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H-COAL™ PILOT-PLANT STARTUP-OIL STUDY

MAY 1982

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ABSTRACT

This bench-unit program simulated the Catlettsburg H-Coal™ Pilot Plant start-up procedure in Run 6, which used catalytic cracker slurry oil, an aromatic petroleum oil. The program was designed to explore the reasons for Pilot Plant operational difficulties which included reactor product instability, vacuum pumping difficulties and unexpected product yields. Startup oil, hydroclone overflow and withdrawn catalyst were obtained from the Pilot Plant for use in this run with Illinois No. 6 coal.

In this run the ratio of startup oil/coal was varied in Periods 1-10, from 2 to 0.2, while the ratio of recycle oil to coal was increased from 0 to 1.8. Recycle oils generated during this run were used as slurry oils in Periods 11 and 12. Initially, the C₄-975°F total distillate yield was 57.4 W %. As the startup oil feed was eliminated later in the run, the C₄-974°F yield fell to 45.4 W %.

Ancillary studies were performed on separator bottoms in order to establish their viscosity and settling characteristics as a function of time. Viscosity measurements were also made on bottoms containing 1-30 W % 975°F- distillate. For comparison, viscosities were measured as a function of temperature using PDU Run 5 hydroclone overflow samples which were vacuum distilled to contain <33 W % 975°F distillate.

The propensity toward carbon formation was investigated by a series of Ramsbottom carbon residue (RCR) measurements on separator bottoms which covered the range of startup oil/coal ratios from 2 to 0. Finally, microautoclave tests were performed on startup oil and hydroclone overflow oils in order to assess their ability to dissolve coal.

CONCLUSIONS AND RECOMMENDATION

CONCLUSIONS

1. The high initial total distillate yield of 57.4 W % C₄-975°F reflects the high activity of the fresh catalyst and the high startup oil/coal ratio of 2.0. By Period 12 the distillate yield fell to 45.4 W % with the startup oil/coal ratio lowered to zero and the catalyst having aged.
2. Ramsbottom carbon residue (RCR) measurements on separator bottoms demonstrate that the coking tendency is high for feed conditions in which startup oil/coal is >1.
3. Initially, the viscosity of bottoms containing 1-30 W % 975°F- distillate was comparable to PDU Run 5 hydroclone overflow samples which were distilled to the same 975°F- distillate content. Prolonged operation at feed conditions in which the startup oil/coal ratio was >1 with its inherent high coking tendency contributed to the high viscosity measured in Period 12A followed by plugging in the reactor and termination of the run.
4. In separator bottoms which were held at 400°F, over 80% of the ash settled out within the first 24 hours.
5. In separator bottoms held at 400°F the viscosity increased by only a factor of ~two over a five day period.
6. In spite of low microautoclave conversion (to THF solubles) for startup oil (51%) and hydroclone overflow oil (61%) from Catlettsburg Run 6 versus ~75% for tetralin and hydrogenated anthracene, coal conversion and yields were high with the startup oil.

RECOMMENDATION

Pilot Plant startup oil is recommended for use in the H-Coal Process provided that the period of operation for the startup oil/coal ratio of >1 be as brief as possible in order to reduce the propensity toward carbon formation.

OBJECTIVES AND SCOPE OF WORK

A bench scale run was designed in order to investigate the suitability of Pilot Plant startup oil for the H-Coal Process. Previously, Catlettsburg Pilot Plant Run No. 6 (February-April 1981) which used Ashland decant oil as part of the startup oil had experienced operating difficulties. These difficulties included reactor product instability, vacuum bottoms pumping problems and unexpected product yields. Product instability was manifested by solids accumulating in the reactor effluent separator, rapid settling of stripper bottoms in storage and hydroclone efficiency deterioration with time. In addition to the high viscosity of the vacuum bottoms, the flaked bottoms had a high distillate content. The product yields displayed high naphtha, low heavy distillate and high soluble residuum.

In order to establish the suitability of the startup oil for continued use in the Pilot Plant, Pilot Plant conditions were adhered to as closely as possible in the bench-scale operations. Startup oil (which contained coal-derived oil in addition to reclaimed decant oil), hydroclone overflow oil and withdrawn catalyst were obtained from Catlettsburg. A feed schedule was followed which started with Illinois No. 6 coal and startup distillate oil only. Subsequently, the slurry oil consisted of a startup oil HRI No. 4341/hydroclone overflow oil HRI No. 4314 mixture, hydroclone overflow oil only and finally, a combination of recycle slurry oils.

In addition to establishing the product yields for this run which utilized Pilot Plant startup oil, correlative studies were conducted in order to determine the viscosity of bottoms and their settling characteristics. A comparison was made with the performance of PDU Run 5 on Illinois No. 6 coal feed. The hydrogen donating ability of startup oil and hydroclone overflow oil from Pilot Plant Run No. 6 was assessed by microautoclave testing. The propensity for carbonaceous buildup in the liquid product was assessed by Ramsbottom carbon residue measurements made on separator bottoms.

DETAILS OF OPERATION

Bench-scale Run 177-158 was initiated as a once-through operation using Illinois No. 6 coal plus Pilot Plant distillate startup oil for startup. After one period the preheater plugged which required shutdown and system cleaning. Run 177-159 started with another catalyst charge, 65 V % of which had been withdrawn from the Catlettsburg reactor. Uninterrupted operation (except for restart in Period 3) was carried out for 12.5 periods when plugging in the reactor caused the operation to be terminated. Ten periods were conducted in the once-through mode using startup oil, startup oil/hydroclone overflow mixture and hydroclone overflow as slurry oils. Operation in Periods 11 and 12A used recycle fluids from the bench unit vacuum still overhead, filter liquid and separator bottoms.

The unit conditions and feed schedules are presented in Table 1. The analyses of the feed materials are contained in Appendix III. It is noteworthy that the startup oil contains a volatile, petroleum-derived oil, plus coal-derived liquids. Vacuum distillation of Pilot Plant startup oil (HRI No. 4341) yielded 63 V % in the vapor at 650°F compared with 60 V % for hydrogenated anthracene oil. The startup oil contains significantly more hydrogen than the hydrogenated anthracene oil, (1) 9.9 vs. 6.9 W %.

(1) Startup oil in experimental units has usually been slightly hydrotreated anthracene oil.

TABLE 1

CATLETTSBURG SUPPORT RUNS
UNIT CONDITIONS AND FEED PROGRAM

Coal: HRI 4004, Illinois No. 6, -70 mesh
 Catalyst: 177-158: 169cc HRI 3830 (fresh Co-Mo)
 218cc HRI 4340 (recovered Co-Mo)
 177-159: 156cc HRI 3830
 294cc HRI 4340
 Pressure: 2500 psig
 Reactor Volume: 1000cc

RUN	<u>177-158</u>		<u>177-159</u>					
PERIODS	S/U	1	1	2	3 (restart)	4-6	7-10	11-12A
Conditions	--	#1	--	#2	--	#3	#4	#5
Reactor Temperature °F	800-805	825	800-840	840	800-850	850	850	850
Feed, g/hr								
Coal	100-200	333	100-500	500	100-500	500	500	500
Pilot Plant Startup Oil (HRI 4341)	1000	1000	1000	1000	900-500	500	100	0
Hydroclone Overflow (HRI 4314)	0	0	0	0	500	500	900	0
Separator Bottoms	0	0	0	0	0	0	0	700
VSOH	0	0	0	0	0	0	0	100
Filter Liquid	0	0	0	0	0	0	0	200

RESULTS

YIELDS

The observed yields are summarized in Table 2. For Periods 2, 4-6, 8-10, and 11-12A the applicable startup oil/coal ratios were 2, 1, 0.2 and 0, respectively. Initially, there was a high total C₄-975°F distillate yield of 57.4 W % which fell as the startup oil/coal ratio was lowered late in the run. On total recycle in Periods 11-12A, with a startup oil/coal ratio of zero, the C₄-975°F yield was 45.4 %. It is difficult to determine the startup oil effect on product yield distribution since a competing effect of HOF feed was superimposed on the process. However, the total C₄-975°F distillate yield fell once the use of startup oil in the startup procedure was completed. Table 2 includes average yield values for PDU Run 5 on Illinois No. 6 coal. Auxiliary data for the run have been incorporated in Appendix I.

TABLE 2

YIELDS FROM RUN 177-159, W % DRY COAL

Period	RUN 177-159				
	<u>PDU 5</u>	<u>2</u>	<u>4-6</u>	<u>8-10</u>	<u>11-12A</u>
<u>PRODUCT</u>					
C ₁ -C ₃	11.2	8.2	8.9	10.0	11.6
C ₄ -400°F	18.7	16.2	15.7	19.1	15.3
400-975°F	29.1	41.2	37.3	31.9	29.9
975°F+	19.7	8.4	12.6	16.0	18.3
C ₄ -975°F	47.8	57.4	52.7	51.0	45.4
Conversion	94.7	94.7	95.4	96.0	95.2
<u>Feed g/hr</u>					
Coal		500	500	500	500
Startup Oil		1000	500	100	
HOF			500	900	
Separator Bottoms					700
VSOH					100
Filtered Liquid					200
Startup Oil/Coal		2	1	0.2	0

VISCOSITY OF SEPARATOR BOTTOMS

The viscosity stability of the separator bottoms has been determined. The rheological consistency plot (log viscosity vs log shear rate) for the representative period 2B separator bottoms is shown in Figure 1. Since the angle α with the horizontal is between 0° and -45° , the separator bottoms acts as a pseudo-plastic liquid. Consequently, one must deal with apparent viscosities which are a function of shear rate. Data are shown in Table 3 for temperatures of 122° and 155°F . These tests apply to separator bottoms which have been held at 400°F for intervals up to 120 hours. The apparent viscosity (measured twice daily) increased as the amount of startup oil decreased in the feed for Periods 2B, 6B and 9B (see Table 1). The apparent viscosity of separator bottoms increased by only a factor of $\sim$ two over a five-day period.

FIGURE 1

RHEOLOGICAL CONSISTENCY PLOT

Run 177-159 - 2B Separator Bottoms

TABLE 3

RUN 177-159 VISCOSITY DATA

Period	Sample	Date	Time	Apparent Viscosity, cp							
				122°F			150°F				
				Shear Rate (Sec ⁻¹)	79.2	39.6	15.84	79.2	39.6	15.84	
2B	Sep.Btms.	5/30	0900		18	20	30	13	16	25	
			1600		20	23	35	14	16	28	
	24 hrs	5/31	0900		19	22	34	14	18	30	
			1600		22	25	38	16	20	32	
	48 hrs	6/1	0900		21	24	34	15	19	31	
			1600		22	25	35	16	20	32	
	72 hrs	6/2	0900		23	26	35	16	19	29	
			1600		24	26	36	16	19	29	
	96 hrs	6/3	0900		24	27	38	18	22	34	
			1600		26	28	38	17	20	30	
	120 hrs	6/4	0900		26	29	38	17	20	30	
			1600		29	31	40	20	22	32	
				Shear Rate (sec ⁻¹)	39.6	15.84	7.92	39.6	15.84	7.92	
6B	Sep.Btms.	6/4	0900		90	104	125	56	71	98	
			1600		83	101	128	49	66	88	
				Shear Rate (sec ⁻¹)	20.4	10.2	4.08	20.4	10.2	4.08	
Repeat of start	24 hrs	6/5	0900		130	158	212	81	105	162	
			1600		145	168	225	94	112	162	
	48 hrs	6/6	0900		136	160	212	99	122	188	
			1600		135	160	212	84	108	150	
	72 hrs	6/7	0900		142	162	212	88	108	150	
			1600		156	178	225	96	118	162	
	96 hrs	6/8	0900		164	178	212	94	110	150	
			1600		224	248	312	129	152	212	
	120 hrs	6/9	0900		220	245	312	128	152	212	
			1600		234	260	325	129	152	212	
					Shear Rate (Sec ⁻¹)	10.2	4.08	2.04	10.2	4.08	2.04
	9B	Sep.Btms	6/8	0900		822	938	1075	350	450	575
				Shear Rate (Sec ⁻¹)	16.8	8.4	3.36	16.8	8.4	3.36	
	24 hrs	6/8	0900		780	830	950	322	365	475	
			1600		755	800	925	310	355	450	
	48 hrs	6/9	0900		732	775	875	310	355	450	
			1600		760	810	925	310	355	450	
	72 hrs	6/10	0900		782	830	950	330	375	475	
			1600		865	905	1000	332	370	475	
	96 hrs	6/11	0900		870	930	1050	345	385	475	
			1600		968	1010	1125	355	395	500	
					Shear Rate (Sec ⁻¹)						
	168 hrs	6/15	0900	off scale	1455	1600	1.68	475	520	625	
					1750	1875	2050	490	535	650	
					180°			200°F			
				880	955	1125	455	525	700		

Separator Bottoms were held at 400°F until viscosity measurements were made.

SETTLING CHARACTERISTICS OF SEPARATOR BOTTOMS - MOISTURE AND ASH DATA

Table 4 presents the results of moisture and ash measurements on Illinois No. 6 coal (HRI No. 4004) used in Run 177-159. The original ash content was ~11 W %. The settling characteristics of separator bottoms from Periods 2B, 6B and 9B, held at 400°F for intervals up to five days, are shown in Table 4. A suction device was used to draw off ~70% of the supernatant liquid before determining the ash content. The weight of sample ashed, the percentage of original sample ashed, and the percent and weight of ash collected are noted in the last three columns of Table 4.

The ashing calculations are sensitive to the percent ash in the separator bottoms. Daily analyses of separator bottoms conducted during the run reported 3.38 and 7.41 W % ash for Periods 2B and 9B. Ramsbottom carbon residue measurements cited later in this report yielded 3.08 and 8.20 W % ash for Periods 2B and 9B separator bottoms. Using the high percent ash measurement in the separator bottoms aided in the interpretation of the settling data. For separator bottoms from Period 6B the ash in the original sample at 24 hours was calculated as follows:

$$\frac{133.5 \text{ g}}{0.324} \times 0.0702 \text{ W \% ash} = 28.925 \text{ g ash in original sample}$$

After 24 hours 24.8 g ash settled out. This corresponds to 85.7 % of the ash settling out within 24 hours. In all other instances the percent of settled ash exceeded that reported in this case. It is concluded that over eighty percent of the ash settled out of separator bottoms held at 400°F within the first 24 hours. There was little change displayed in the weight of ash collected at times >24 hours.

TABLE 4

RUN 177-159 - MOISTURE AND ASH DATA

<u>Period</u>	<u>Sample</u>	<u>Date</u>	<u>Time</u>	<u>W % H₂O</u>	<u>W % Ash</u>	<u>% Of Original Sample Size(g)</u>	<u>W Ash(g)</u>
1B	Coal	5/29	0500	2.84	11.08		
4B	HRI #4004	6/2		1.30	10.45		
7B	Burning Star No. 6			1.43	11.08		
10B		6/8		1.21	10.93		

SETTLING CHARACTERISTICS OF SEPARATOR BOTTOMS

2B	Separator Bottoms	5/30	0900		3.03		
	Can No. 1	5/31	24 hrs		13.40	83.0 (24%)	11.1
	Can No. 2	6/1	48 hrs		12.83	91.5 (29.9%)	12.1
	Can No. 3	6/2	72 hrs		13.17	103.4 (26.1%)	13.6
	Can No. 4	6/3	96 hrs		13.28	91 (27.0%)	12.1
	Can No. 5	6/4	120 hrs		14.19	89.5 (26.3%)	12.7
6B	Separator Bottoms	6/4			7.02		
	Can No. 1	6/5	24 hrs		18.58	133.5 (32.4%)	24.8
	Can No. 2	6/6	48 hrs		18.72	95 (34.2%)	17.8
	Can No. 3	6/7	72 hrs		16.52	82 (39%)	13.5
	Can No. 4	6/8	96 hrs		17.54	99 (38.5%)	17.4
	Can No. 5	6/9	120 hrs		18.78	125 (34.4%)	23.5
9B	Separator Bottoms	6/8			7.84		
	Can No. 1	6/9	24 hrs		14.95	276 (54.3%)	41.3
	Can No. 2	6/10	48 hrs		22.32	191 (38.5%)	42.6
	Can No. 3	6/11	72 hrs		20.36	168 (41.3%)	34.2
	Can No. 4	6/12	96 hrs		21.67	174 (35.4%)	37.7
	Can No. 5	6/15	168 hrs		8.94	No sample decanted	

VISCOSITY OF BOTTOMS CONTAINING 1-30 W % 975°F-

This study was made to examine the possible effect of startup oil on the vacuum bottoms pumping difficulties experienced in Pilot Plant Run 6. The viscosity of separator bottoms which had been vacuum distilled in order to yield 975°F distillate concentrations from 1-30 W % was measured as a function of temperature. Measurements were made for the representative Periods 5A, 9B, and 12A at three temperatures in order to establish the temperature dependency.

The temperature limit of the viscometer was nominally 500°F. However, this limit was relaxed for mixtures which did not flow at 500°F and the measurement range was extended to 550°F. The material behaves like a Bingham plastic body with, in most cases, a well-defined yield point. Figures 1 through 9 in Appendix IV demonstrate the linear relationship between shear stress and shear rate. The results of these measurements are shown in Table 5, where data on PDU Runs 3, 4, and 5 samples are cited for comparison.

For Period 12A (on recycle) a distillate content of ≥ 16 W % 975°F- was required in order to keep the viscosity at 500°F ≤ 3000 centipoise (at a shear rate of 34 Sec^{-1}). Comparable PDU 3, 4 and 5 samples containing 11, 15 and 12 W % distillate were 1.5 - 3 times less viscous than the 177-159 samples. For the earlier periods, 5A and 9B, where the decant oil/coal ratio was 1 and 0.2, respectively, the measured viscosities were substantially less than those reported for comparable mixtures in PDU 3-5.

The only viscosity data⁽¹⁾ on Pilot Plant vacuum bottoms was 105 centipoise at 34 sec^{-1} sheer rate at 400°F for 27 W % distillate. This value fits in well with data from Period 5A from the current bench run where μ was 162 cp in a mixture containing 25 W % distillate.

(1) Letter of February 23, 1982 from D. P. Malone, to N. Stewart, EPRI.

TABLE 5

VISCOSITY OF BOTTOMS CONTAINING 1-30 W % 975°F⁻ CONTENT VS. TEMPERATURE

- The viscosity of various concentrations (1-30 W %) of 975°F⁻ distillate in vacuum bottoms has been measured from Periods 5A, 9B and 12A.
- The material behave like a Bingham plastic body with, in most cases, a well-defined yield point (see graphs).
- Comparison is made with viscosity data from PDU 3, 4 and 5.

Period	Fraction	975°F ⁻ Content (W % Distillate)	Viscosity (centipoise) at 34 Sec ⁻¹ Shear Rate					
			350°F	400°F	425°F	450°F	475°F	500°F
5A	800°F ⁺	~25	490	162		88		
	880°F ⁺	18				615	282	244
	968°F ⁺	1				no flow	5,590	2,450
9B	800°F ⁺	27	2,100	443		195		[193]
	880°F ⁺	13			4,470	1,720	788	
	967°F ⁺	1						no flow 5,750
12A	800°F ⁺	30	3,890	618		271		(200)
	880°F ⁺	16				no flow	5,090	3,050
	935°F ⁺	~ 6				no flow		11,500
PDU-3-4A	859°F ⁺	10.9						1,550 613
PDU-4-6B	857°F ⁺	14.6						1,025 500
<hr/>								
PDU-5-19A	745°F ⁺	12.5	0-10 Minutes in Viscometer at 500°F 1,640* (1290)					
			10-20 Minutes in Viscometer at 500°F 2,160* (1780)					
			60-75 Minutes in Viscometer at 556°F 710** (556)					
			75-90 Minutes in Viscometer at 562°F 560** (340)					
<hr/>								
Pilot Plant		27		105				

[] data measured after an elapsed 1-2 week interval

() data are coefficient of rigidity (i.e. plastic viscosities)

* Brookfield RVT-5 Viscometer data in one gallon can at 100 rpm

** Brookfield RVT-3 Viscometer data in one gallon can at 100 rpm

VISCOSITY OF PDU RUN 5 BOTTOMS CONTAINING ≤ 33 W %
975°F- DISTILLATE

Since the Pilot Plant operations on Illinois No. 6 coal were modelled on PDU Run 5, for comparison purposes, it was deemed appropriate to measure the viscosity of PDU 5 bottoms containing various amounts of distillate (≤ 30 W %) as a function of temperature. Hydroclone overflow samples from the representative Periods 11A, 17A, 22A, and 29A of PDU 5 were vacuum distilled to 800°F to yield 800°F+ fractions which contained 15.0, 20.6, 28.0, and 33.2 W % distillate, respectively. Plots of shear stress vs shear rate (shown in Figures 1 through 4, Appendix V) display the linear relationship and the positive intercept (yield value) associated with Bingham plastic flow. The viscosity in centipoise is shown in Table 6 at a shear rate of 34 Sec⁻¹ at 400, 450, and 500°F. A distillate concentration of 15.0 W % at 500°F had a viscosity of 3300 centipoise.

The viscosity of a vacuum bottoms sample from Period 19A (cited in the PDU Run 5 report) is shown for comparison. The vacuum bottoms sample had a higher solids content (38.7 vs. 20.5) and a lower resid content (46.6 vs. 64.5) than the 11A HOF samples. Separator bottoms having 16 W % 975°F distillate from Run 159, Period 12A, operating on recycle without the use of startup oil, had a viscosity at 500°F of 3050 cp (see Table 5) which is comparable with PDU 5, 11A HOF sample with 15 W % distillate having a viscosity of 3280 cp at 500°F.

TABLE 6

VISCOSITY OF PDU RUN 5 BOTTOMS CONTAINING ≤ 33 W % 975°F- DISTILLATE

SAMPLE	800-975°F (W%)	975°F+ Oil (W)	Solids (W%)	Viscosity (centipoise) at 34 Sec ⁻¹ Shear Rate		
				400°F	450°F	500°F
11A-HOF 800°F+	15.0	64.5	20.5	no flow	24,500	3,280
17A-HOF 800°F+	[20.6]		17.3	1250	400	150
22A-HOF 800°F+	28.0	51.8	20.2	1260	400	176
29A-HOF 800°F+	33.2	46.6	20.2	510	203	112
19A Vacuum Bottoms	12.5	48.8	38.7			

0-10 Minutes in Viscometer 1640* (1290)
10-20 Minutes in Viscometer 2160* (1780)

[] Based on Distillation Data in PDU 5 Report

* Brookfield RVT-5 Viscometer data in one gallon can at 100 rpm

() Data are coefficient of rigidity (i.e. plastic viscosities)

RAMSBOTTOM CARBON RESIDUE (RCR) MEASUREMENTS ON SEPARATOR BOTTOMS

The propensity toward carbon formation was investigated by a series of RCR measurements on separator bottoms from Periods 2, 4A, 9 and 11. The startup oil/coal feed ratio in these periods was 2, 1, 0.2 and 0 respectively. These data are shown in Table 7, where the RCR organic residues increase from 10.5 in Period 2 to 19.5 in Period 11. In H-Oil practice, it has been found that when the RCR of the 975°F⁺ product exceeds ≈40% (depending to some extent on the nature of the feed) operational difficulties are encountered as a consequence of coking.

The RCR values in excess of ash cited in Table 7 represent the combined effects related to the use of startup oil in coal processing. In Table 8 an effort has been made to separate the unconverted coal from the RCR values. Ash, unconverted coal and resid percentages are listed which had been measured in the daily analysis of separator bottoms during the course of the run. The amount of coal in the RCR sample was estimated as follows:

$$\text{Period 2:} \quad \text{coal in RCR sample} = 1.54 \quad \frac{3.08}{3.38} = 1.40$$

$$\text{RCR in excess of coal} = 10.50 - 1.40 = 9.10$$

$$\frac{\text{RCR in excess of coal}}{\text{resid}} = \frac{9.10}{5.92} = 1.54$$

The dimensionless criterion, $\frac{\text{RCR in excess of coal}}{\text{resid}}$, falls from 1.54 in period 2 where the decant oil/coal ratio = 2 to 0.68 in period 11, where the ratio is 0. This criterion shows that the coking tendency is high for feed conditions in which decant oil is used in high concentrations relative to coal. Consequently, it is recommended that startup conditions where the ratio of startup oil/coal of >1 be as brief as possible.

TABLE 7

RAMSBOTTOM CARBON RESIDUE (RCR) MEASUREMENTS
 RUN 177-159 SEPARATOR BOTTOMS

PERIOD	SAMPLE (g)	RCR RESIDUE (g)	ASH (g)	RCR RESIDUE (W %)	ORGANIC RCR RESIDUE (W %)
2	0.3761	0.0512	0.0116	13.6	10.5
4a	0.3890	0.0853	0.0233	21.9	15.9
9	0.9585	0.2725	0.0786	28.4	20.2
11	0.7172	0.2081	0.0682	29.0	19.5

PERIOD	W% ASH (RCR)	RCR IN EXCESS OF ASH	W% Ash (from	W% COAL UNCONVERTED daily analysis)	RESID W%	COAL IN RCR SAMPLE (estimated)	RCR IN EXCESS OF COAL resid
2	3.08	10.50	3.38	1.54	5.92	1.40	1.54
4A	5.99	15.90	5.89	2.72	15.64	2.77	0.84
9	8.20	20.20	7.41	3.51	25.80	3.88	0.63
11	9.51	19.50	9.06	3.75	22.70	3.94	0.68

MICROAUTOCLAVE TESTS ON SOLVENTS

Microautoclave testing is a quick and inexpensive means of ascertaining the ability of slurry oils to dissolve coal. Tests have been performed on startup oil and hydroclone overflow oil from Run 6 at Catlettsburg with Illinois No. 6 coal. The results of microautoclave tests are as follows:

Conditions: 750°F, 10 minutes
no H₂, 1 gm coal and 8 gms solvent

Illinois No. 6 Coal (3.02% H₂O, 10.54% ash)

<u>Solvent</u>	<u>% Conversion to THF Solubles</u>
Startup Oil (HRI #4341)	51.23, 51.76
Hydroclone Overflow	62.16, 61.94*
(HRI # 4314)	58.93, 62.29

$$\% \text{ Conversion} = \frac{\text{dry coal sample} - \text{insoluble}}{\text{m.a.f. coal}} \times 100$$

7.83 ash in original HOF sample
6.25% of the ash free material is THF insoluble

*in HOF calculation, insolubles = THF insoluble material (ash in solvent + Wt THF insolubles in ash-free solvent)

The performance of Pilot Plant startup oil is substantially inferior to hydrogenated anthracene oil, 51% vs. ~75%, based on the results of microautoclave tests.

Microautoclave data* on H-Coal Pilot Plant solvents from Catlettsburg Run No. 6 reveal that the original P-469 tank solvent had a solvent quality (based on Tetralin = 100%) of 74%. On our test basis, this yields a THF soluble conversion of $0.74 \times \sim 75\% = 55\%$. This value agrees with a value for composite slurry oil consisting of startup oil (HRI 4341) of 51% and hydroclone overflow (HRI 4314) of 61%.

*Technical Advisory Committee Minutes of March 25, 1981,
TAC Meeting, Exhibit 3 by D. Malone.

CATALYST

For Run 177-158 169 cc of American Cyanamid HDS-1442A fresh catalyst (HRI 3830) and 281 cc of recovered HDS-1442A from Catlettsburg (HRI 4340), which constituted 38 V % of new catalyst, were charged to the reactor. The recovered catalyst from Catlettsburg contained 13.3 W % carbon and had a catalyst age of 1317 lbs coal/lb catalyst after 1400 hours of service. At the end of Run 177-158 the catalyst was withdrawn and replaced by another catalyst charge comprising 156 cc HRI 3830 and 294 cc HRI 4340 for Run 177-159. An analysis of the recovered catalyst from Run 177-159 is shown in Table 8. The carbon content of the recovered catalyst was 19.3 W % after 275 hours and an additional catalyst age of 499 lbs coal/lb catalyst experienced in Run 177-159.

TABLE 8

ANALYSIS OF RECOVERED CATALYST FROM RUN 177-159
 "STANDARD" CATALYST OPERATIONS

Catalyst Age, Pounds Coal/Pound Catalyst	499
Hours of H-Coal Operation	275
Weight of Solids Recovered, grams	432.1
Removed by Toluene Washing, W %	
Sieve Analysis of Washed Solids, W %	
+20 Mesh Extrudate	70.1
+20 Mesh Lumps	1.9
20/30	1.5
30/100	14.2
100/325	11.6
325	0.7
+20 Mesh Extrudate	
W % Toluene Soluble	0.0
Bulk Density, grams/cc	0.962
Oil Free Extrudate Analysis	
Carbon, W %	19.32
Hydrogen, W %	1.33
Sulfur, W %	3.52
Molybdenum, W % (Fresh Catalyst 9.73% Mo)	6.51
Titanium, W %	2.17
Iron, W %	0.22
Sodium, W %	0.45
Calcium, W %	0.13
Pore Volume,	
D>30 Å, cc/gram extrudate	0.237
Pore Volume*,	
D>30 Å, cc/gram estimated fresh catalyst	
Weight Loss on Ignition (850°F, 2% O ₂), %	28.6
Analysis of Ignited Extrudates	
Carbon, W %	0.93
Hydrogen, W %	0.55
Sulfur, W %	0.681
Molybdenum, W %	9.03
Pore Volume,	
D>30 Å, cc/gram extrudate	
Pore Volume*,	
D>30 Å, cc/gram estimated fresh catalyst	

*Assumes no loss of molybdenum

APPENDIX I

DATA SUMMARY

Period	SU	1	1	2	3	4	5	6	7	8	9	10	11	12A
Hours of Run - Beginning	0	24	0	24	48	71	95	119	143	167	191	215	239	263
- End	12	36	24	48	71	95	119	143	167	191	215	239	263	275
Catalyst Age - Lbs Coal/lb Catalyst														
- Beginning	0		0	38	83	119.3	164.2	209.2	254.1	298.1	343.1	388.0	432.0	477.0
- End			38	83	119.3	164.2	209.2	254.1	298.1	343.1	388.0	432.0	477.0	499.4
OPERATING CONDITIONS														
Coal Feed, % Moisture		1.86	2.84			1.30			1.43			1.21		
% Ash (Dry Co l)		11.25	11.08			10.45			8.99			10.93		
Coal Feed Rate, Lbs/Hr/Ft ³ Reactor		2.08	26.4	31.2	26.1	31.2	31.2	31.2	30.6	31.2	31.2	30.6	31.2	31.2
Lbs/Hr/Ft ³ Catalyst		46.2	58.7	69.3	58.0	69.3	69.3	69.3	69.3	69.3	69.3	68.0	69.3	69.3
Hydrogen Rate, MSCF/Ton Coal,														
Reactor Outlet (Vapor Phase)		65.0	50.5	43.5	49.7	46.3	47.3	42.9	42.2	41.7	41.8	41.9	43.4	42.2
Reacted (Hydrogen Balance)		23.0		24.5		22.3	23.6	28.7		23.1	21.2	30.5	20.2	21.7
Slurry Oil, Recycled Filtered Oil,														
Lbs/Lb Coal		0	0	0	0	0	0	0	0	0	0	0.0	0.400	0.400
Composition														
IBP, °F													385	362
EP, °F													975	975
% @ 650°F													82.0	76.5
W% Residue @ EP													26.2	28.0
Hot Separator Reactor														
Slurry Recycle, Lbs/lb Coal		0	0	0	0	0	0	0	0	0	0	0	1.400	1.400
W% Distillate to 975°F		88.5	91.5	89.2	76.1	75.8	77.4	73.7	70.0	63.0	63.3	69.3	64.5	60.6
Non-Distillate Oil, 975°F+														
Benzene-Soluble		8.1	2.8	5.9	15.2	15.6	14.6	17.7	19.6	25.4	25.8	19.7	22.7	25.9
Benzene-Insoluble														
Unreacted Ash-Free Coal		3.4	1.9	1.5	2.8	2.7	2.6	3.0	3.3	3.6	3.5	3.6	3.8	3.8
Ash			3.9	3.4	6.0	5.9	5.5	6.2	7.1	8.0	7.4	7.5	9.1	9.7
Vacuum Distillate Recycle, Lbs/Lb Coal		0	0	0	0	0	0	0	0	0	0	0	0.235	0.211
Vacuum Bottoms Recycle, Lbs/Lb Coal		0	0	0	0	0	0	0	0	0	0	0	0	0
Makeup Oil (HRI 4314), Lbs/Lb Coal		0	0	0	1.1856	1.0000	1.0000	1.0000	1.8007	1.8000	1.8000	1.8000	0	0
Makeup Oil (HRI 4341), Lbs/Lb Coal		3.2052	2.460	1.989	1.3987	1.0175	1.0173	1.0168	0.2258	0.2069	0.2123	0.2566	0	0
Temperature, °F														
Reactor			834	840	838	852	850	850	850	850	851	852	850	850
Reheater			781	781	782	782	782	780	780	780	780	780	780	780
Hot Separator, Reactor Outlet			650	643	645	658	652	650	648	654	649	644	650	649
Pressure, Reactor Outlet, psig			2492	2490	2500	2505	2500	2500	2498	2495	2498	2498	2495	2500
% Hydrogen in Vent Gas		97.0	94.8	95.0	94.1	95.2	95.1	93.7	92.9	93.6	93.6	92.8	98.2	90.2
Hydrogen Partial Pressure														
Hot Separator, psig		1943	1731	1844	1852	1875	1859	1835	1846	1828	1856	1856	1866	1804
Collected Products, W % of Feed Coal														
Light Liquid Hydrocarbons		21.42	15.92	12.87	14.56	17.87	15.17	15.15	17.14	14.20	14.13	15.00	15.48	15.27
Heavy Liquid														
(Vacuum Distillate)		0	0	0	0	0	0						14.28	12.75
Filtered Liquids		0	0	0	0	0	0						0.00	0.00
Separator Bottoms		343.88	273.64	249.82	309.19	253.76	268.92	253.73	254.11	251.90	263.11	247.42	24.42	26.18
Filter Cake		0	0	0	0	0	0						10.59	7.97
C ₁ -C ₃ Hydrocarbon in Gas Product		7.78	9.55	7.67	9.70	8.31	8.87	9.04	7.81	10.26	8.41	9.85	9.12	11.18
C ₄ -C ₆ Hydrocarbon in Gas Product		4.21	3.73	2.91	4.67	3.45	4.19	3.63	3.15	4.14	4.90	4.58	4.54	4.96
CO ₂ +CO		0.30	0.67	0.74	0.35	0.26	0.24	2.69	1.92	2.87	1.13	1.06	0.99	1.55
Collected Water Phase														
(with NH ₃ & H ₂ S)		17.17	28.23	15.60	10.38	13.83	14.33	13.43	12.91	12.18	12.18	11.09	11.24	11.15
H ₂ S in Gas Products		0.45	0.48	0.53	1.46	0.39	0.56	0.59	1.62	0.63	1.17	1.55	2.03	2.76
Total Collected Products		395.21	332.22	290.14	350.31	297.87	312.52	298.24	298.66	296.18	305.03	290.54	92.99	93.77
Normalized Collected Products		426.54		305.21		307.60	307.94	306.05		306.72	306.94	313.64	105.29	105.70
Coal Conversion, W % of M.A.F. Coal*			94.4	94.7	95.6	95.5	95.4	95.2	96.2	96.4	95.9	95.8	95.2	95.2
Forced Ash Balance*														
Normalized Product Distribution, W % of Dry Coal														
CO ₂	0.14			0.47		0.01	0.00	2.47		2.58	0.64		0.61	0.97
CO	0.19			0.31		0.26	0.24	0.31		0.42	0.50		0.54	0.82
C ₁ -C ₃ Hydrocarbons	8.55			8.24		8.63	8.76	9.33		10.71	8.51		10.42	12.87
C ₄ in Gas Products	1.12			1.03		1.56	1.53	1.32		1.71	2.21		1.88	2.77
C ₅ in Gas Products	0.99			0.72		0.79	1.02	0.82		0.89	0.91		1.00	0.99
C ₆ in Gas Products	2.52			1.40		1.76	1.63	1.57		1.72	1.83		2.33	1.94
IBP-400°F in Liquid	12.73			13.06		13.41	11.08	10.18		11.77	14.46		9.87	9.74
400-650°F Distillate	29.97			19.60		36.48	22.95	31.76		26.05	34.28		17.09	16.07
650-975°F Distillate	0.49			21.60		1.11	19.36	0.28		2.59	-7.56		14.83	12.32
975°F+ Residue Oil	18.86			8.42		12.12	9.65	15.90		20.10	21.48		17.93	18.64
Unreacted Ash Free Coal				4.80		4.10	4.22	4.35		3.31	3.81		4.28	4.14
Ash	12.85			10.54		10.54	10.54	10.54		10.54	10.54		10.54	10.54
Water (Net Production)	16.98			12.91		11.77	11.86	11.48		0.33	10.09		10.61	10.56
NH ₃ (ced Nitrogen)	xx			0.63		0.63	0.63	1.35		0.63	0.63		0.63	0.54
H ₂ S (ced Sulfur)	0.45			2.76		2.80	2.79	2.82		2.80	3.44		2.75	2.75
Total (100.0 + H ₂ Reacted)	106.11			106.51		105.92	106.27	104.44		106.13	105.76		105.35	105.77
C ₄ -400°F	17.36			16.21		17.51	15.25	13.35		16.09	19.43		15.08	15.45
C ₄ -975°F	47.82			57.42		55.09	57.56	45.39		44.73	46.14		47.00	43.84

*Solids (unreacted coal and ash) adjusted to give 10.54 W % ash recovery. Liquids in separator bottoms adjusted to maintain normalized product total.

APPENDIX II
OPERATING HISTORY

OPERATING HISTORY

On May 15, 1981, 169 cc of American Cyanamid HDS-1442A fresh catalyst (HRI 3830) and 281 cc of recovered HDS-1442A from Catlettsburg (HRI 4330) were charged to the reactor. The unit was pressure tested up to 2900 psia of hydrogen, leak tested and tightened up. On May 16th the bottom head was insulated and the lines were tape-wrapped. Hydrogen flow to the unit commenced on May 20th. The reactor and preheater temperatures were raised to 430°F. On May 21st with the reactor temperature at 800°F coal was cut in at 100 grams per hour starting Period 1.

On May 22, the temperature was raised to 815°F; the coal rate was raised to 200 grams/hr. Period 1A started at 0900 hours on May 22nd. The reactor temperature was increased to 835°F and the coal feed rate was raised to 333 grams/hr. In period 2A on May 23rd the reactor temperature was raised to 850°F and the coal feed rate to 500 grams/hr. The charge pump stopped functioning and started losing pressure. The recycle temperature started falling. At 1530 hours with a 2000 psia P across the unit, shutdown procedures were invoked. The unit was turned around on May 24th. A restriction in the top coupling of the preheater was freed. Several leaks in pumps and bleed lines were eliminated.

Fresh catalyst, 95 grams of HRI 3800 and 324.6 grams of HRI #4336 (withdrawn from the Catlettsburg reactor) were charged on May 26th. The unit was brought back up to temperature. On May 28th coal was cut in at 100 grams/hr thereby starting run 177-159 Period 1A. Throughout the period the coal rate was raised to 500 grams/hr and the reactor temperature was raised to 850°F. Some trouble was experienced with the charge pump. Changing the stroke appeared to be helpful. The charge pump stopped temporarily on May 29th.

On May 30th trouble was experienced with the main feedline valve and R/S and L/S charge pumps hot checks. Temperatures were cut back to 500°F. The charge pumps, buffer, #2 recycle and circulating pumps were shut down and the reactor by-pass valve was opened. The hydrogen pressure in the unit was lowered. After repairs by Maintenance, the reactor ΔP by-pass valve was closed thus bringing the pressure to 2500 pisa

hydrogen. Leak testing was completed satisfactorily. The temperature was brought back up to 800°F at 0900 hours on May 31st. Coal was cut in at 100 grams/hr with HRI 4314 feed at 500 grams/hr thus starting period 3A. Throughout the period the coal feed rate was raised to 500 grams/hr with the reactor temperature at 850°F and the HRI 4341 oil rate at 400 grams/hr. On June 1st the main block valve in the charge inlet line failed to hold. Packed coal was removed from around the stem which had prevented the valve from closing. Period 5A began at 0900 hours on June 2nd. In Period 6A the hydrogenflow was lowered twice.

In Period 7 #4341 oil was cut out to the charge pot and HRI 4004 and 4314 were charged. The charge pot started to plug. By increasing the stroke of the pump and in bringing steam to the circulating line, the plug was broken. This procedure was followed again in order to break successfully another plug. On June 5th at 0330 hours the reactor and overall ΔP went up and the recycle discharge temperature fell to 711°F. The line from the bottom of the R/S charge pot to the #1 circulating line was plugged with coal. The line was cleared at 0800 hours and switched to #2 pump. At 1830 hours the L/S charge pump pressure fell (the unit pressure was at 2500 psia and the charge pump pressure was 1850 psia). The gauge was checked and appeared to be performing normally. Hydrogen was diverted from the 177 unit on June 6th, while the 130 unit was being pressurized. The overall ΔP went up 50 psia and the recycle temperature fell. The reactor ΔP increased to 50 psi and then fell to 11 psia. Hydrogen was shut off to the unit and the unit stabilized as the overall ΔP fell to 10.5 psia and the recycle temperature lined out at 695°F. The L/S hot let-down valve was removed, cleaned and reinstalled.

On June 7th at 1930 hours the L/S charge pump stopped. The buffer pump rate was increased. The charge was transferred from the L/S to the R/S pot. The R/S charge pump was cut in and the buffer pump was set back to normal. The L/S charge pump was removed, cleaned and reinstalled. On June 8th the R/S charge pump pressure gauge read 1250 psia. The gauge was blocked in, the line was blown clear and reassembled. At 0130 hours the R/S charge pot was drained of slurry which was transferred into the L/S charge pot. The contents of the L/S charge pot could not be circulated. The contents were switched back to the R/S. Steam was turned on the L/S charge pot and the #3 circulating lines. At 0300 hours 2000 grams of separator bottoms were sent to the pressure filter. Two thousand grams of separator bottoms were sent to the vacuum still. Since the L/S charge pot circulating pump stopped and

the pot contents would not drain, the pump suction line was removed. A plug of thick slurry was rodded out of the charge pot. Five hundred grams of HRI 4341 were added to the slurry in the L/S pot along with 500 grams of HRI 4341 oil. The diluted slurry was circulated through the L/S pot. At 0325 hours the R/S charge pump stopped. The L/S charge pump with diluted slurry was cut in at 0340 hours. The R/S pot contents were added to the L/S pot. The R/S charge pump was depressurized. A plug was found from the checks to the gauge block valve.

At 1305 hours June 8th, the plugs were cleared from the suction side No. 1 circulating pump. The circulating line to the charge pot upper section was replaced. The discharge line (up to the pressure gauge) was also plugged. The R/S charge pump was repacked. The line was flushed and pumped clear. The R/S charge pot was drained and refilled with HRI 4341 oil. At 1300 hours, the R/S charge pump fed approximately 200 grams of 4341 oil into the unit in order to clean the R/S inlet line. Since the No. 1 recycle pump was losing suction, the No. 2 recycle pump was switched on-stream.

On June 9th, at 0800 hours, it was found that the windings for No. 1 pressure filter were broken off. Some delay was incurred in setting up the No. 2 filter. At 1450 No. 1 filter windings were replaced. At 2245 hours the reactor and overall ΔP were 16.5 psia. At 2248 hours the reactor and overall ΔP had increased to 50⁺ psia. At 2300 hours the charge oil on L/S was cut out and the No. 4341 oil in R/S pot was cut in. At 2308 hours the inlet pressure was 3000 pisa and the outlet pressure was 2500 pisa. The charge pump cutout and hydrogen and the vent flows were zero. At 2325 hours the backpressure B/P was dropped to 2000 psia (inlet pressure was still at 3000 psia). At 2330 hours with the backpressure at 1500 psia, the plug broke free. The unit and reactor ΔP were at 50⁺ psia.

At 0015 hours on June 10th the inlet pressure was 3000 psia and the backpressure was 2000 psia. The charge pump stopped-was restarted-stopped again. The recycle and buffer pumps stopped. The backpressure was lowered to 1500 psia, however, the restrictions did not break. Sucking back on the charge pump did not help. At 0030 hours the back pressure was lowered to 2000 psia, however, the restriction held. The hydrogen flow was cut off; the overall and reactor ΔP by-pass

valve was opened in order to equalize the pressure; heat was cut to 300°F; and the unit was depressurized. The L/S charge pot circulated slurry mix while the R/S charge pot was circulating HRI No. 4341 oil. Since the hot separator level was at the 100% level an unsuccessful attempt was made to drain through the by-pass valve. The buffer and recycle pump pressures were still 3000 psia. Depressurizing at the pump drains dropped these pressures to zero. Insulation was taken off the top and bottom heads and air was passed over the reactor in order to help cool the unit. Purging of the unit with nitrogen started. The bottom of the separator could still not be drained through the by-pass valve. The unit was depressurized of nitrogen. At 0900 hours a restriction was found in the recycle tube of the top head. A hard restriction in the reactor top was located 21 inches down. The bed had slumped approximately three feet in the reactor.

APPENDIX III

ANALYSES OF FEED MATERIALS

TYPICAL COAL ANALYSES AND PROPERTIES

Illinois No. 6, Burning Star

Run No.	177-158, 159
HRI No.	4004
Moisture, W %	2.25
Proximate, Dry Basis, W %	
Ash	10.78
Volatile Matter	35.54
Fixed Carbon	53.68
Ultimate, Dry Basis, W %	
Carbon	70.18
Hydrogen	4.64
Nitrogen	1.00
Sulfur	3.28
Ash	10.78
Oxygen (Difference)	10.12
Sulfur Forms Dry Basis, W %	
Pyritic	1.08
Sulfate	0.04
Sieve Analysis, Screen Size (USS), W %	
+50	0.00
50/70	0.05
70/100	5.45
100/140	17.01
140/200	15.45
200/325	13.26
-325	48.78

TABLE III-2

STARTUP OIL ANALYSIS

Description	Hydroclone Overflow from Pilot Plant Startup HRI No. 4314	Pilot Plant Startup Oil HRI No. 4341	Clarified* Slurry Oil 3/29
Gravity, °API	-11.0	14.0	5.1
Vacuum Distillation (ASTM D-1160 Modified)			
IBP, °F	533	429	407
10 V %, °F	610	497	589
20 V %, °F	654	527	--
30 V %, °F	695	554	692
40 V %, °F	730	581	--
50 V %, °F	779	606	766
60 V %, °F	858	644	--
69 V %, °F	923	--	--
70 V %, °F	--	684	843
80 V %, °F	--	730	--
90 V %, °F	--	810	955
98 V %, °F	--	959	--
V % at 650°F	19	63	--
Carbon, W %	82.38	88.46	89.70
Hydrogen, W %	7.10	9.91	8.4
Sulfur, W %	0.97	0.32	1.06
Nitrogen, W %	0.33	0.23	0.33
Toluene Insolubles, W %	24.64	--	
Quinoline Insolubles, W %	12.15	--	
Ash, W %	7.80	--	

*For comparison purposes. Data from Pilot Plant Report Coal Run No. 6,
May 20, 1981, p. 19.

APPENDIX IV
BINGHAM PLASTIC FLOW CHARACTERISTICS
OF
SEPARATOR BOTTOMS CONTAINING 1-30 W % 975°F DISTILLATE

RUN 177-159-5A SEPARATOR BOTTOMS
800°F FRACTION

~25% 975°F~

FIGURE IV-2

RUN 177-159-5A SEPARATOR BOTTOMS 968°F⁺ FRACTION

1 W % 975°F⁻

RUN 177-159-9B SEPARATOR BOTTOMS 800°F⁺ FRACTION

27 W % 975°F⁻

RUN 177-159-9B SEPARATOR BOTTOMS 880°F⁺ FRACTION

13 W % 975°F⁻

RUN 177-159-9B SEPARATOR BOTTOMS 967°F⁺ FRACTION

1 W % 975°F⁻

FIGURE IV-7

RUN 177-159-12A SEPARATOR BOTTOMS 800°F⁺ FRACTION

30 W % 975°F⁻

RUN 177-159-12A SEPARATOR BOTTOMS 880°F⁺ FRACTION

14 W % 964°F⁻

RUN 177-159-12A SEPARATOR BOTTOMS 935°F⁺ FRACTION

~6 W % 975°F⁻

APPENDIX V

BINGHAM PLASTIC FLOW CHARACTERISTICS

OF

PDU RUN 5 HOF SAMPLES CONTAINING <33 W % 975°F- DISTILLATE

FIGURE V-1

PDU HOF-11A 800°F⁺ FRACTION

15.9 W % 975°F⁻

FIGURE V-2

PDU HOF-17A 800°F⁺ FRACTION

FIGURE V-3

PDU HOF-22A 800°F⁺ FRACTION

28.0 W % 975°F⁻

PDU 5 HOF-29A 800°F⁺ FRACTION

33.2 W % 975°F⁻

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