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SOLVENT REFINED COAL (SRC) PROCESS

Monthly Report for the
Period November 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 Department of Energy during the month of November 1977. The Fort Lewis Pilot Plant processed coal during 28 days of SRC II operation in November. The feed coal on November 1 was Illinois No. 6 coal from Peabody's River King Mine. On November 2, the feed was switched to Pittsburgh seam coal from Consol's Blacksville No. 2 Mine. Four material balance runs were completed on the Pittsburgh seam coal. At the Merriam Laboratory three additional SRC II runs were completed on the Blacksville coal. These provided information on the effect of varying dissolver temperature and the effect of dissolver residence time.

I. Summary

Five hundred thirty-four tons of coal were processed during 28 days of SRC-II operation in November with coal being injected 81.5% of the time. On November 2, the coal feed was switched from Illinois 6 to Pittsburgh seam coal from the Blacksville No. 2 Mine. Four material balance runs were completed on the Pittsburgh coal.

The metering of slurry flow rates continued to be a problem in November. This resulted in the material balance runs being made at lower coal concentrations and shorter residence times than planned. The new quadrant-edged orifice meter responded well to changes in flow, but the actual rates were not known until after the runs had been completed and a meter proving test had been run.

Solids buildup on the balls and seats of the slurry charge pump checks was a recurrent problem with the Pittsburgh coal. Switching from the new slurry mix system to the original eductor system had no apparent effect on this problem.

To facilitate the handling of the Pittsburgh coal, two steam-heated dryers were installed, the pulverizer inlet chute was modified, and the primary crusher was recommissioned to break up the large lumps.

A 4-foot carburized section of the mineral residue dryer drum was cut out, and a new section was installed.

Compressor valve spring failures were a major problem in November. New springs of Inconel 750X are being fabricated in an attempt to improve compressor reliability.

The new EPRI filter "C" was set in place, and the bridge crane was installed above the filter. Completion of the installation is expected by March 10.

Field construction for the Lummus antisolvent deashing system is scheduled to start by December 15.

Three additional SRC II runs were made with the Pittsburgh seam coal from the Consol Blacksville Mine No. 2 at the P&M Laboratory at Merriam. These indicated that light gas yields and insoluble organic material yields increased significantly with increasing temperature while SRC and total distillate yield decreased.

Increased residence time was seen to increase light hydrocarbon yield, to increase total distillate yield, to decrease SRC yield, and to have no significant effect on insoluble organic matter yield.

II. Pilot Plant Operations, Engineering, and Maintenance

A. Coal Receiving and Preparation - (Area 01)

Five hundred thirty-four tons of coal were processed during 28 days of SRC II operation in November with coal being injected 81.5% of the time. On November 2, the coal feed was switched from Illinois 6 to Pittsburgh seam coal from Consol's Blacksville No. 2 Mine. All operation through the remainder of the month was with the Pittsburgh coal.

Plugging in the gravimetric feeder and in the pulverizer air lock and inlet chute was a major problem early in the month. This problem was eliminated by drying the feed coal to about 5% moisture content, modifying the pulverizer inlet chute, and using the primary crusher to break up the large lumps.

The Durco circulating pumps performed satisfactorily during the month. Two seal failures were experienced, but these were attributed to inadequate seal flush and improper installation.

The preheater charge pumps were a source of recurrent problems in November. The pumps were opened seven times to remove the buildup of solid deposits on the balls and seats of the check valves. The suction piping was modified to permit circulation through the pump suction manifolds, but no marked improvement was noted. The pump packings were also replaced seven times during the month. The packing problem was attributed to inadequate packing flush which is generally limited to about 200 lb/hr.

The modified new mixing system was in operation November 3 through 9 with only one brief outage caused by agitator binding. However, this system was suspected as a possible cause of the solids buildup in the charge pump checks. The eductor mixing system was returned to operation for the remainder of the month, but no effect of the mixing method on solids buildup was noted.

Recycle slurry flow metering continued to be a problem. The reliability of the liquid purge flows to the new quadrant-edged orifice meter was greatly improved by the installation of larger metering valves. The meter responded to changes in flow rate, but the absolute flows were not known until after a meter proving test in December.

The installation of a used steam-heated pulp dryer over the track hopper for drying the feed coal was completed on November 2. Tests showed that the coal could be dried to about 5% moisture content at rates up to about 3000 lb/hr. The installation of a second dryer was completed on November 16.

B. Slurry Preheating and Dissolving - (Area 02)

Area 02 processed coal slurry 28 days in November. All operation except five hours during a plant startup was in the SRC II mode. All operation was with a full dissolver and with a preheater outlet temperature of 800°F. Dissolver conditions varied from 860°F and 1900 psig to 870°F and 2000 psig.

An emergency shutdown occurred on November 10 when a leak developed in the body of a 2-inch, high pressure, Rockwell-Edwards globe valve in the line from the bottom of the high pressure flash drum to the flare. Subsequent inspection showed that a high pressure slurry leak to the flare had eroded through the valve seat and the stainless steel body. The valve was sent to Oak Ridge National Laboratories for a detailed failure analysis. Other valves in similar service were X-rayed, but results were inconclusive.

C. Mineral Separation and Drying - (Area 03)

The high pressure quench water pumps continued to be a problem in November. Unstable pressure control seems to be the basic problem. Head gaskets failed on four separate occasions during the month. A cracked weld on the pressure relief valve of quench pump "A" was also repaired. An engineering study was initiated to reevaluate the pump pressure control system.

The pressure control loop on the oil-water separation system was steam traced to eliminate plugging by ammonium salts.

A 4-foot section of the mineral residue dryer drum, including the entire carburized section, was replaced in November. End seal repairs are in progress.

Work on the installation of Stellite agitator and drum bearings in filter "B" was almost complete at the end of November.

D. Solvent Recovery - (Area 04)

The new vacuum flash system continued in service throughout the month with no major problems. The handling of vacuum bottoms product containing as much as 30% ash and 50% total solids was demonstrated.

The vacuum flash drum Kay-Ray level instrument was inoperable all month; however, a differential pressure level transmitter performed satisfactorily. The pressure drop across the demister pad was negligible, indicating no buildup of heavy material.

E. Gas Recovery and Recompression - (Area 05)

1. Gas Recompression

Valve spring failures were experienced in the fresh hydrogen

compressor and both recycle compressors in November. These springs are made of carbon steel and may be subject to fatigue in the presence of traces of H_2S, CO, and CO_2. New springs of Inconel 750X are being fabricated in an attempt to increase compressor valve reliability.

Other compressor problems included malfunctions of the high temperature shutdown circuit and the lubricating oil heater on the fresh hydrogen compressor and two packing failures on recycle compressor "B".

2. Naphtha Absorption Unit

In early November, the naphtha unit was charged with middle distillate instead of naphtha in an attempt to reduce foaming and carryover of liquid into the recycle hydrogen compressors. No foaming problems have been experienced with the middle distillate; however, it has been necessary to increase the liquid circulation rate in order to maintain recycle hydrogen purity at 90%.

III. Process Analytical Data

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

The average analysis of the raw Pittsburgh seam coal fed during November showed 11.39% ash and 5.25% moisture. Mineral and petrographic analyses were obtained on a composite sample, and results are shown below.

Mineral Analysis of Ash from Pittsburgh Coal, Weight %

Silica, SiO_2	47.83
Alumina, Al_2O_3	20.22
Titania, TiO_2	0.88
Ferric oxide, Fe_2O_3	17.55
Lime, CaO	6.77
Magnesia, MgO	0.93
Potassium oxide, K_2O	1.59
Sodium oxide, Na_2O	0.94
Sulfur trioxide, SO_3	1.58
Phosphorus pentoxide, P_2O_5	0.25
Undetermined	1.46
Total	100.00

Petrographic Analysis of Pittsburgh Coal, Volume %

Vitrinoids	72.9
Exinoids	6.0
Resinoids	Nil

Reactive Semifusionoids	0.2
Total Reactive Constituents	79.1
Inert Semifusionoids	0.5
Micrinoids	7.7
Fusinoids	3.1
Mineral Matter	9.6

The average analysis of the dried, pulverized coal was:

Carbon	72.61%	-40 mesh	99.36%
Hydrogen	4.91%	-100 mesh	95.61%
Nitrogen	1.34%	-140 mesh	91.35%
Sulfur	2.89%	-200 mesh	75.10%
Oxygen (by difference)	5.94%		
Ash	12.12%		
Moisture	0.19%	Total Iron	1.63%

The analyses of weekly composites of pulverized coal for forms of sulfur were:

	11-6-77	11-13-77	11-20-77	11-27-77
Pyritic Sulfur	1.67%	1.73%	1.85%	1.82%
Sulfate Sulfur	0.05%	0.03%	0.04%	0.01%
Organic Sulfur	1.66%	1.16%	0.78%	0.92%
Total Sulfur	3.38%	2.92%	2.67%	2.75%

B. Solvent Recovery - (Area 04)

Typical analyses of the SRC II liquid products from Pittsburgh coal are shown below. Boiling range data are based on ASTM D-86 distillations.

	SRC II Liquids from Pittsburgh Coal		
	Naphtha	Middle Distillate	Heavy Distillate
Naphtha (IBP-350°F), Vol.%	100	6	0
Middle Distillate (350-550°F), Vol.%	0	77	1
Heavy Distillate (550-850°F), Vol.%	0	17	99
Specific Gravity, 60/60°F	0.803	0.973	1.074
Viscosity @ 100°F, cSt	0.66	2.97	50.09
Pour Point, °F	---	-49	25
Flash Point, °F	---	160	---

Typical elemental analyses of the liquid products were:

	Naphtha	Middle Distillate	Heavy Distillate
Carbon, Wt %	84.52	85.91	89.43
Hydrogen, Wt %	12.40	9.09	7.40
Nitrogen, Wt %	0.50	0.92	1.20
Sulfur, Wt %	0.42	0.22	0.42
Oxygen (by difference), Wt %	2.16	3.86	1.55

C. Product Solidification - (Area 08)

During November, the plant vacuum bottoms product had an average ash content of 23.32% and fusion point of 351°F. Typical elemental analyses showed 68.08% carbon, 3.68% hydrogen, 1.41% nitrogen, and 2.18% sulfur.

D. Process Waste Disposal - (Area 09.1)

Average analyses of waste water streams in November were:

	Bio-Unit Feed	Bio-Unit Effluent	Plant Effluent (Composite)
pH	7.2	7.0	7.1
Phenol, ppm	65.2	0.17	0.03
Chemical Oxygen Demand, ppm	1333	194	25.4
Biological Oxygen Demand, ppm	172	10.5	9.7
Total Suspended Solids, ppm	55	37	5.6

E. Special Studies

Because of the difficulty experienced in unloading the wet, fine, Pittsburgh seam coal, a brief study was conducted to determine the extent of drying required to avoid handling problems. A representative sample taken from the storage pile contained 8.6% moisture and 72.5% of material smaller than one-half inch mesh. The slump of 3-inch diameter by 5-inch high cylinders of partially dried Pittsburgh coal was compared with that of Illinois coal. Results indicated that the Pittsburgh coal should be dried to about 4-5% moisture content to avoid plugging problems in the feed system.

IV. Process Evaluation

Three material balance runs (77SR-12, 77SR-13, and 77SR-14) were completed in November with Pittsburgh seam coal from the Blacksville No. 2 Mine. A fourth run (77SR-15) was made early in December. Operating conditions and preliminary results for all four runs are reported in Tables 1 and 2, respectively.

Prior to the start of these runs, modifications were made to the gas chromatograph system to eliminate many of the gas balance problems experienced in previous runs, and the results appeared to be in good order.

The runs were designed for a 25% coal concentration in the feed slurry and a nominal dissolver residence time of one hour. Unfortunately, slurry flow metering problems resulted in a higher recycle slurry flow rate than expected. This caused the coal concentration, residence time, and hydrogen-to-slurry ratio all to be lower than originally planned. Results of meter proving tests early in December were used to obtain the corrected values reported in Table 1. Individual aspects of each run are discussed below.

1. Run 77SR-12

The first SRC II material balance run with Pittsburgh seam coal was made at a dissolver temperature of approximately 860°F and pressure of 1900 psig. The yields from this run, shown in Table 2, indicate that SRC and IOM yields were higher than would be expected at similar conditions with western Kentucky or Illinois 6 coals. In addition, the distillate yield was lower with the Pittsburgh coal, and the gas yield was higher.

2. Run 77SR-13

In this run, an attempt was made to achieve more desirable product yields by increasing the system pressure from 1900 to 2000 psig. The dissolver temperature remained at 860°F. An attempt was also made to reach maximum hydrogen purity (and thus maximum hydrogen partial pressure at the dissolver inlet) to achieve maximum conversion to distillate products. The yields shown in Table 2 indicate that this change was successful in increasing distillate yield and decreasing gas and IOM yield. The SRC yield for run 77SR-13 appears to be slightly greater than in the previous run, but this difference is within the accuracy of the yield determination procedure.

3. Run 77SR-14

This run was designed to study the effect of higher dissolver temperature (870°F) on the Pittsburgh coal yield structure. The preliminary results (Table 2) show that the increase in temperature caused a decrease in residual SRC yield. The distillate yield during run 77SR-14 was not significantly different from that for the previous run; however, the water plus loss yield was significantly higher than in the previous run. Since water yield would not be expected to account for the entire difference, part of the increase in the water plus loss yield may have been attributable to unrecovered gas or distillate. Therefore, it is possible that the gas or distillate yields for run 77SR-14 may have been higher than shown in Table 2. Completion of the yield normalization procedure for these runs will result in product distributions which can be more readily compared.

4. Run 77SR-15

In run 77SR-15, reactor temperature was decreased (to 837°F) to complete the study of the effect of temperature on product yields.

During the run, the distillate returning from the No. 3 oil-water separator to the slurry recycle stripper was decreased as much as possible in order to increase the solids concentration in the reaction system. This move resulted in a decreased SRC yield; however, further deletion of distillate was avoided when the concentration of vacuum bottoms (SRC + IOM + Ash) in the recycle slurry approached 70% by weight.

Liquid yield data reported for run 77SR-15 are considered less accurate than normal since the run lasted for only 38.4 hours compared to the typical 70-hour material balance period. Liquid distribution was also affected by several large plant inventory transfers made after run 77SR-15 began. As a result, the actual distribution of liquid products obtained may be different from that shown in Table 2. The gas and vacuum bottoms (SRC, Ash and IOM) yields for this run were obtained during a period when the plant was well lined out, and should be an accurate representation of the yields of those components at the reaction conditions of run 77SR-15.

Comparison of the gas and vacuum bottoms yields for run 77SR-15 with those of the three previous mass balance runs shows a distinct trend towards lower hydrocarbon gas and IOM yields and a higher SRC yield with lower temperatures. This suggests a decreased conversion of heavy SRC-like material to lighter components such as hydrocarbon gas and naphtha, and a decrease in the formation of IOM (coke-like material) in the dissolver.

V. Special Projects

A. EPRI Filter "C"

Engineering drawings for this project are essentially complete, and requisitions have been written for 95% of the material needed to complete the installation. The filter was set in place and the bridge crane was installed above the filter in November. Mechanical piping work is 35% complete. The electrical drawings were completed in November, and preparation of the bid package was started. Completion of the installation is expected by about March 10.

B. Lummus Antisolvent Deashing System

The engineering work on this project is about 70% complete, and all vessels, exchangers, and pumps have been ordered. Field construction is expected to start by December 15.

VI. Merriam Laboratory

Three additional SRC II runs were made with the Blacksville Mine No. 2 coal. Process conditions and result are summarized in Table 3. Although mechanical problems persisted, it was possible to approach line-out operation and obtain product distributions in all three trials. There has been some improvement in reliability of the slurry pumping system; however, fouling of the high pressure pump checks remains a problem with the Pittsburgh seam coal in SRC II operation.

Dissolver temperatures ranging from 445°C to 465°C (833°F to 869°F) have been investigated. Results are shown in Figure 1. The results at 455°C are less reliable than the others and are given less weight. The results indicate that with increasing dissolver temperature the light hydrocarbon gas yield increases, the insoluble organic yield

increases, SRC yield decreases, and total distillate oil yield decreases.

Run GU 192R was made with the nominal liquid residence time increased to 1.3 hours. Results showing the effect of residence time are shown in Figure 2. The increase in residence time produced increased light gas yield, increased total distillate oil yield, and decreased SRC yield. No significant effect of residence time on insoluble organic matter yield was observed.

VII. Future Plans

During the next period, the SRC Pilot Plant will continue process development of the SRC II process on the Pittsburgh seam coal from the Blacksville Mine No. 2. The P&M Laboratory will also continue process development with the Blacksville coal and will investigate the effect of hydrogen partial pressure on process operability and yields.

VIII. Travel

A. Fort Lewis

Trip No.	Number Attending	Destination	Purpose
1	1	Newark, NJ and Washington, DC	Lummus Contract

B. Merriam

1	1	Decatur, IL	IBT Meeting
2	1	Seattle, WA	Visit-Pilot Plant
3	1	Seattle, WA	Visit-Pilot Plant
4	1	Seattle, WA	Meeting with Subcontractors

FIGURE 1

SRC II Yields vs Temperature

Blacksville Mine No. 2 Coal, 30% Coal, 1 Hr. Residence Time

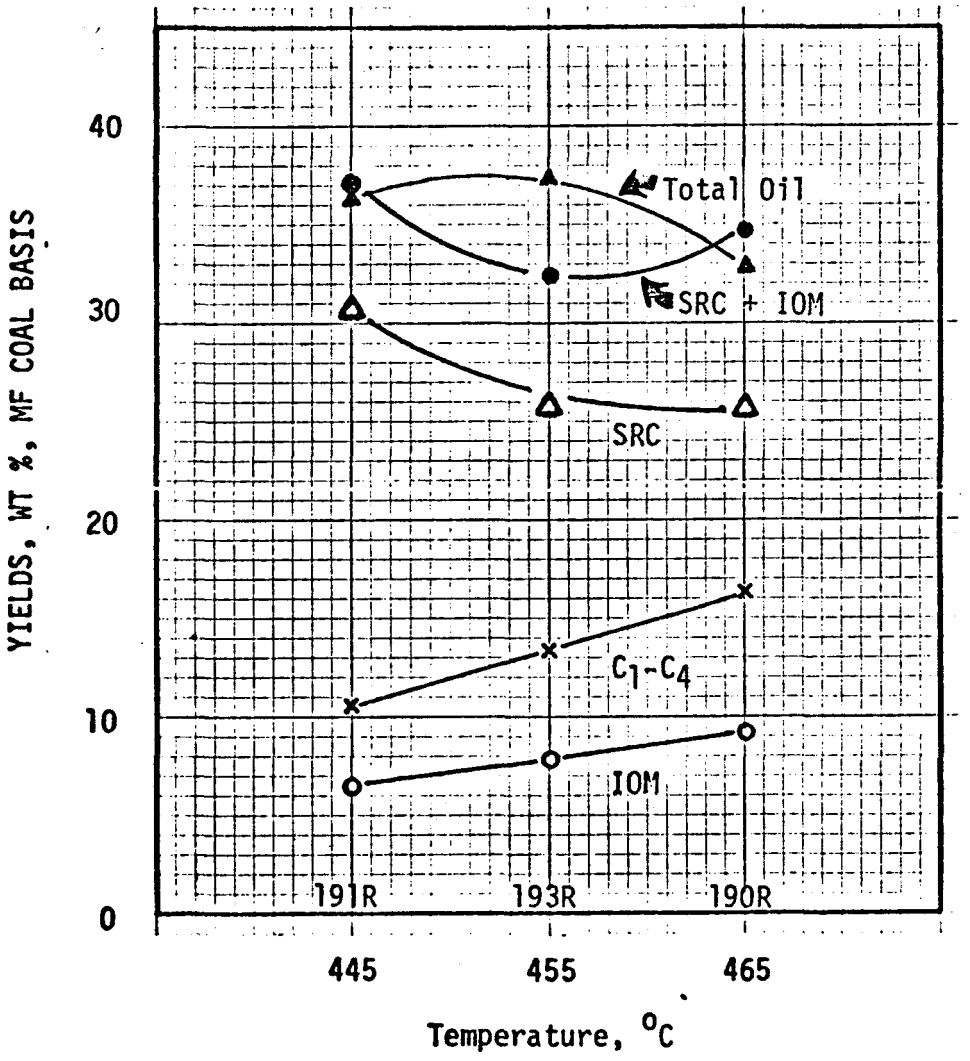

FIGURE 2

SRC II Yields vs Residence Time
Blacksville Mine No. 2 Coal, 30% Coal, 455°C

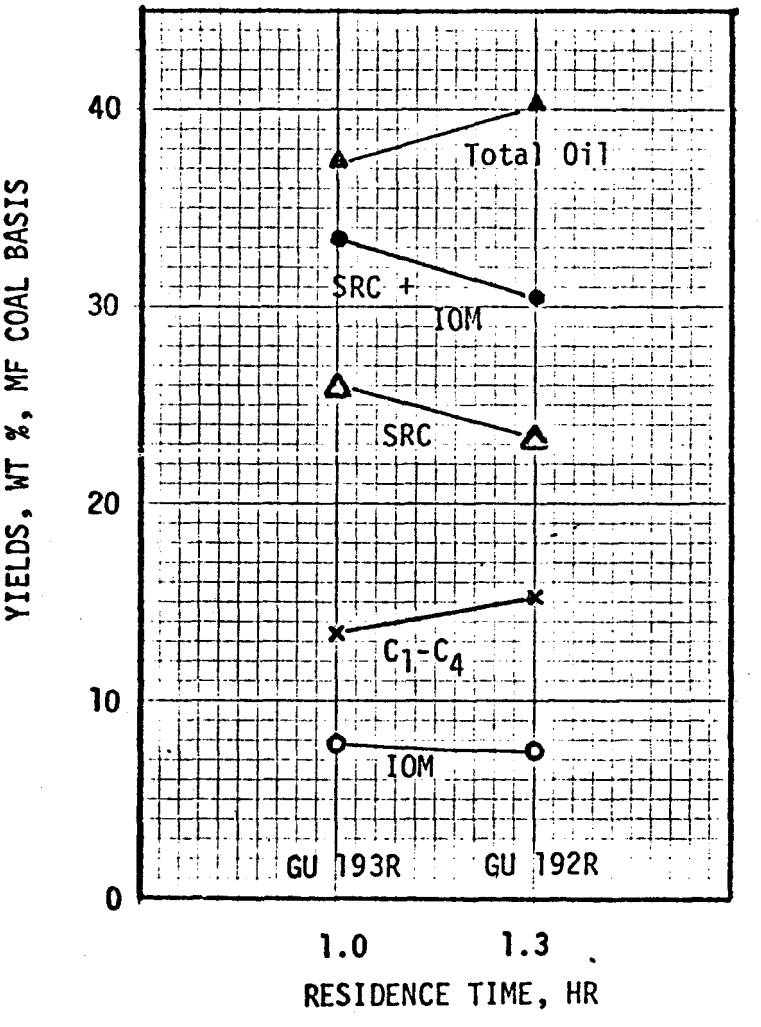

TABLE 1

MATERIAL BALANCE RUN CONDITIONS

PITT SEAM COAL (BLACKSVILLE #2)

	Run 77SR-12	Run 77SR-13	Run 77SR-14	Run 77SR-15
Raw Coal Feed, lbs/hr:	1741	1768	1745	1764
Net Dehumidified Coal Feed, lbs/hr:	1673	1675	1677	1675
Moisture Free Coal Feed, lbs/hr:	1673	1673	1675	1670
Solvent Feed (as Seal Flush), lbs/hr:	404	408	407	412
Recycle Slurry Feed Rate, lbs/hr:	5834	6372	6333	6054
Slurry Feed to Preheater, lbs/hr:	7911	8455	8417	8141
Slurry Blend Composition:				
% Coal	21.1	19.8	19.9	20.6
% Solvent	34.2	30.7	30.7	25.1
% Vacuum Bottoms	26.4	28.7	26.9	32.9
% Ash (from Recycle Slurry)	9.8	12.2	13.0	13.0
% IOM (from Recycle Slurry)	8.5	8.6	9.5	8.4
Recycle Gas to Preheater, lbs/hr:	553.2	452.3	451.9	457.8
Recycle Quench Gas, lbs/hr:				
Top	183.9	204.3	190.0	236.3
Middle	0	0	0	0
Bottom	0	0	0	0
Recycle Purge Gas, lbs/hr:	22.4	31.7	29.3	31.7
Total Recycle Gas (Mass), lbs/hr:	759.5	688.3	671.2	725.8
Total Recycle Gas (Volume), SCFH:	70992.4	76404.1	74183.1	88144.3
Recycle Hydrogen Purity % (Mole):	89.2	92.9	93.1	94.5
Hydrogen to Slurry Ratio: lbs H_2/100 lbs slurry	4.26	4.47	4.37	5.45
Slurry Heater Outlet Temperature, °F:	813	808	810	811
Dissolver Pressure, psig:	1909	2012	2006	2007
Average Dissolver Temperature, °F:	862	862	870	837
Feet from Dissolver Inlet				
4	859	858	865	837
8	862	861	868	839
12	863	863	871	838
16	864	864	872	837
20	864	864	872	836
24	862	862	870	834
28	863	863	871	835

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TABLE 2

MATERIAL BALANCE RUNS 77SR-12 THROUGH 77SR-15

PITT SEAM COAL (BLACKSVILLE #2)

PRELIMINARY RESULTS, YIELDS AS PERCENT OF M.F. COAL

Component	Run 77SR-12		Run 77SR-13		Run 77SR-14		Run 77SR-15	
H_2	-3.2		-5.0		-5.1		-3.6	
C_1	8.0		6.8		6.8		5.1	
C_2	4.1		4.2		4.2		3.0	
C_3	3.2	16.6	3.5	16.1	3.5	16.1	2.6	12.1
C_4	1.3		1.6		1.6		1.4	
CO	0.6		-0.1		-0.1		0.1	
CO_2	1.3		1.3		1.3		1.0	
H_2S	1.2		1.4		1.2		1.1	
H_2O Plus Losses	7.8		6.1		10.9		5.3	
Naphtha (to 380°F)	6.5		4.5		5.2		3.5	
Middle Dist. (380-550°F)	15.7	27.3	23.4	30.2	22.8	30.6	19.7	34.2
Heavy Dist. (550-850°F)	5.1		2.3		2.6		11.0	
SRC*	27.6		28.7		24.0		30.2	
IOM	9.7	37.3	8.8	37.5	8.9	32.9	7.7	37.9
Ash	11.1		12.5		12.2		11.9	
Total	100.0		100.0		100.0		100.0	
*Average Fusion Point °F	387		329		316		330	

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TABLE 3

	GU 191R	GU 192R	GU 193R
Conditions			
Coal Used	------- Pittsburgh Seam ------- Blacksville Mine No. 2		
Nominal Residence Time, hr	1.07	1.29	1.01
Coal Feed Rate lb/hr/ft^3	20.0	16.7	21.2
Slurry Formulation			
% Coal	30.0	30.0	30.0
% UFCS	70.0	70.0	70.0
% Recycle Solvent	--	--	--
Slurry Blend Composition			
% Coal	30.0	30.0	30.0
% Solvent (Recycle UFCS)	19.9	23.1	20.2
% SRC (Recycle UFCS)	31.6	25.7	28.1
% Ash (Recycle UFCS)	11.8	13.0	13.2
% Insoluble Organic Matter (Recycle UFCS)	6.7	8.2	8.5
Hydrogen Feed Rate			
Wt % based on slurry	5.0	6.0	4.7
MSCF/ton of coal	62.7	75.3	59.2
Nominal Dissolver Temperature, ^oC	445	455	455
Pressure, psig	1900	1900	1900
Yields, wt % based on MF coal			
H_2O	4.9	4.5	4.5
CO, CO_2, H_2S, NH_3	3.6	3.8	3.5
C_1-C_4	10.6	15.2	13.3
Naphtha (C_5-193^oC)	12.0	13.4	11.8
Middle distillate (193-249^oC)	2.9	3.5	4.0
Heavy distillate (>249^oC)	21.4	23.4	21.6
Total Oil (C_5-heavy distillate)	36.3	40.3	37.4
SRC	30.6	23.1	25.7
Insoluble Organic Matter	6.5	7.4	7.8
Ash	11.4	11.7	12.0
Total	103.9	106.0	104.2
H_2 Reacted, gas balance	3.9	6.0[a]	4.2
product analysis			
MAF Conversion, %	92.6	91.7	91.4
Lineout Index	1.06	1.01	0.94

a) Believed high due to gas leak in system.