of preheater temperature and dissolver temperature on the SRC II process. In addition, current plans call for the introduction of Illinois #6 coal (River King Mine) into the system in early September. A test is planned to watch the effects of Illinois coal as it is introduced and to determine any variances from the results achieved for Western Kentucky #9 coal.

V. Special Projects

EPRI Filter

The Johns-Manville filter arrived by truck in good shape August 16. Total estimated cost of the installation including Phase II engineering and some form of parallel dry cake removal system is now $1,146,000.

Lummus Deashing System

Engineering is proceeding on the Lummus Deashing System. Purchase orders have been issued for all pumps and all major vessels (except the settler). A letter stating that the Total Installed Cost is expected to be $3.2 to $3.5 million was received from Lummus August 11.

VI. Merriam Laboratory

Results for eight SRC II runs are reported. These runs were exploratory runs to determine the suitability of coals other than the normally used Kentucky coal for the SRC II process. Two Pittsburgh Seam coals (Consol Ireland and Blacksville No. 2 Mines) and one Illinois No. 6 coal were investigated. Run conditions and results are summarized in Table II. In some cases reported yields are subject to a higher than normal uncertainty due to experimental problems associated with slurry pumping, but the results satisfactorily demonstrate the operability of the SRC II process with the coals investigated.

The Pittsburgh seam coal investigated initially (Ireland Mine, GU 181R) in this period was found to be as fully satisfactory for SRC II operation as the Kentucky coal. In fact, yields with this coal were found to be more favorable than those obtained with a Kentucky coal under the same conditions. For example, organic residue yield (SRC + insoluble organic matter) was 28.0% for the Pittsburgh Seam coal in comparison to 31.3% for Kentucky No. 9 and 14 coal (GU 180R). This Pittsburgh Seam coal is of high iron content (2.5% iron vs 2.0% iron for the Kentucky coal).

The other Pittsburgh Seam coal (Blacksville No. 2 Mine, GU 184R) was found to be less reactive with an organic residue yield of 37.5% being observed under the same conditions. The Blacksville coal was lower in iron content and higher in apparent rank (lower oxygen content) than the Ireland coal. The importance of these factors is, as yet, uncertain.

The initial run with the Illinois No. 6 coal was made under the same conditons (GU 182R). Reactivity of this coal was intermediate with an organic residue yield of 33.5% being observed. Reduction of the coal concentration in the feed slurry from 30 to 25% and an increase in the temperature from 455 to 460°C resulted in a decrease in organic residue yield to 27.2%. Four additional runs explored operability and yield at a higher coal concentration (35%) and two levels of temperature (455, 460°C).

VII. Future Plans

In the next period the SRC Pilot Plant will continue to develop the SRC II Process on the Illinois No. 6 coal from the River King Mine. The Merriam Laboratory will make exploratory SRC II runs using the Pittsburgh Seam coal from the Blacksville No. 2 Mine. Operability and yields will be explored over process conditions achievable at Fort Lewis with emphasis on conditions yielding organic residues compatible with hydrogen generation needs.

VIII. Travel

A. Fort Lewis

Trip No.	Number Attending	Destination	Purpose
1	1	Denver, CO	Witness loading of the J-M Filter
2	2	Newark, NJ Washington, D.C.	Lummus Contract Meetings
3	1	Wilsonville, AL	Wilsonville Coordination Meeting
4	1	Washington, D.C.	Coal Liquefaction Material Balance Procedures Meeting
5	1	Denver, CO	Maintenance Management School
6	1	Merriam, KS	Maintenance Assist for Merriam Lab

B. Merriam

1	1	Decatur, IL	Meeting with IBT
2	1	Seattle, WA	ERDA Meeting
3	1	Washington, D.C.	Procurement Meeting
4	1	Chicago, IL	ACS Meeting - Present paper on SRC II

TABLE I

RESULTS OF SRC II MATERIAL BALANCE RUNS USING KENTUCKY #9 AND #14 COAL

Conditions	Run 77SR-1	Run 77SR-2	Run 77SR-3	Run 77SR-4	Run 77SR-5	Run 77SR-6	Run 77SR-7
Dehumidified Coal Feed, lb/hr	1730	1626	1973	2467	2591	2314	1984
Feed Slurry Composition, wt %							
Coal	26.1	24.2	24.6	29.9	30.6	33.5	29.5
Solvent	37.8	38.4	31.1	28.4	41.6	35.9	33.4
SRC	22.4	27.3	27.8	26.3	20.2	19.8	23.9
Ash	8.6	10.3	10.6	9.2	5.1	6.0	8.2
IOM	5.1	4.8	5.9	6.2	2.5	4.8	5.0
Iron	2.0	2.1	1.92	1.91	1.36	1.34	1.57
Hourly Space Rates, lb/hr ft^3							
Coal	18.8	17.7	21.4	26.8	28.2	25.2	21.6
Total Slurry	72.0	73.0	87.2	89.8	92.0	75.1	73.1
Nominal Dissolver Res. Time, hrs	1.00	0.98	0.82	0.80	0.78	0.96	0.98
Hydrogen Purity, mole %	94.0	90.5	92.2	90.4	91.8	91.0	89.8
H_2/Slurry Ratio, lb H_2/100 lb Slurry	5.70	5.82	5.50	5.67	5.14	5.66	5.93
Dissolver Pressure, psig	1912	1913	1896	1899	1911	1905	1920
Average Dissolver Temp., °F	861	858	859	862	857	859	861
Yields, % M.F. Coal							
H_2 (consumption)	-3.3	-4.7	-3.1	-3.7	-2.0	-2.1	-3.6
Hydrocarbon Gas (C_1C_4)	13.9	14.3	14.9	13.9	10.0	14.0	16.6
Total Distillate (to 850°F)	38.4 (41.8)	35.6 (41.4)	32.6 (36.2)	40.5 (41.7)	34.3 (39.2)	30.8 (31.3)	36.7 (39.2)
SRC	20.6 (17.2)	19.7 (13.9)	20.2 (16.6)	22.6 (21.4)	28.4 (23.5)	26.9 (26.4)	20.2 (17.7)
ASH + IOM	12.4	14.2	14.5	15.7	12.5	15.4	14.1
Liquid Distribution, % of Total Liquid							
Naphtha (to 380°F)	25 (23)	25 (20)	18 (16)	25 (24)	24 (21)	31 (30)	28 (26)
Middle Distillate (380°-550°F)	60 (55)	52 (45)	49 (44)	45 (44)	46 (40)	57 (57)	48 (45)
Heavy Distillate (550°-850°F)	15 (22)	23 (35)	33 (40)	30 (32)	30 (39)	12 (13)	24 (29)
Total Liquid Produced, Bbl/Ton M.A.F. Coal	2.45 (2.67)	2.45 (2.85)	2.11 (2.34)	2.64 (2.72)	2.17 (2.48)	2.01 (2.05)	2.41 (2.57)
M.A.F. Conversion	94.2	94.2	94.0	92.8	94.8	91.6	93.4
Average Fusion Point of SRC, °F	345 (400)	310 (400)	345 (400)	384 (400)	336 (400)	394 (400)	362 (400)

(Yields in parenthesis are the result of correcting SRC yield to correspond to a 400°F Fusion Point)

TABLE II

Summary of SRC II Process Conditions and Yields
(Merriam Laboratory)

	GU 181R	GU 182R	GU 183R	GU 184R	GU 185R	GU 186RA	GU 186RB	GU 186RC	GU 186RD
Conditions									
Coal Used (Mine)	Pittsburgh Seam (Ireland)	Illinois No.6 (River King)	Illinois No.6 (River King)	Pittsburgh (Blacksville)	Illinois No. 6 (River King)				
Nominal Residence Time, hr	0.99	0.98	1.01	1.01	1.00	1.00	0.98	0.99	0.99
Coal Feed Rate, lb/hr/ft^3	21.8	22.0	17.7	21.7	25.1	25.3	25.6	25.3	25.4
Slurry Composition									
% Coal	30.0	30.0	25.0	30.0	35.0	35.0	35.0	35.0	35.0
% UFCS	70.0	70.0	75.0	70.0	65.0	65.0	65.0	60.0	60.0
% Recycle Solvent	--	--	--	--	--	--	--	5.0	5.0
Hydrogen Feed Rate									
Wt % based on slurry	4.59	4.54	4.76	4.63	4.65	4.63	4.57	4.62	4.61
MSCF/ton of coal	57.6	57.0	71.7	58.1	50.1	49.8	49.2	49.7	49.6
Nominal Dissolver Temperature, °C	455	455	460	455	460	450	455	455	460
Pressure, psig	1900	1900	1900	1900	1900	1900	1900	1900	1900
Yields, wt % based on coal									
H_2O	5.7	8.2	6.2	3.4	B	5.9	7.8	7.8	8.1
C_1-C_4	12.9	10.8	12.2	12.0		11.7	12.4	11.2	11.2
CO, CO_2, H_2S, NH_3	4.6	4.3	5.4	3.1	R	4.9	4.8	4.4	4.5
C_5+ (gas)	7.2	3.2	5.4	4.4	E	6.5	7.4	5.2	4.5
Naphtha, light oil, <249°C	6.0	6.7	8.3	6.7		9.3	8.4	9.2	9.9
Heavy distillate, >249°C	25.5	24.4	26.4	22.3	A	24.8	22.5	17.2	17.1
Total Oil (C_5-heavy distillate)	38.7	34.3	40.1	33.4	K	40.6	38.3	31.6	31.5
SRC	22.1	27.4	20.6	28.2		21.6	20.2	28.8	27.0
Insoluble Organic Matter	5.9	6.1	6.6	9.3	D	6.3	7.6	7.6	8.6
Ash	13.9	12.3	12.9	14.6	O	12.5	12.6	12.7	12.8
Total	103.8	103.4	104.0	104.0	W	103.5	103.7	104.1	103.7
H_2 Reacted					N				
gas balance	3.8	3.4	4.0	4.0		3.5	3.7	4.1	3.7
product analysis									
MAF Conversion	93.21	92.99	92.38	89.11		92.78	91.41	91.25	90.18