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COAL CLEANING EFFECTS DURING H-COAL CATALYTIC
LIQUEFACTION OF A WESTERN KENTUCKY COAL

Final Report

By
Richard F. Bernard

December 1978

Work Performed Under Contract No. EW-78-C-22-0265

Hydrocarbon Research, Inc.
Lawrenceville, New Jersey

U. S. DEPARTMENT OF ENERGY

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H-COAL® CATALYTIC LIQUEFACTION
OF A WESTERN KENTUCKY COAL

FINAL REPORT

RICHARD F. BERNARD

DECEMBER 1978

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TABLE OF CONTENTS

	<u>PAGE</u>
ABSTRACT	1
CONCLUSIONS	3
RECOMMENDATIONS	5
OBJECTIVES AND SCOPE OF WORK	7
RESULTS	9
DETAILED DESCRIPTION OF TECHNICAL PROGRAM	40
Physical and Mechanical Details	41
Program Details	46
APPENDICES	
A - Detailed Daily Operating Data	51
B - Operating History	58
C - PETC Sampling Program	65
D - Calculation of Unconverted Coal and Benzene Insoluble Residuum Quantities from Analytical Data	69
E - Calculation of Normalized Product Distribution	71

LIST OF TABLES

<u>TABLE NO.</u>		<u>PAGE</u>
1	Summary of Operating Results	13
2	Production Composition and Viscosity	14
3	Analyses of Recovered Catalysts	31
4	Commercial Plant for Liquefaction of Kentucky No. 11 Coal	38
5	Vacuum Still Bottoms Composition	39
6	Startup Oil Analysis	43
7	Feed Coal Analysis	44
8	Process Conditions	46
9	Typical Catalyst Analysis	47

LIST OF FIGURES

<u>FIGURE NO.</u>		<u>PAGE</u>
1	Coal Conversion	15
2	C ₁ -C ₃ Hydrocarbon Yields-Dry Coal Basis	16
3	C ₁ -C ₃ Hydrocarbon Yields-Converted Coal Basis	17
4	Total C ₄ -400°F Distillate Yields-Dry Coal Basis	18
5	Total C ₄ -400°F Distillate Yields Converted Coal Basis	19
6	400-650°F Distillate Yields-Dry Coal Basis	20
7	400-650°F Distillate Yields-Converted Coal Basis	21
8	650-975°F Distillate Yields-Dry Coal Basis	22
9	650-975°F Distillate Yields-Converted Coal Basis	23
10	C ₄ -975°F Distillate Yields-Dry Coal Basis	24
11	C ₄ -975°F Distillate Yields-Converted Coal Basis	25
12	975°F+ Residual Oil Yields-Dry Coal Basis	26
13	975°F+ Residual Oil Yields-Converted Coal Basis	27
14	Hydrogen Consumption-Dry Coal Basis	28
15	Hydrogen Consumption-Converted Coal Basis	29
16	Pore Size Distribution - Run 177-143 Discharged Catalyst	32
17	Pore Size Distribution - Run 177-143 Regenerated Catalyst	33
18	Pore Size Distribution - Run 177-144 Discharged Catalyst	34
19	Pore Size Distribution - Run 177-144 Regenerated Catalyst	35
20	Calculated Viscosity of Vacuum Bottoms Slurry in Kentucky No. 11 Coal Operation for Various Distillate/Resid Ratios	37
21	Bench Unit Flowsheet	45
22	Pore Size Distribution of H-Coal Catalyst	48

ABSTRACT

Two H-Coal[®] bench-scale liquefaction tests were performed to compare the hydroliquefaction behavior of two Kentucky No.11 coals from the same mine: a run-of-mine coal with 17.49 W % ash and a deep-cleaned coal with 6.21 W % ash. The tests were conducted using a syncrude mode of operation.

The deep-cleaned coal exhibited greater coal conversion and greater residual oil yield than the run-of-mine coal. On a dry coal basis, the deep-cleaned coal yielded approximately 19% more C₄ to 975°F distillate than the run-of-mine coal. The process requirement of a pumpable vacuum still bottoms product would result in a 10% higher C₄ to 975°F yield from the deep-cleaned coal than from the run-of-mine coal in a commercial H-Coal plant.

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CONCLUSIONS

- * The deep-cleaned Kentucky No.11 coal exhibited greater coal conversion than the run-of-mine coal. Deep cleaning may remove a fraction of the unreactive m.a.f. coal present in the run-of-mine coal.
- * The deep-cleaned coal yielded more residual oil than the run-of-mine coal. Also, the fraction of residual oil which was toluene-soluble was less for the deep-cleaned coal than for the run-of-mine coal. The high ash level of the run-of-mine coal may increase the conversion of residual oil.
- * There was no marked difference between the two coals in the rates of catalyst deactivation for conversion of toluene-soluble and toluene-insoluble residuum.
- * The total C₄ to 975°F distillate yield on a converted coal basis was about the same for both coals; on a dry coal basis, the deep-cleaned coal yielded approximately 19% more C₄ to 975°F distillate than the run-of-mine coal.
- * In a 25,000 ton per day H-Coal plant operating in the syncrude mode at a catalyst age of 300 lbs coal/lb catalyst, the deep-cleaned coal would yield approximately 10% more C₄ to 975°F product than the run-of-mine coal.
- * On a converted coal basis, the yields of H₂S, NH₃, and H₂O were higher for the run-of-mine coal than for the deep-cleaned coal.
- * The hydrogen consumption on a converted coal basis was higher for the run-of-mine coal than for the deep-cleaned coal.

RECOMMENDATIONS

- * A bench-scale run with the deep-cleaned Kentucky No. 11 coal and increased residual oil recycle should be performed. The increased residual oil recycle should further increase the distillate oil yield resulting in a major yield improvement.
- * Petrographic analysis of the deep-cleaned and run-of-mine coals should be made. The analysis may help explain the differences in yields from the two coals and indicate direction for further process improvements.
- * A detailed technical and economic process evaluation should be performed for the deep-cleaned coal case including fuel and energy balances, net plant product and properties, integration with the coal washing process, and kinetic analysis of the yield data.

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OBJECTIVES AND SCOPE OF WORK

The objective of the work performed under DOE/PETC Contract No. EW-78-C-22-0265 for "Conduct of H-Coal Catalytic Liquefaction Tests" was to determine the hydroliquefaction behavior in the H-Coal Process of a coal with a normal, run-of-mine ash content and a coal from the same mine which had been deep-cleaned to a low ash content. The two Kentucky No. 11 feed coals for evaluation were provided by PETC. The work was divided into five tasks: coal handling and analysis, comparative evaluation and review of run data, and special sample handling.

Coal handling and analysis was to include preparation of the coal to meet bench unit feed specifications and the elemental and mineral analyses of the feed coal for characterization and development of material balance comparisons. Two H-Coal liquefaction tests, one for each of the feed coals, were to be performed in a one-pound-per-hour coal feed ebullated bed reactor unit for an operating period of approximately eight to ten days each. Process conditions were to be typical of the syncrude mode of operation of the H-Coal Process. Similar operating conditions and procedures were to be used in each of the runs.

Process and product analysis was to include weighting and metering all feed and product streams. Distillate samples were to be analyzed for specific gravity, sulfur, distillation and viscosity. Slurry and solid products were to be routinely analyzed for sulfur, ash, solvent extraction and distillation. Gas analyses were to be performed on all gas streams and elemental analyses performed on all product streams for material balance.

Comparative evaluation and review of run data was to include reduction of all process and analytical data from the runs, and comparative evaluation of the data from both runs to assess the effect of ash content of the feed coal on process performance.

Special sample handling was to provide for collection of feed and product stream samples from both runs in specially-cleaned sample containers to minimize sample contamination. These samples were to be delivered to PETC for trace element analysis.

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OPERATING RESULTS

PRODUCT DISTRIBUTION

Deep-cleaned Kentucky No.11 coal with 6.21 W % ash was evaluated in Run 177-143. Run-of-mine (ROM) Kentucky No.11 coal with 17.49 W % ash was tested in Run 177-144. Both runs used the syncrude mode of operation with nominal process conditions of 31.2 pounds coal per hour per cubic foot space velocity, 850°F reactor temperature and 2250 psig unit pressure. A summary of the operating results for both runs is given in Table 1. This table includes the important process conditions, normalized yields and product properties. The results are averages of Periods 2 through 8A for Run 177-143 and of Periods 3 through 8 for Run 177-144. Figures 1 through 15 present the daily coal conversion, reacted hydrogen, and normalized yields as a function of catalyst age for both runs. Here the yields are given on both a dry coal basis and a converted coal basis. Detailed daily operating data may be found in Appendix A.

Coal conversion, evaluated by filtration of the separator bottoms charged to the vacuum still, averaged 92.19% for the R.O.M. coal and 95.90% for the deep-cleaned coal. As seen in Figure 1, the coal conversions were fairly constant throughout the run, with standard deviations of 0.22% for the R.O.M. coal and 0.18% for the deep-cleaned coal.

Operating experience with other coals has shown that a portion of the M.A.F. coal is not reactive even at high severity processing levels. The amount of non-reactive coal may be associated with the ash level of the coal. Consequently, with the higher-ash R.O.M. coal, a larger amount of non-reactive coal may be present. With equivalent conversion of the reactive fraction of the coal, the higher-ash coal would give lower M.A.F. coal conversions. Alternatively, with equivalent amounts of non-reactive M.A.F. coal, the lower conversion of the higher-ash coal could result from a lower conversion of the reactive portion of the M.A.F. coal. Since this study was conducted at a constant space velocity of 31.2 lbs/hr/ft³ it is not possible to determine the amount of M.A.F. non-reactive coal in the two coals; liquefaction tests at a range of severity levels would be necessary for this determination.

Previous H-Coal bench unit operations show that a difference in yields of over 2 W % probably indicates a significant change in performance.

The C₁-C₃ gas yield averaged 10.23 W % of dry coal for the higher-ash coal versus 12.01 W % of dry coal for the deep-cleaned coal. However, on a converted coal basis, the higher-ash coal yielded

13.45 W % C_1 - C_3 gases while the deep-cleaned coal yielded 13.36%. The difference between the yields on a converted coal basis is not statistically significant. The slopes of the trend lines in Figures 2 and 3 are not statistically significant.

The C_4 -400°F naphtha yield averaged 22.89 W % of dry coal for the higher-ash coal and 26.95 W % of dry coal for the deep-cleaned coal. On a converted coal basis, the yield for the higher-ash coal was 30.09 W % versus 29.96 W % for the deep-cleaned coal. Again, the difference between the yields on a converted coal basis is not statistically significant.

The trend lines for the C_4 -400°F naphtha yield for the two coals on a dry coal and a converted coal basis are shown in Figures 4 and 5. The slopes of the trend lines for Run 177-144 (R.O.M. coal feed) in both Figures 4 and 5 are not statistically significant. The C_5 yield in Period 8 of Run 177-144 (catalyst age 327.1 pounds coal/pound catalyst) deviated considerably from earlier periods and may account for the insignificant trend line. For the deep-cleaned coal (Run 177-143), the C_4 -400°F naphtha yield decreased with increasing catalyst age.

The 400-650°F distillate yield on dry coal was 17.39 W % for the higher-ash coal and 18.67 W % for the deep-cleaned coal. On converted coal the yield was 22.86 W % for the higher-ash coal and 20.76 W % for the deep-cleaned coal. For both bases, the differences are not statistically significant. As shown in Figures 6 and 7, the yield decreased with increasing catalyst age for the deep-cleaned coal; the slopes of the trend lines for the higher-ash coal are not significant.

On a dry coal basis, the 650-975°F distillate yield was 3.35 W % for the higher-ash coal and 6.37 W % for the deep-cleaned coal. On a converted coal basis, the 650-975°F distillate yield was lower for the higher-ash coal, 4.40 W %, versus 7.08 W % for the deep-cleaned coal. For both coals the 650-975°F distillate yield increased with increasing catalyst age.

The total C_4 -975°F distillate yield was lower for the higher-ash coal, 3.71 barrels per ton M.A.F. coal, than for the deep-cleaned coal, 3.88 barrels per ton M.A.F. coal. The total C_4 -975°F distillate yield was 4.02 barrels per ton converted coal for the higher-ash coal and 4.05 barrels per ton converted coal for the deep-cleaned coal. These differences are not considered significant. Figures 10 and 11 show a declining total C_4 -975°F distillate yield with increasing catalyst age. The trend lines for the higher-ash coal (Run 177-144) are not significant, again possibly due to the atypical C_5 yield in Period 8.

A review of analytical data provides no basis for rejecting the validity of the C_5 yield in Period 8 of Run 177-144. However, eliminating the C_4 to 975°F distillate yield data point in the analysis of data results in averages of 3.03 and 3.99 barrels of C_4 to 975°F distillate per ton of dry coal and converted coal, respectively. The trend lines for the C_4 to 975°F distillate yields on this basis are also shown in Figures 10 and 11.

The total 975°F+ residual oil yield was lower for the higher-ash coal, 11.10 W % dry coal, than for the deep-cleaned coal, 17.85 W % dry coal. The same difference was noted on a converted coal basis, 14.59 W % for the higher-ash coal versus 19.84 W % for the deep-cleaned coal. For both coals the yield of 975°F+ residual oil increased with increasing catalyst age.

A higher fraction of the 975°F+ residual oil was toluene-soluble for the higher-ash coal than for the deep-cleaned coal, 84.95 W % versus 77.76 W %. There was 0.45 W % sulfur in the 975°F+ residual oil from the higher-ash coal and 0.50 W % sulfur from the deep-cleaned coal. The difference in the sulfur contents of the two products is not statistically significant. Also, there was no significant trend in the sulfur content of the 975°F+ residual oil. The higher-ash, R.O.M. coal appeared to increase the residual oil conversion and the toluene-soluble fraction of the residual oil compared to the deep-cleaned coal.

The higher-ash coal gave higher yields of net water, ammonia and hydrogen sulfide on a converted coal basis. However, the H_2S yields for both coals corresponded to greater than stoichiometric conversion of the organic sulfur, the H_2O yields on a converted coal basis for the R.O.M. coal and the deep-cleaned coal correspond to 133% and 94%, respectively, stoichiometric conversions of oxygen by ultimate analysis. This again suggests reduction of oxides present in the ash.

Hydrogen consumption (as MSCF per ton feed coal) was higher for the deep-cleaned coal, 23.21 versus 21.56 for the higher-ash coal. However, on a converted coal basis, the higher-ash coal consumed more hydrogen, 29.33 MSCF per ton converted coal, than the deep-cleaned coal, 26.71 MSCF per ton converted coal. As shown in Figures 14 and 15 the hydrogen consumption for the deep-cleaned coal declined with increasing catalyst age while the trend was not clear with the high-ash coal.

The viscosities of the total separator overhead, vacuum distillate, and pressure filter liquid products were measured during the course of the run and are presented in Table 2. Viscosities were measured with a Saybolt Universal Viscometer at an appropriate temperature for the sample. The total separator overhead product averaged 30 SUS at 100°F for both runs. For Runs 177-143 and 177-144, the vacuum overhead product viscosities averaged 46 and 39 SUS at 130°F, respectively. This is consistent with the 650 to 975°F yield being lower in run 177-144 (run-of-the-mine coal) than Run 177-143 (deep-cleaned coal) as practically all of this fraction is found in the vacuum overhead.

The pressure filter liquid viscosity averaged 67 SUS at 210°F for Run 177-143 versus 43 SUS at 210°F for Run 177-144. Also, the 975°F+ residual oil in the pressure filter liquid averaged 28.3 W % in Run 177-143 and 20.9 W % in Run 177-144. These data are in better agreement with the lower residual oil yield of Run 177-144 (run-of-mine coal) than they are with Run 177-143 (deep-cleaned coal). Although there is considerable scatter in the data, there is a trend of increasing pressure filter liquid viscosities with increasing catalyst age. Viscosity data for these product streams may be found in Appendix A, Detailed Daily Operating Data.

TABLE 1

SUMMARY OF OPERATING RESULTS

Run Number	177-143	177-144
Kentucky No. 11 Coal	Deep-Cleaned	Run-of-Mine
HRI Number	4018	4017
Ash in Feed Coal, W % (Dry Coal)	6.21	17.49
Catalyst Age, Lbs Coal/Lb Catalyst	31.8-317.8	55.1-327.1
Coal Feed Rate, Lbs/Hr/Ft ³ Reactor	30.7	31.2
Reactor Temperature, °F	850	850
Pressure, psig	2243	2252
Recycle, Lbs/Lb Coal		
Vacuum Distillate	0.30	0.29
Filtered Liquid	0.69	0.69
Separator Bottoms	4.09	4.13
Coal Conversion, W % M.A.F. Coal	95.90	92.19

Normalized Yields	W % Dry Coal	W % MAF Coal	W % Converted Coal	W % Dry Coal	W % MAF Coal	W % Converted Coal
C ₁ -C ₃ Gases	12.01	12.81	13.36	10.23	12.40	13.45
C ₄ -400°F Naphtha	26.95	28.73	29.96	22.89	27.74	30.09
400-600°F Distillate	18.67	19.91	20.76	17.39	21.08	22.86
650-975°F Distillate	6.37	6.79	7.08	3.35	4.06	4.40
975°F ⁺ Distillate						
Total	17.85	19.03	19.84	11.10	13.45	14.59
Toluene-Soluble	13.88	14.80	15.43	9.43	11.43	12.40
Toluene-Insoluble	3.97	4.23	4.41	1.67	2.02	2.20
Unconverted Coal	3.95	4.21		6.67	8.08	
Ash	6.35			18.11		
H ₂ O (Net)	9.64	10.28	10.72	9.70	11.76	12.75
CO + CO ₂	0.63	0.67	0.70	0.57	0.69	0.75
H ₂ S	2.72	2.90	3.02	3.97	4.81	5.22
NH ₃	1.23	1.31	1.37	1.95	2.36	2.56
Total (100 + H ₂ Reacted	106.39			105.94		
C ₄ -975°F Distillate						
Barrels/Ton Dry Coal		3.64			3.06	
Barrels/Ton M.A.F. Coal		3.88			3.71	
Barrels/Ton Converted Coal		4.05			4.02	
Sulfur in 975°F ⁺ Oil, W %		0.50			0.45	
Toluene-Soluble in 975°F ⁺ , W %		77.76			84.95	
Hydrogen Consumption,						
MSCF/Ton Feed Coal		23.21			21.56	
MSCF/Ton MAF Coal		24.75			26.17	
MSCF/Ton Converted Coal		26.71			29.33	

TABLE 2

PRODUCT COMPOSITION AND VISCOSITY

	<u>Deep-Cleaned Coal</u> <u>Run 177-143</u>	<u>Run-of-Mine-Coal</u> <u>Run 177-144</u>
Separator Overhead		
SUS at 100°F	30	30
Vacuum Still Overhead		
650°F ⁺ , W %	39.7	26.3
SUS at 130°F	46	39
Pressure Filter Liquid		
650-975°F, W %	31.9	32.4
975°F ⁺ , W %	28.3	20.9
SUS at 210°F	67	43

COAL CONVERSION

FIGURE 2

C₁-C₃ HYDROCARBON YIELDS - DRY COAL BASIS

FIGURE 3

C₁-C₃ HYDROCARBON YIELDS - CONVERTED COAL BASIS

FIGURE 4

TOTAL C₄-400°F DISTILLATE YIELDS - DRY COAL BASIS

FIGURE 5

TOTAL C₄-400°F DISTILLATE YIELD - CONVERTED COAL BASIS

400-650°F DISTILLATE YIELD - DRY COAL BASIS

FIGURE 7

400-650°F DISTILLATE YIELD - CONVERTED COAL BASIS

FIGURE 8

650-975°F DISTILLATE YIELD - DRY COAL BASIS

FIGURE 9

650-975°F DISTILLATE YIELD - CONVERTED COAL BASIS

FIGURE 10

C₄ TO 975°F DISTILLATE - DRY COAL BASIS

FIGURE 11

C₄-975°F DISTILLATE YIELD - CONVERTED COAL BASIS

FIGURE 12

975°F+ RESIDUAL OIL YIELD - DRY COAL BASIS

FIGURE 13

975⁰F+ RESIDUAL OIL YIELD - CONVERTED COAL BASIS

FIGURE 14

HYDROGEN CONSUMPTION - DRY COAL BASIS

FIGURE 15

HYDROGEN CONSUMPTION - CONVERTED COAL BASIS

RECOVERED CATALYST

Analyses of the catalyst recovered from Runs 177-143 and 177-144 are reported in Table 3. The carbon content of the catalyst from the deep-cleaned coal was higher than that from the run-of-mine coal run, 14.97 W % and 12.91 W %, respectively. These values are in the range observed in H-Coal operations with this catalyst. The catalyst from the deep-cleaned coal run also exhibited higher metals levels than the catalyst from the run-of-mine coal run. Titanium content was 1.61 W % for the deep-cleaned coal and 0.922 W % for the run-of-mine coal. Iron content was 1.87 W % for the deep-cleaned coal and 0.878 W % for the run-of-mine coal. Again, the titanium and iron contents of both catalysts are typical of the levels found on this catalyst in H-Coal operations. Typical fresh catalyst metals levels are 0.3 W % iron and 0.0 W % titanium.

The pore size distributions of the discharged catalyst and regenerated catalyst for both runs are shown in Figures 16,17,18 and 19. The discharged catalyst and regenerated catalyst from the two runs exhibited similar alterations of the fresh catalyst pore size distribution. In both discharged catalysts, approximately 40% of the fresh catalyst pore volume was lost. About 70% of the reduction of pore volume occurred in pores with diameters in the range of 39 to 100 Å.

After regeneration at 850°F and 2% O₂, pore volume of pores less than 64 Å diameter decreased further to approximately one-third of that of the fresh catalyst. Pores with diameters less than 39 Å virtually disappeared. After regeneration, pore volume in pores with diameters of 64 to 100 Å was about three times that of the fresh catalyst. Approximately 92% of the fresh catalyst pore volume was regenerated in the discharged catalyst from Run 177-143; virtually all the pore volume was regained in the catalyst from Run 177-144 after regeneration.

The changes in pore volume and pore size distribution for the discharged and regenerated catalysts from both runs were again similar to that experienced in other H-Coal operations which used this catalyst.

TABLE 3

ANALYSES OF RECOVERED CATALYSTS

Run Number	177-143	177-144
Kentucky No.11 Coal Feed	Deep-Cleaned	Run-Of-Mine
HRI Number	4018	4017
Catalyst Age, Lbs Coal/Lb Catalyst	337.8	327.1
Hours of H-Coal Operation	192	180
Weight of Solids Recovered, Grams**	1212	475
Removed by Benzene-Washing, W %	64.1	24.7
Sieve Analysis of Washed Solids, W %		
+20 Mesh Extrudate	79.0	71.0
+20 Mesh Lumps	2.1	4.6
20/30	1.6	1.3
30/100	4.8	5.7
100/325	11.3	16.6
325/	1.5	0.6
+20 Mesh Extrudate		
W % Benzene-Soluble	0.70	0.75
Oil-Free Extrudate Analysis		
Carbon, W %	14.97	12.91
Hydrogen, W %	1.00	1.05
Sulfur, W %	4.77	4.39
Molybdenum, W % (Fresh Catalyst, 9.73% Mo)	6.72	7.19
Titanium, W %	1.61	0.922
Iron, W %	1.87	0.878
Sodium, W %	0.085	0.0296
Calcium, W %	0.0903	0.0888
Pore Volume, D >30 Å, cc/gram Extrudate ⁰	0.290	0.322
Pore Volume*, D >30 Å, cc/gram Estimated Fresh Catalyst	0.420	0.436
Weight Loss on Ignition (850°F, 2% O ₂), %	21.7	20.7
Analysis of Ignited Extrudates		
Carbon, W %	0.23	0.20
Hydrogen, W %	0.45	0.28
Sulfur, W %	0.64	0.49
Molybdenum, W %	8.82	8.98
Pore Volume, D >30 Å, cc/gram Extrudate ⁰	0.577	0.633
Pore Volume*, D >30 Å, cc/gram Estimated Fresh Catalyst	0.636	0.686

*assumes no loss of molybdenum

**amount of solids recovered indicative of efficiency of the shutdown oil wash period.

PORE SIZE DISTRIBUTION - RUN 177-143 DISCHARGED CATALYST

FIGURE 16

PORE SIZE DISTRIBUTION - RUN 177-143 DISCHARGED CATALYST REGENERATED AT 850°F

FIGURE 17

PORE SIZE DISTRIBUTION - RUN 177-144 DISCHARGED CATALYST

EQUIVALENT PORE DIAMETER (MICRONS) = $175/\text{PSI}$

FIGURE 18

PORE SIZE DISTRIBUTION - RUN 177-144 DISCHARGED CATALYST REGENERATED AT 850°F

FIGURE 19

IMPACT OF COAL FEEDSTOCK ON THE H-COAL PROCESS WITH VISCOSITY LIMIT

In the syncrude mode of operation, the total C_4 to 975°F distillate yield is greater for deep-cleaned coal than for the run-of-mine coal, 3.64 barrels per ton dry coal versus 3.06 barrels per ton dry coal, respectively. However, in the H-Coal Process, the total distillate yield is influenced by the pumpability of the vacuum still bottoms product. An estimated limit for the vacuum still bottoms slurry viscosity is 25 poise for pumpability. Figure 20 shows the viscosity of vacuum still bottoms slurry at 600°F as a function of the distillate to resid ratio for the deep-cleaned coal and run-of-mine coal.

Table 4 presents the yields from a 25,000 ton per day commercial H-Coal plant with a pumpable vacuum still bottoms slurry for both coals. This analysis is based on charging 25,000 ton per day dry coal to the liquefaction section and charging supplementary coal to a partial oxidation unit, in addition to the vacuum still bottoms slurry, for hydrogen balance. The total C_4 to 975°F yield for the deep-cleaned coal is approximately 10% greater than that for the run-of-mine coal.

Gases produced in the syncrude mode with Illinois No. 6 coal exceed that required for plant fuel requirements. As the deep-cleaned coal produces more gas than the run-of-mine coal, the sale of excess gas as a by-product would yield an additional credit to the deep-cleaned case.

IMPACT OF COAL FEEDSTOCK WITH DEEP VACUUM DISTILLATION

In H-Coal Process Development Unit operations, pumpability of the vacuum still bottoms product has been maintained at average solids levels as high as 45 W % with maximums to 55 W %. This process constraint would permit a deeper cut than 975°F in the vacuum still in operations with deep-cleaned coal. Since the run-of-mine coal converted to a lesser degree, had a higher ash level and yielded less residual oil compared to the deep-cleaned coal, a 975°F cut in the vacuum still would produce an unpumpable bottoms product with 69 W % solids. A lower cut point would then be required to render a pumpable vacuum still bottoms product. Operation of the vacuum still in this way results in a total C_4^+ oil yield of approximately 3.9 barrels per ton dry coal for the deep-cleaned coal and 2 barrels per ton dry coal for the run-of-mine coal. The effect of deep cleaning on total C_4^+ distillate production through a higher allowable cut point in the vacuum still is dramatic. The composition of the vacuum still bottoms streams for these cases is shown in Table 5.

FIGURE 20

CALCULATED VISCOSITY OF VACUUM BOTTOMS SLURRY
IN KENTUCKY NO. 11 COAL OPERATION FOR
VARIOUS DISTILLATE/RESID RATIO
(T = 600°F)

TABLE 4

COMMERCIAL PLANT FOR LIQUEFACTION
OF KENTUCKY NO. 11 COAL

(Basis: 25,000 T/D Dry Coal Feed to Coal Hydrogenation)

	<u>Deep Washing</u>	<u>Run-of-Mine</u>
<u>Net Liquid Products, B/D</u>		
C ₄ -400°F Naphtha	43,763	42,338
400-975°F Distillate	<u>36,337</u>	<u>29,904</u>
Total	80,100	72,242
<u>Vacuum Bottoms to Hydrogen Plant</u>		
Total T/D	7,677	9,290
Composition, W %		
Distillate	0	4.6
Residuum	66.9	35.4
Unconverted Coal + Ash	<u>33.1</u>	<u>60.0</u>
	100.0	100.0
Supplementary Coal to Hydrogen Manufacture, T/D *	4,892	5,902

*Run-of-Mine, Containing 16.3% Ash.

The requirement of hydrogen balance for the process may somewhat reduce the depth of the cut in the vacuum still in operations with deep-cleaned coal, and additional liquid product from the run-of-mine coal may be obtained by coking the vacuum still bottoms product. However, the difference in total C₄+ distillate between the two coals is still expected to be significant.

TABLE 5
VACUUM STILL BOTTOMS COMPOSITION

Basis: 100 Pounds Dry Coal Feed

Kentucky No.11 Coal	<u>Deep-Cleaned</u>		<u>Run-Of-Mine</u>	
	Normal	Adjusted	Normal	Adjusted
975°F-, Pounds	-	-	-	17.28
975°F+, Pounds	17.85	12.45	11.10	11.10
Ash, Pounds	6.21	6.21	17.49	17.49
Unreacted Coal, Pounds	3.95	3.95	6.67	6.67
W % Solids	36.3	45.0	68.5	45.0

PHYSICAL AND MECHANICAL DETAILS

UNIT DESIGN AND OPERATION

Figure 21 is a flowsheet of the H-Coal bench unit used in these operations. The reactor diameter is 0.814" and the height is 10 feet, to give a reactor volume of about 1000 cc. Expansion of the catalyst charge in the reactor is controlled by the velocity of the fluids passing through the reactor.

Feed coal was slurried with recycle oil and recycle slurry and pumped to reactor pressure with a metering pump. This feed slurry was mixed with hot recycle oil which had been pumped from the reactor outlet and reheated concurrently with make-up hydrogen in a downflow recycle oil heater (to 760-780°F) to compensate for heat losses. The hydrogen-slurry mixture was then fed to the bottom of the H-Coal reactor. After contacting the catalyst in the reactor, the hot oil recycle stream was separated from the reactor mixture at reactor temperature and the balance of vapors and liquid passed to a hot separator. The temperature of the hot separator was held about 200°F below reactor temperature. The overhead vapors from the hot separator passed to a light product cooler-condenser and cold separator to provide a vent gas stream and a light liquid product after pressure release.

The bottoms slurry from the hot separator (separator bottoms) was flashed at atmospheric pressure and a portion recycled directly as part of the coal slurry medium. The balance was fed to the two recycle oil recovery systems - a batch vacuum distillation operation and a batch plate filtration operation.

Vacuum distillation was carried out to supply distillate oil to serve as a purge medium for the seal of the hot oil recycle pump which was pumped to reactor pressure by a buffer oil pump. The vacuum bottoms which resulted became a net product of the system. Vacuum distillations were carried out at 12-hour intervals using the balance of separator bottoms available after a specified charge to the pressure filter to supply material to be recycled subsequently over an equal time period. Distillation pressure was 1-2 mm Hg (abs) and final pot temperature was 650°F which resulted in a final overhead temperature of 550-580°F equivalent to an atmospheric boiling point of about 975°F.

The batch plate filtration provided a larger portion of the recycle slurring medium and was carried out at six-hour intervals to provide slurry oil for the feed coal for the subsequent operating period. The filtration was achieved on a horizontal, approximately 12-inch-diameter plate. The filter medium was two sheets of Whatman, Grade 42, filter paper with a third sheet of Whatman, Grade 202, Reeve-Angel filter paper between. An analysis of the lower papers indicated that very little solids penetrated below the first layer. The filtration was performed at 300°F with a pressure of 150 psig imposed on the liquid by means of bottled nitrogen. Filtration was continued after nitrogen breakthrough and until the collection of additional liquid ended.

The recycled filtration oil was returned to the H-Coal system directly with the feed coal through the slurry feed pump. The final portion of the recycled material returning with the feed coal through the slurry feed pump was separator bottoms which had been neither filtered nor distilled. The recycled vacuum distillate was mixed with feed coal in the H-Coal system when the feed slurry mixed with internal recycle downstream of the preheater.

The net products of the operation were (1) the vent gas stream, (2) the two flash gas streams from pressure release of the liquids condensed at high pressures, (3) light liquids and water from the cold separator, (4) net filtered liquid remaining after recycle slurry oil requirements were met, (5) filter cake, (6) any vacuum distillate remaining after meeting recycle oil requirements, and (7) the vacuum bottoms slurry from the vacuum still. All net streams were weighed or metered and analyzed.

Startup was initiated by pressuring the unit to run pressure and establishing the unit vent rate. The reactor was then heated to 300°F at which time No.2 fuel oil was cut in to establish initial liquid rates and levels. Reactor heatup continued to 650°F and startup oil (L-467, hydrogenated anthracene oil) was cut in to replace the No.2 fuel oil. At this temperature the recycle pump was started and set for the target recycle rate. Two hours after L-467 was cut in the recycle of separator bottoms to the slurry feed pot began. Reactor heatup then continued to 850°F. L-467 was used in place of pressure filter liquid and vacuum overhead during startup.

At 850°F coal feed was started at a reduced rate of 60 grams/hour. The coal rate was increased gradually over a 14-hour period to the target rate. Pressure filtration runs were begun six hours after coal was cut in, and vacuum distillation runs were begun twelve hours after coal cut-in. Pressure filter liquid and vacuum overhead were recycled when they became available.

An analysis of L-467, hydrogenated anthracene oil used for startup, is given in Table 6. The startup oil had been prepared by hydrogenating Allied Chemical Company 24CB creosote oil "anthracene" at 2700 psig, 750°F, 1.3 V_{oil}/Hr/V_{catalyst} with American Cyanamid HDS-1442A catalyst.

TABLE 6

STARTUP OIL ANALYSIS

Gravity, °API	6.5
ASTM Distillation	
IBP, °F	432
10 V %	471
20	502
30	532
40	548
50	572
60	602
70	626
Volume % @ 650°F	78
Carbon, W %	90.08
Hydrogen, W %	8.65
Sulfur, W %	<0.03
Nitrogen, W %	0.35

COAL FEED

Two drums of Kentucky No.11 coal were provided by PETC for use in this program. The run-of-mine coal was identified as HRI No.4017 and had an ash level of 18.02 W %. The deep-cleaned coal was identified as HRI No.4018 and had an ash level of 6.42 W %. The water content and grind of both coals met the bench unit feed coal specifications as received, and no further coal preparation was required. Analyses of both coals are given in Table 7.

FEED COAL ANALYSES

TABLE 7

Run Number	177-143	177-144
Kentucky No.11 Coal Feed	Deep-Cleaned	Run-Of-Mine
HRI Number	4018	4017
Grind Description	-70 Mesh	-70 Mesh
Moisture, W %	3.14	3.30
Hours of Run	0-192	0-180
Proximate, Dry Basis, W %		
Ash	6.42	18.02
Volatile Matter	39.56	
Fixed Carbon	54.02	
Ultimate, Dry Basis, W %		
Carbon	74.27	63.29
Hydrogen	4.91	3.99
Nitrogen	1.55	1.93
Sulfur	3.70	6.30
Ash	6.42	18.02
Chlorine*	0.07	0.02
Oxygen (Difference)	9.08	6.45
Sulfur Forms, W % (Dry)*		
Pyritic	1.00	3.46
Sulfate	0.24	0.55
Organic	2.07	1.88
Total	3.31	5.89
Mineral, W % Ignited Basis*		
Phosphorous Pentoxide, P ₂ O ₅	0.29	0.26
Silica SiO ₂	38.80	37.68
Ferric Oxide Fe ₂ O ₃	31.65	33.41
Alumina Al ₂ O ₃	22.50	20.59
Titania TiO ₂	1.07	0.89
Lime CaO	1.52	1.42
Magnesia MgO	0.80	0.45
Sulfur Trioxide SO ₃	0.83	0.92
Potassium Oxide K ₂ O	2.05	1.71
Sodium Oxide Na ₂ O	0.28	0.29
Barium Oxide BaO	0.09	0.13
Strontium Oxide SrO	0.00	0.00
Manganese Oxide Mn ₃ O ₄	0.06	0.05
Undetermined	0.06	2.20
Heating Value, Btu/Lb	13,020	11,148

* Analyses performed by Commercial Testing and Engineering Company, Chicago, Illinois

BENCH UNIT FLOW SHEET

FIGURE 21

PROGRAM DETAILS

PROCESS CONDITIONS

The process conditions selected for the comparative liquefaction tests are shown in Table 8. These conditions are typical of H-Coal operations in the syncrude mode. Recycle rates were selected to ensure that the reactor slurry composition remained operable in the event of unexpectedly low residual oil or coal conversion in either of the two runs.

TABLE 8

PROCESS CONDITIONS

Space Velocity, Lbs/Hr/Ft ³	31.2
Coal Feed Rate, Lbs/Hr	1.1
Recycle Rates, Lbs/Lb Coal	
Separator Bottoms	4.0
Pressure Filter Liquid	0.7
Vacuum Overhead	0.3
Excess Gas Rate, SCFH	26.0
Reactor Temperature, °F	850
Pressure, psig	2250
Internal Recycle Rate, cc/Hr	23,120

For each run a 450 ml charge of American Cyanamid HDS-1442A catalyst, HRI 3830, was used. Each catalyst charge was riffled from the same lot of HRI No.3830 and then calcined at 850°F for one hour.

American Cyanamid HDS-1442A catalyst is commercially available, and is the standard catalyst used in H-Coal operations. It is a moderate-density, cobalt-molybdate-on-alumina preparation and contains 0.691 cc/gram pore volume. The pore size distribution is bi-modal, with about 30% of the pore volume having diameters in the 500-5000 Å range and 60% in the 40-100 Å range, centered around 55 Å. Typical catalyst analyses and pore size distributions for this catalyst are given in Table 9 and Figure 22, respectively.

TABLE 9

TYPICAL CATALYST ANALYSIS - AMERICAN CYANAMID HDS-1442A

HRI Number	3830
Bulk Density, gm/cc, Compacted	0.580
Particle Density, gms/cc	
In Mercury	0.983
In Heptane	0.908
From Weight Dimensions	0.917
Skeletal Density, gms/cc	
In Mercury, 60,000 psi	2.93
In Heptane	3.57
Heptane Wetted Density, gms/cc	1.445
Heptane Absorbed, cc/gm	0.821
Average Particle Weight, Milligram	9.20
Particle Length	
Average, mm	4.69
Range, mm	2.4-12.0
Particle Diameter, Average, mm	1.65
Chemical Analysis by Supplier	
MoO ₃ , W %	16.0
Co, W %	3.2
Na ₂ O, W %	0.02
Analysis by HRI	
Molybdenum, W %	9.73
Cobalt, W %	2.75
Iron, W %	0.3
Titanium, W %	0.0
Crush Strength, Lb/mm	3.2
Attrition Test, % Undersize	3.9
Pore Volume (Hg @ 60,000 psi)	
D > 30 Å, cc/gm	0.691
Pore Size Distribution, cc/gm	
D = 30-50 Å	0.142
D = 50-100 Å	0.259
D = 100-200 Å	0.036
D = 200-2000 Å	0.120
D = > 2000 Å	0.134
Maximum, Å*	55

* Position of Greatest Slope of Pore Volume Against Diameter

PORE SIZE DISTRIBUTION OF H-COAL CATALYST

HRI NO. TYPE
— 3830 STANDARD CATALYST HDS 1442A COBALT MOLYBDATE

FIGURE 22

RUN SUMMARY

Four bench unit H-Coal runs were conducted during this program. In the initial two runs, restrictions in the overhead line and in the slurry feed line caused unplanned terminations. In the final two runs, successful continuous operation of approximately eight days' duration was achieved for both the deep-cleaned coal and the run-of-mine coal. A brief summary of each of the four runs follows, and a detailed operating history is given in Appendix B.

Run 177-141

The feed for this run was run-of-mine Kentucky No.11 coal, HRI No.4017. Operations began on September 25, 1978. After twelve hours of coal operation a plug developed in the line to the overhead separator. The charge pumps and hydrogen feed were lost due to the high unit pressure, and the run was terminated.

Run 177-142

Run-of-mine Kentucky No.11 coal HRI No. 4017 was used as feed in this run. Coal operations began on September 30. Restrictions again began developing in the line to the overhead receiver. The plug was in the valve just downstream of the bottoms separation and a water purge into the bottoms separator was used to control this restriction. On October 2 the reactor plugged when the feed pump was lost for an extended period, and the run was terminated.

Run 177-143

Several unit modifications were made prior to this run to eliminate the difficulties experienced in the two earlier runs. The coal feed for this run was deep-cleaned Kentucky No.11. Coal operations began on October 6 and continued for eight full periods until October 14 when unit shutdown was voluntarily initiated.

Run 177-144

The coal feed for this run was run-of-mine Kentucky No.11. Coal operations began on October 17, 1978. Unit shutdown was begun on October 25 when the supply of this coal feed was exhausted. Seven-and-one-half days of coal operations were achieved.

APPENDIX A

DETAILED DAILY OPERATING DATA

RUN 177-143 - DEEP-CLEANED KENTUCKY NO.11 COAL (HRI 4018) -70 MESH GRIND
AMERICAN CYANAMID HDS-1442A CATALYST (HRI 3830) 1/16" DIAMETER, 271.0 GRAMS, 450 cc

Period	1A	1B	2	3*	4	5	6	7	8A	8B
Hours of Run, Beginning-End	0-12	12-24	24-48	48-72	72-96	96-120	120-144	144-168	168-180	180-192
Catalyst Age, Lbs Coal/Lb Catalyst										
Beginning	0	10.1	31.8	76.0	120.2	162.8	207.0	251.3	295.6	317.8
End	10.1	31.8	76.0	120.2	162.8	207.0	251.3	295.6	317.8	337.8
<u>OPERATING CONDITIONS</u>										
Coal Feed, % Moisture		3.36	3.25	3.34	3.48	3.30	3.81	3.28		3.45
% Ash (Dry Coal)		5.77	5.84	6.01	6.40	5.95	6.41	6.34		6.53
% Iron on Ash		22.23	22.63	23.20	23.37	22.46	22.23	21.54		23.20
Coal Feed Rate, Lbs/Hr/Ft ³ Reactor	14.2	30.6	31.2	31.2	30.0	31.2	31.2	31.2	31.2	28.2
Lbs/Hr/Ft ³ Catalyst	31.5	68.0	69.2	69.2	66.7	69.2	69.4	69.4	69.4	62.8
Hydrogen Rate, MSCF/Ton Coal										
Reactor Outlet (Vapor Phase)	87.6	46.2	45.0	42.3	43.3	44.4	42.3	41.0	39.9	47.7
Reacted (Hydrogen Balance)			25.1	23.8	24.1	23.3	22.6	22.4	21.2	
Slurry Oil, Recycled Filtered Oil										
Lbs/Lb Coal	0.769	0.714	0.700	0.715	0.685	0.700	0.700	0.656	0.700	0.699
Composition - IBP, °F	-	345	346	402	376	379	346	394		356
EP, °F	-	975	975	975	975	975	975	975		975
V % @ 650°F	-	65.5	51	48	44	44	44	40		43
W % Residue @ EP	-	11.9	24.3	27.7	28.9	29.8	29.5	29.5		28.2
Hot Separator Reactor Slurry Recycle,										
Lbs/lb Coal	8.85	4.47	4.40	4.06	4.36	4.30	4.15	3.80	3.86	3.76
W % Distillate to 975°F	-	85.55	72.73	71.87	71.80	67.38	68.44	70.92	66.74	68.49
Non-Distillate Oil, 975°F										
Toluene-Soluble	-	7.23	16.71	16.29	16.89	20.47	19.81	17.20		19.80
Toluene-Insoluble	-	0.65	3.59	5.24	4.58	5.67	5.44	5.71		5.37
Unreacted Ash-Free Coal	-	3.44	2.81	2.63	2.55	2.39	2.33	2.36		2.39
Ash	-	3.13	4.16	3.97	4.18	4.09	3.98	3.81		3.95
Vacuum Distillate Recycle, Lbs/Lb Coal	0.000	0.255	0.200	0.288	0.313	0.275	0.300	0.396	0.30	0.332
Coal in Gross Feed Slurry, W %	7.5	15.4	15.9	16.5	15.7	15.9	16.2	17.1	17.1	17.3
Make-Up Oil, Lbs/Lb Coal	2.64	0.071	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Temperature, °F										
Reactor	851	851	851	850	850	850	850	850	850	849
Preheater	783	788	786	786	787	787	782	784	788	784
Hot Separator, Reactor Outlet	583	583	579	587	596	592	605	605	609	610
Pressure, Reactor Outlet psig	2250	2235	2235	2240	2240	2245	2250	2245	2250	2240
% Hydrogen in Vent Gas	93.2	92.8	91.3	90.3	89.9	90.1	89.7	89.7	88.9	93.9
Hydrogen Partial Pressure										
Hot Separator, psig	1935	1811	1763	1742	1736	1754	1744	1746	1713	1837
<u>OPERATING RESULTS</u>										
Collected Products, W % of Feed Coal										
Light Liquid Hydrocarbons	58.32	24.32	27.63	22.33	23.19	20.99	20.63	18.89	19.70	21.21
Heavy Liquid (Vacuum Distillate)	242.56	26.53	9.90	13.50	13.57	15.13	15.14	13.79	15.58	-13.64
Filtered Liquid	-	-	-	-	-	-	-	-	-	-
Vacuum Still Bottoms	1.10	8.96	9.33	16.85	18.16	21.33	21.47	23.59	23.42	9.27
Filter Cake	2.01	4.93	6.67	7.59	6.79	7.10	7.10	6.45	7.22	6.78
C ₁ -C ₃ Hydrocarbons in Gas Product	12.91	6.83	9.96	10.96	11.58	11.19	11.61	11.20	11.37	6.61
C ₄ -C ₆ Hydrocarbons in Gas Product	29.30	9.42	11.88	11.72	12.33	13.14	11.55	12.17	14.24	13.30
CO ₂ + CO	0.96	0.43	0.52	0.56	0.71	0.67	0.61	0.57	0.47	0.30
Collected Water Phase (w/NH ₃ & H ₂ S)	9.67	12.48	13.28	13.38	13.47	13.56	13.49	12.62	13.82	13.21
H ₂ S in Gas Products	2.37	1.55	2.03	2.09	2.08	2.14	2.12	2.16	2.27	1.03
Total Collected Products	359.20	95.45	91.20	98.98	101.88	105.25	103.72	100.74	108.09	58.07**
Normalized Collected Products			106.67	106.33	106.39	106.20	106.01	105.96	105.64	-
Vacuum Bottoms, Concentration Charge										
W % of Unit Inventory (9200 Grams)	17.3		10.7	1.1	2.2	0.4	-----	0.5	-----	
W % of Coal Feed	18.5		8.2	0.84	1.69	0.16	-----	0.19	-----	
Coal Conversion, W % (M.A.F.)			95.82	95.76	95.83	96.31	95.99	95.81	95.80	95.80

* Key period, with complete elemental and distillation analyses. Other periods use interpolated values from key period.

** Switched slurry feed pump and feed slurry holder at 188 hours of run. Lost indeterminate amount of feed slurry.

Period	1A	1B	2	3	4	5	6	7	8A	8B
OPERATING RESULTS (CONTINUED)										
Normalized Product Distribution, W % of Dry Coal										
CO ₂			0.39	0.49	0.52	0.39	0.36	0.42	0.46	
CO			0.24	0.13	0.24	0.30	0.29	0.20	0.00	
C ₁ -C ₃ Hydrocarbons			12.01	12.14	12.47	11.62	12.31	12.07	11.47	
C ₄ in Gas Product			4.48	5.13	5.19	5.22	5.02	5.24	5.83	
C ₅ in Gas Product			8.55	6.91	6.96	7.25	6.20	6.40	7.48	
C ₆ in Gas Product			1.30	0.92	1.13	1.18	1.01	1.48	1.05	
IBP-400°F in Liquid			17.38	14.33	14.25	13.06	13.05	11.44	11.21	
400-650°F Distillate			18.59	20.89	20.19	18.62	17.83	17.18	17.40	
650-975°F Distillate			1.77	5.29	4.91	7.09	8.72	8.24	8.57	
975°F+ Residual Oil										
Toluene-Soluble			13.24	11.78	12.93	14.03	14.55	15.42	15.24	
Toluene-Insoluble			2.89	3.73	3.66	4.14	4.08	5.14	4.11	
Unreacted Ash-Free Coal			4.32	4.28	4.00	3.72	3.66	3.88	3.77	
Ash			6.40	6.45	6.55	6.36	6.26	6.27	6.18	
Water (Net Production)			11.24	10.05	9.60	9.40	9.05	8.97	9.19	
NH ₃ (Forced Nitrogen)			1.36	1.32	1.31	1.26	1.14	1.10	1.12	
H ₂ S (Forced Sulfur)			2.75	2.68	2.70	2.76	2.71	2.69	2.75	
Total (100.0 + Hydrogen Reacted)			106.89	106.55	106.62	106.41	106.25	106.16	105.84	
Normalized Net C₄+ Liquid Recovery										
W % of Dry Coal, Toluene-Soluble			65.32	65.27	65.57	66.45	66.38	65.40	66.78	
Toluene-Insoluble:			2.89	3.73	3.66	4.14	4.08	5.14	4.11	
Barrels per Ton of Dry Coal			4.51	4.45	4.46	4.50	4.41	4.40	4.48	
Barrels per Ton of M.A.F. Coal			4.82	4.76	4.77	4.80	4.71	4.70	4.78	
Barrels C ₄ to 975°F- Distillate per Ton M.A.F. Coal			4.02	3.99	3.95	3.91	3.79	3.68	3.83	
COLLECTED H-COAL PRODUCT ANALYSES										
Light Liquid Hydrocarbons										
Gravity, °API	35.6	34.2	32.3	32.6	32.4	31.9	32.3	31.7	31.5	31.5
IBP, °F	160	156	142	164	138	138	145	149	147	147
10 V %	235	235	230	233	225	227	226	220	218	218
30	311	319	328	310	312	300	300	306	298	298
50	386	382	393	382	386	375	368	378	373	373
70	439	438	442	435	436	432	432	435	435	435
90	508	506	509	515	522	510	532	530	520	520
EP	595	608	610	630	617	618	620	620	626	626
V % @ 400°F	56	58	55	58	57	60	61	58	58	58
Carbon, W %				87.21				87.94		
Hydrogen, W %				11.64				11.89		
Sulfur, W % (Caustic-Washed)	<0.030	<0.030	0.04	<0.03	0.04	<0.03	<0.03	0.09	0.05	
Nitrogen, W %				0.250				0.36		
Viscosity, SUS @ 100°F	29	30	30	29	30	31	31	31	30	
Vacuum Distillate										
Gravity, °API		14.8	11.0	10.1	9.0	10.2	5.5	5.3		6.8
IBP, °F				334				367		
10 V %				421				459		
30				492				545		
50				566				627		
70				646				724		
90				808				864		
EP				913				950		
V % @ 650°F				71				55		
Residue, W %				3.5				3.2		
Carbon, W %				88.53				88.77		
Hydrogen, W %				8.93				8.31		
Sulfur, W %		<0.03	0.14	<0.03	0.22	<0.03	0.05	0.06	0.06	
Nitrogen, W %				0.62				0.86		
Viscosity, SUS @ 130°F		36	41	41	49	40	53	55		46
Filtered Liquid										
Gravity, °API		9.3	3.0	1.2	0.2	-1.0	-2.0	-1.6		-3.0
IBP, °F		345	346	402	376	379	346	394		356
10 V %		436	456	475	472	473	460	467		472
30		506	544	556	558	583	569	600		577
50		579	648	702	702	736	733	764		741
70		706	873	894	893	938	900	920		900
90		975	-	-	-	-	-	-		-
EP		975	975	975	975	975	975	975		975
V % @ 400°F		2.5	2	-	1.5	1	2	<1		<1
V % @ 650°F		65.5	51	48	44	41	44	40		43
V % @ EP		89.5	79	75.5	75	74	76.5	75		77
W % Residue @ EP		11.9	24.3	27.7	28.9	29.8	29.5	29.5		28.2
Carbon, W %				88.94				88.65		
Hydrogen, W %				7.83				7.44		
Sulfur, W %		0.08	0.12	0.15	0.13	0.23	0.14	0.19	0.19	
Nitrogen, W %				0.93				1.04		
Viscosity, SUS @ 210°F		66	52	53	66	76	83	72		79

Period	1A	1B	2	3	4	5	6	7	8	9
COLLECTED H-COAL PRODUCT ANALYSES (CONTINUED)										
Vacuum Still Bottoms Slurry										
W % of Separator Bottoms	0.45	14.70	27.74	28.53	28.80	33.32	32.24	30.70	33.94	32.19
Toluene-Soluble Oil (Soxhlet), W %		50.82	61.83	58.63	59.69	62.16	63.00	60.18	63.81	63.63
Toluene-Insoluble, Organic, W %		27.87	23.12	27.50	25.41	25.10	24.46	27.04	23.89	24.10
Estimated Insoluble Filterable Oil, W %		4.44	12.97	18.29	16.32	17.66	17.13	19.13	16.39	16.68
Estimated Unreacted Coal, W %		23.43	10.15	9.21	9.09	7.44	7.33	7.91	7.50	7.42
Ash, W %		21.31	15.05	13.87	14.90	12.74	12.54	12.78	12.30	12.27
Carbon, W %				76.48				77.48		
Hydrogen, W %				4.67				4.68		
Sulfur, W %		3.44	2.72	2.64	2.66	2.44	2.58	2.54		2.32
Nitrogen, W %				1.33				1.55		
IBP, °F				975				960	968	
V % @ EP				0				1	0	
W % Residue @ EP				98.3				97.5	98.0	
Filter Cake										
W % of Separator Bottoms	2.55	6.47	8.68	9.61	9.01	9.24	9.22	8.96	9.35	8.83
Toluene-Soluble Oil, W %		47.41	31.80	24.68	24.63	23.54	27.51	29.30		25.57
Toluene-Insoluble, Organic, W %		27.70	28.23	30.93	29.35	29.15	27.82	28.21		29.02
Estimated Toluene-Insoluble Oil, W %		0.33	1.28	1.47	1.27	1.52	1.70	1.90		1.55
Estimated Unreacted Coal, W %		27.37	26.95	29.46	28.08	27.63	26.12	26.31		27.47
Ash, W %		24.89	39.97	44.39	46.02	47.31	44.67	42.49		45.41
Carbon, W %				43.53				45.37		
Hydrogen, W %				2.83				3.06		
Sulfur, W %		5.95	6.53	7.69	7.51	7.35	7.12	6.95		6.96
Nitrogen, W %				0.67				0.69		
Iron, W % of Ash		25.23	24.26	24.46	22.52	22.90	24.91	24.05		25.24
975°F+ Residual Oil										
W % of Filtered Liquid		11.9	24.3	27.7	28.9	29.8	29.5	29.5		28.2
Toluene-Soluble Oil (Soxhlet), W %		78.88	78.48	81.97	75.25	75.13	81.78	70.46		73.60
Toluene-Insoluble, Organic, W %		19.20	21.20	17.85	24.11	24.61	17.64	27.67		25.80
Ash, W %		0.92	0.32	0.18	0.64	0.26	0.58	1.87		0.60
Sulfur, W %		0.61	0.46	0.60	0.54	0.48	0.57	0.44		0.44
Nitrogen, W %				1.65				1.87		
Collected Water Product										
Gravity, °API	7.8	7.2	6.9	6.8	6.8	6.8	6.6	6.7	6.8	6.6
Sulfur, W %				3.58				3.78		
Ammonia, W %				5.11				5.07		
Carbon, W %				1.74				0.18		
Phenols, mg/l				9040				9280		
COLLECTED H-COAL PRODUCT FRACTION ANALYSES										
Light Liquid Hydrocarbon Cuts										
IBP-400°F										
Gravity, °API				43.0				41.4		
Carbon, W %				85.38						
Hydrogen, W %				13.13						
Sulfur, W % (H ₂ S-Free)				<0.03						
Nitrogen, W %				0.13				0.18		
400°F-End Point										
Gravity, °API				20.7				18.9		
Sulfur, W % (H ₂ S-Free)				<0.03				<0.03		
Nitrogen, W %				0.32				0.53		
Vacuum Still Overhead Cuts										
IBP-650°F										
Gravity, °API				15.3				14.5		
Sulfur, W %				<0.03				<0.03		
650°F-End Point										
Gravity, °API				-0.1				-2.9		
Sulfur, W %				0.09				<0.03		
Nitrogen, W %								1.12		
Filtered Liquid Cuts										
IBP-650°F										
Gravity, °API				14.5				13.1		
650-975°F										
Gravity, °API				-1.0				-3.6		
975°F+ Residue										
Sulfur, W %		0.61	0.46	0.60	0.54	0.48	0.57	0.44		0.44
Nitrogen, W %				1.65				1.87		
Vacuum Still Bottoms										
IBP-975°F Oil										
Sulfur, W %								0.41		
Nitrogen, W %								1.33		

RUN-177-144 - RUN-OF-MINE KENTUCKY NO.11 COAL (HRI 4017) -70 MESH GRIND

AMERICAN CYANAMID HDS-1442A CATALYST (HRI 3830) 1/16" DIAMETER, 264.8 GRAMS, 450 CC

Period	1	2A	2B	3	4*	5	6	7	8
Hours of Run, Beginning-End	0-12	12-24	24-36	36-60	60-84	84-108	108-132	132-156	156-180
Catalyst Age, Lbs Coal/Lb Catalyst									
Beginning	0	10.3	32.5	55.1	100.5	145.8	191.7	236.4	281.8
End	10.3	32.5	55.1	100.5	145.8	191.7	236.4	281.8	327.1
OPERATING CONDITIONS									
Coal Feed, W % Moisture	2.87		3.07	3.08	3.34	3.35	3.36	3.57	3.50
W % Ash (Dry Coal)	18.76		18.27	17.33	17.83	17.62	17.37	17.44	17.34
W % Iron on Ash	21.43		20.79	20.87	20.48	18.85	18.74	18.96	21.84
Coal Feed Rate, Lbs/Hr/Ft ³ Reactor	14.2	30.6	31.2	31.2	31.2	31.2	31.2	31.2	31.2
Lbs/Hr/Ft ³ Catalyst	31.5	68.0	69.4	69.4	69.4	69.4	69.4	69.4	69.4
Hydrogen Rate, MSCF/Ton Feed Coal									
Reactor Outlet (Vapor Phase)	92.8	46.8	43.0	42.8	43.7	43.1	40.2	43.4	44.5
Reacted (Hydrogen Balance)				23.3	21.0	21.6	21.2	20.6	21.7
Slurry Oil, Recycled Filtered Oil, Lbs/Lb Coal	0.58	0.71	0.70	0.63	0.69	0.70	0.70	0.70	0.70
Composition									
IBP, °F	341		344	357	379	349	392	369	374
EP, °F	888		975	975	975	975	975	975	975
V % @ 650°F	88		69	58	51.5	52	47	47	46
W % Residue @ EP	3.0		13.5	18.2	19.8	22.4	21.4	21.0	22.4
Hot Separator Reactor Slurry Recycle, Lbs/Lb Coal	7.13	3.69	4.08	4.39	4.04	4.15	4.14	4.03	4.04
W % Distillate to 975°F	97.93	87.39	78.79	70.93	69.23	66.38	65.19	66.89	66.08
Non-Distillate Oil, 975°F+									
Toluene-Soluble			7.37	8.43	10.35	14.23	16.27	14.71	15.52
Toluene-Insoluble			2.71	2.12	2.78	1.24	2.47	2.56	3.23
Unreacted Ash-Free Coal			2.98	4.91	4.67	4.99	4.25	4.38	4.08
Ash			8.15	13.61	12.97	13.16	11.82	11.46	11.09
Vacuum Distillate Recycle, Lbs/Lb Coal	0	0.29	0.30	0.27	0.30	0.30	0.29	0.30	0.30
Coal in Gross Feed Slurry, W %	8.2	17.2	16.4	15.9	16.6	16.3	16.3	16.6	16.6
Make-Up Oil, Lbs/Lb Coal	2.83	0.133	0	0	0	0	0	0	0
Temperature, °F									
Reactor	850	849	850	850	850	850	850	851	851
Preheater	778	785	786	786	788	786	785	785	787
Hot Separator, Reactor Outlet	584	599	618	608	596	596	600	601	598
Pressure, Reactor Outlet, psig	2245	2245	2255	2260	2250	2245	2250	2250	2255
% Hydrogen in Vent Gas	95.6	93.5	92.4	91.6	92.2	91.8	91.0	92.4	91.7
Hydrogen Partial Pressure, Hot Separator, psig	2012	1836	1759	1749	1772	1752	1733	1777	1791
OPERATING RESULTS									
Collected Products, W % of Feed Coal									
Light Liquid Hydrocarbons	48.79	23.57	23.87	29.53	23.39	22.16	19.80	19.39	20.29
Heavy Liquid (Vacuum Distillate)	134.32	22.94	7.35	1.86	7.74	4.08	4.88	11.13	10.75
Filtered Liquid	130.73	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Vacuum Still Bottoms	2.89	7.58	10.28	12.21	17.44	17.77	18.41	20.86	21.44
Filter Cake	3.74	7.76	14.37	17.01	18.64	18.25	17.57	15.94	15.83
C ₁ -C ₃ Hydrocarbons in Gas Product	13.02	8.40	9.41	9.17	9.24	9.90	9.89	10.11	8.45
C ₄ -C ₆ Hydrocarbons in Gas Product	9.86	6.66	6.80	9.99	7.89	8.12	8.82	6.20	11.17
CO ₂ + CO	0.34	0.34	0.21	0.49	0.39	0.41	0.49	0.32	0.51
Collected Water Phase (w/NH ₃ & H ₂ S)	6.59	11.21	14.62	13.55	13.42	13.97	13.73	13.75	13.71
H ₂ S-in-Gas-Products	1.23	2.76	3.53	3.51	3.02	3.13	3.22	2.91	2.92
H ₂ O Injected	0	0	-0.97	0	0	-0.40			
Total Collected Products	351.51	91.22	89.47	97.32	101.17	97.39	96.81	100.61	105.07
Normalized Collected Products				106.19	105.59	105.75	105.64	105.48	105.77
Vacuum Bottoms, Concentration Charge									
W % of Unit Inventory (9190 Grams)	7.5	9.7	6.1	6.4	1.7	0.8	0.8	-----	0.5 -----
W % of Coal Feed	25.2	15.2	9.3	4.9	1.30	0.61	0.61	-----	0.19 -----
Coal Conversion, W % (M.A.F.)			91.82	92.44	92.19	91.88	92.44	91.42	92.28

* Key period, with complete elemental and distillation analyses. Other periods use interpolated values from key periods.

Period	1	2A	2B	3	4	5	6	7	8
OPERATING RESULTS (CONTINUED)									
Normalized Product Distribution, W % of Dry Coal									
CO ₂				0.37	0.20	0.16	0.80	0.11	0.30
CO				0.18	0.21	0.30	0.33	0.24	0.23
C ₁ -C ₃ Hydrocarbons				10.31	9.89	11.04	10.54	10.87	8.70
C ₄ in Gas Product				5.70	4.56	4.45	4.74	4.62	4.16
C ₅ in Gas Product				4.16	2.79	3.56	3.87	0.87	6.39
C ₆ in Gas Product				1.38	1.08	1.05	1.32	1.18	0.95
IBP-400°F in Liquid				16.68	13.08	13.72	13.16	11.61	12.26
400-650°F Distillate				16.31	19.70	16.35	14.72	19.23	18.03
650-975°F Distillate				0.72	3.77	2.88	3.37	4.77	4.60
975°F+ Residual Oil									
Toluene-Soluble				7.20	7.60	10.07	10.23	10.57	10.89
Toluene-Insoluble				1.33	1.97	0.84	1.73	1.89	2.27
Unreacted Ash-Free Coal				6.86	6.79	7.04	6.60	6.73	6.00
Ash				19.01	18.88	18.54	18.36	17.59	16.29
Water (Net Production)				10.42	9.37	9.97	10.23	9.43	8.79
NH ₃ (Forced Nitrogen)				2.03	1.99	1.93	1.93	1.89	1.93
H ₂ S (Forced Sulfur)				3.73	3.87	4.03	3.91	4.06	4.19
Total (100.0 + Hydrogen Reacted)				106.39	105.78	105.95	105.84	105.68	105.98
Normalized Net C ₄ + Liquid Recovery									
W % of Dry Coal, Toluene-Soluble				52.14	52.59	52.09	51.41	52.86	57.28
Toluene-Insoluble				1.33	1.97	0.84	1.73	1.89	2.27
Barrels per Ton of Dry Coal				3.63	3.54	3.45	3.46	3.44	3.86
Barrels per Ton of M.A.F. Coal				4.48	4.36	4.23	4.24	4.18	4.61
Barrels C ₄ to 975°F= Distillate									
per Ton M.A.F. Coal				3.97	3.80	3.60	3.55	3.46	3.88
COLLECTED H-COAL PRODUCT ANALYSES									
Light Liquid Hydrocarbons									
Gravity, °API	25.2	26.9	29.3	29.1	30.8	31.8	32.9	32.7	33.4
IBP, °F	175	175	162	160	158	148	149	151	156
10 V %	265	246	234	232	226	221	218	232	220
30	362	347	321	330	318	310	294	317	295
50	416	405	387	393	392	381	372	384	370
70	449	442	435	443	443	440	432	442	437
90	508	500	487	505	517	504	509	547	518
EP	598	592	590	610	614	612	608	620	612
V % @ 400°F	44	48	57	54	54	58	59	56	60
Carbon, W %					87.23				86.73
Hydrogen, W %					11.32				11.88
Sulfur, W % (H ₂ S-Free)	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
Nitrogen, W %					0.14				0.19
Viscosity, SUS @ 100°F	31	30	31	31	30	30	30	29	31
Vacuum Distillate									
Gravity, °API	10.1		11.5	9.4	11.4	11.0	9.6	12.2	11.6
IBP, °F					321				315
10 V %					418				443
30					482				505
50					539				560
70					620				622
90					820				730
EP					925				812
V % @ 650°F					76				76
Residue @ EP, W %					2.6				2.3
Carbon, W %					88.66				89.17
Hydrogen, W %					9.09				9.29
Sulfur, W %	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
Nitrogen, W %					0.36				0.35
Viscosity, SUS @ 130°F	35		35	41	38	39	42	37	38
Filtered Liquid									
Gravity, °API	9.9		7.0	5.1	4.2	3.3	2.7	3.8	3.0
IBP, °F	341		344	357	379	349	392	369	374
10 V %	435		442	453	461	449	483	454	468
30	475		500	517	543	537	569	560	563
50	517		564	607	640	637	658	686	744
70	574		686	764	804	800	816	835	817
90	675								
EP	888		975	975	975	975	975	975	975
V % @ 400°F	2		2	2	< 1	2	< 1	< 1.5	< 2
V % @ 650°F	88		69	58	51.5	52	47	47	46
V % @ EP	97.5		89	86.5	82.5	81	82	81	80
Residue @ EP, W %	3.0		13.5	18.2	19.8	22.4	21.4	21.0	22.4
Carbon, W %					90.02				89.80
Hydrogen, W %					8.39				8.29
Sulfur, W %	< 0.03		< 0.03	< 0.03	< 0.03	0.03	0.04	0.07	< 0.03
Nitrogen, W %					0.61				0.70
Viscosity, SUS @ 210°F	28		33	37	40	42	49	45	43

Period	1	2A	2B	3	4	5	6	7	8
<u>COLLECTED H-COAL PRODUCT ANALYSES (CONTINUED)</u>									
Vacuum Still Bottoms Slurry									
W % of Separator Bottoms	2.11	12.83	21.58	29.57	31.59	34.20	35.44	33.68	34.48
Toluene-Soluble Oil (Soxhlet), W %			35.84	35.57	34.78	46.46	45.45	44.89	46.67
Toluene-Insoluble, Organic, W %			26.38	21.94	23.78	17.43	19.78	20.79	21.19
Estimated Insoluble Filterable Oil,			12.55	6.61	8.87	3.47	7.28	7.67	9.36
Estimated Unreacted Coal, W %			13.83	15.33	14.91	13.96	12.50	13.12	11.83
Ash, W %			37.78	42.49	41.44	36.11	34.77	34.32	32.14
Carbon, W %					52.41				58.36
Hydrogen, W %					3.46				3.83
Sulfur, W %			5.68	5.92	5.63	4.86	4.91	4.61	4.19
Nitrogen, W %					0.79				0.90
IBP, °F					975				968
V % @ EP					0				0
Residue, W %					98.3				98.3
Filter Cake									
W % of Separator Bottoms	1.94	9.79	17.03	21.16	21.28	20.68	20.06	18.54	18.44
Toluene-Soluble Oil, W %	38.09		24.53	22.22	26.98	25.05	24.03	21.04	23.28
Toluene-Insoluble, Organic, W %	25.39		20.79	21.06	20.01	20.90	20.63	22.32	21.31
Estimated Toluene-Insoluble Oil, W %			0.77	0.59	0.94	0.39	0.73	0.66	0.92
Estimated Unreacted Coal, W %			20.02	20.47	19.07	20.51	19.90	21.66	20.39
Ash, W %	36.52		54.68	56.72	53.01	54.05	55.34	56.64	55.41
Carbon, W %					37.09				31.26
Hydrogen, W %					3.01				2.31
Sulfur, W %	4.60		7.75	8.60	7.63	7.40	7.91	8.21	8.59
Nitrogen, W %					0.38				0.39
Iron, W % of Ash	19.95		24.76	23.03	22.02	20.17	20.67	22.28	22.24
975°F+ Filter Liquid									
W % of Filtered Liquid	3.0		13.5	18.2	19.8	22.4	21.4	21.0	22.4
Toluene-Soluble Oil (Soxhlet), W %			95.48	91.41	84.39	89.22	83.82	82.37	81.71
Toluene-Insoluble, Organic, W %			4.33	8.51	15.36	10.33	16.05	16.87	18.01
Ash, W %			0.19	0.08	0.25	0.45	0.13	0.76	0.28
Sulfur, W %	0.68		0.38	0.49	0.38	0.45	0.67	0.33	0.48
Collected Water Product									
Gravity, °API	10.0	8.0	7.2	6.8	7.0	6.9	6.8	7.0	7.0
Sulfur, W %					4.98				5.78
Ammonia, W %					5.40				5.59
Carbon, W %					1.01				1.08
Phenols, mg/l					4400				4160
<u>COLLECTED H-COAL PRODUCT FRACTION ANALYSES</u>									
Light Liquid Hydrocarbon Cuts									
IBP-400°F									
Gravity, °API					42.3				44.0
400°F-End Point									
Gravity, °API					19.3				20.4
Sulfur, W % (H ₂ S-Free)					< 0.03				< 0.03
Vacuum Still Overhead Cuts									
IBP-650°F									
Gravity, °API					15.1				15.0
Sulfur, W %					< 0.03				< 0.03
650°F-End Point									
Gravity, °API					1.1				2.1
Sulfur, W %					< 0.03				< 0.03
Filtered Liquid Cuts									
IBP-650°F									
Gravity, °API					14.8				14.9
650-975°F									
Gravity, °API					0.0				1.1
975°F+ Residue									
Sulfur, W %					0.38				0.48

Run 177-141

Unit startup began on September 24, 1978. Kentucky No. 11, run-of-mine coal (HRI 4017), was used as feed. Unit back pressure was set at 2250 psig, and hydrogen and fuel oil flows were established at 2245 hours on September 24, 1978. Reactor heatup also began at this time. With the reactor at 650°F, reactor feed was switched to hydrogenated anthracene oil and the internal recycle flow was established. Period 1A began when coal was cut in with the reactor at 850°F on September 25. The charge pump kicked off several times between 0300 and 0330 hours on September 26, 1978. At 0345 hours when the charge pump again kicked off, the pump was taken off line, depressured, circulated and put back on line. The overall unit pressure drop suddenly increased to greater than 50 psi at 0445 hours. However, the reactor pressure drop remained normal at 8-9 psi.

The charge pump kicked off six times between 0700 and 0730 hours. The overall unit pressure drop remained high, exceeding 50 psi, while the reactor pressure drop remained normal. At 0830 hours on September 26, reactor inlet pressure increased to 3150 psig with the unit back pressure at 2100 psig. Fresh feed hydrogen flow was lost because the reactor inlet pressure exceeded the hydrogen compressor discharge pressure. The buffer pump was lost due to the high pressure and the charge pump repeatedly kicked off. At 0835 hours, coal was cut out. The pressure switch setting on the right charge pump was increased and the pump was cut in with hydrogenated anthracene oil. At 0900 hours the windings on the reactor and pre-heater were cut and unit shutdown was initiated. Reactor pressure drop remained normal during this time.

A pressure gauge installed on the top of the hot separator indicated 3000 psig at this point. The cold separator was at 1500 psig, indicating the plug was in the overhead line to the cold separator. The plug broke at 0930 hours; overall unit pressure drop decreased to 7.5 psi. However, unit shutdown continued, since the reactor was without hydrogen or oil flows for approximately one hour. At 1000 hours, separator bottoms could not be withdrawn from the unit and it became necessary to take all reactor liquid through the overhead line and overhead separator.

On September 27, when the unit was down in pressure and temperature, the reactor bottom head was removed and the catalyst fell freely from the reactor. The overhead line to the cold separator was removed and inspected. No restriction was found in the line; the liquid taken overhead in this line during the shutdown probably dislodged or dissolved any remaining material from the plug.

Turnaround maintenance was performed for Run 177-142.

RUN 177-142

Unit startup began on September 29, 1978. The feed used was Kentucky No.11, run-of-mine coal. Startup oil and gas flows were established by 2000 hours and reactor heatup began. Internal recycle was established by 1245 hours.

Period 1A began at 0900 hours on September 30 when coal was cut in. During this period the bottoms letdown valves plugged several times and were cleared by blowing with high pressure hydrogen. At 2355 hours, a restriction occurred in the front end of the system following difficulties with the charge pump. This restriction cleared a few minutes later.

At 0900 hours on October 1, at the beginning of Period 2A, a 50-pound pressure drop developed between the bottoms and overhead separators. This was the same location in which a plug had developed in the earlier run, causing the run to terminate. When the valve in the line to the overhead separator was turned the restriction broke and overall pressure drop returned to normal. The line was insulated after this incident and a pump to inject water into the vapor space of the bottoms separator was installed.

The overall pressure drop began building again at 1300 hours on October 1. The water injection pump was cut in to control the drop and appeared to be effective, although it caused unstable level control. The overall pressure drop returned to normal at 1600 hours. Restrictions in the overall line to the overhead separator occurred again at 0010 and 0115 hours on October 2. These restrictions were cleared by turning the valve in the line. The water injection pump was again cut in from 0330 to 0345 hours, bringing the overall pressure drop down to normal.

Period 3A began at 0900 hours on October 2 with the charge pump still causing difficulties. The water injection pump was again used from 1130 to 1143 hours to bring down the overall pressure drop from 13 psi. From approximately 1800 hours the erratic pumping resulted in reduced charge rates. At 2200 hours the charge pump was lost for approximately ten minutes. When the pump was put back on line, a plug developed in the reactor inlet line and the charge pump pressure increased to 3110 psig. (This plug was attributed to the settling of solids from the low viscosity feed material.) Feed flow could not be reestablished. At 2249 hours the reactor plugged and the recycle and buffer pumps were lost. Unit shutdown was initiated.

After shutdown, all vessels, lines and pumps were inspected for deposits. A hard plug was found in the charge pump discharge mainfold. Very hard material was found in the reactor for two feet above the bubble cap. After drilling through this material the catalyst fell from the reactor. A very small amount of white, crystalline solid was found in the top of the bottoms separator and in the valve in the overhead line (which was the site of the restriction in both this run and Run 177-141). This material was sample and the analysis is given below:

Carbon, W %	0.70-1.15
Hydrogen, W %	7.68
Nitrogen, W %	25.75

RUN 177-143

Prior to this run the valve in the line to the overhead separator was removed. Separate cooling water lines on each charge pot were installed to allow more responsive charge pot temperature control.

Startup gas and oil flows were established at 1600 hours on October 5 and unit heatup began. At 0025 hours on October 6, a 200 psi reactor and overall pressure drop developed but quickly cleared. Internal recycle was started at 0130 hours. Period 1A began at 0900 hours when the coal feed (deep-cleaned Kentucky No.11, HRI 4018) was cut in.

At 0168 hours on October 7, the trim of the separator bottoms let-down valve dropped from the valve stem and unit back pressure fell quickly to 1700 psig. The reactor pressure drop increased momentarily to greater than 50 psi but returned to a normal value. Level control was switched to the other letdown valve and the unit pressure was back up to 2250 psig at approximately 0700 hours. During Period 1B the charge pump kicked out or stopped pumping several times.

Period 2A began at 0900 hours on October 7. The buffer pump was out of service from approximately 0100 to 0700 hours on October 8. An oil-like sludge was found in the buffer pot and buffer check assemblies. Charge pump difficulties continued during Period 2; there seemed to be some restrictions in the block valves in the charge pump manifold and reactor inlet line.

Period 3A began at 0900 hours on October 8. The buffer pump was out of service from approximately 1130 to 1200 hours for cleaning. The sludge was again found in the pump check assemblies and the pump suction line.

Period 4A began at 0900 hours on October 9. The charge pump stopped pumping at 1845 hours and feed flow was not regained for 45 minutes.

Period 5A began at 0900 hours on October 10. Unit operation was smooth during this period.

Period 6A began at 0900 hours on October 11. Again, unit operation was smooth all during the period.

Period 7A began at 0900 hours on October 12. The charge pump stopped pumping at 1925 hours. The duplicate charge pump was placed on line but could not be made to pump either. Feed flow was finally reestablished at 2040 hours. The problems with the charge pump contributed to an excessively high charge pot temperature. The charge pump kicked off frequently during this period.

Period 8A began at 0900 hours on October 13. The charge pump stopped pumping from 0433 to 0445 hours on October 14. The charge pump was again lost from 0510 to 0700 hours due to a plug in the bottom of the charge pot. Unit shutdown was initiated at 0930 hours on October 14, ending Period 8A.

The catalyst bed was found in a settled position and fell freely when the reactor was opened. White, crystalline deposits were found on the wall of the overhead separator and appeared to be similar to the material collected from the valve in the overhead line during Run 177-142. An analysis of the material is given below.

Carbon, W %	15.21
Hydrogen, W %	6.65
Sulfur, W %	0.074
Nitrogen, W %	17.38
Chlorine, W %	0.15

RUN 177-144

This run used Kentucky No.11, run-of-mine coal (HRI 4017). No additional unit modifications were made prior to this run. Unit startup began on October 17, 1978. Oil and gas flows were

established at 0450 hours and unit heatup began. Internal recycle was established at 1400 hours. Period 1A started at 2050 hours when coal feed was cut in.

Period 2A began at 0900 hours on October 18. At 1600 hours slurry feed was lost for about 15 minutes because the charge pump would kick off due to over-pressuring. It was necessary to switch to the duplicate charge pump temporarily while the charge pump was cleared by pumping to atmosphere with clean oil. The slurry feed was again lost at 1845 hours for about 15 minutes because the pump stopped pumping and it was necessary to bleed the checks. At 2230 hours slurry feed was again lost for about 15 minutes because the pump stopped pumping.

Period 3A began at 0900 hours on October 19. Operation was smooth during this period; there were no problems with the charge pump. At this point in the run insufficient vacuum overhead was being generated to meet recycle requirements. The hot separator temperature was lowered from 650 to 625°F in an attempt to increase vacuum overhead production.

Period 4A began at 0900 hours on October 20. During this period it was necessary to discontinue taking separator bottoms, filter liquid and vacuum overhead samples for PETC. This permitted greater separator bottoms charge to the vacuum still and increased vacuum overhead production.

Period 5A began at 0900 hours on October 21. At 2357 hours the charge pump was off for six minutes.

Period 6A began at 0900 hours on October 22. The buffer pump was out of service from approximately 1050 hours to 1355 hours. The buffer pump would not pump at unit pressure. Solids were found in the buffer pot and buffer pump checks. The buffer pot was cleared and the checks replaced. The charge pump kicked off and was out for about five minutes at 2356 hours.

Period 7A began at 0900 hours on October 23. At 2145 hours and at 2245 hours it was necessary to use vacuum still overhead from PDU Run 130-82 for recycle as no vacuum overhead from the run in progress was available. The internal recycle temperature began falling at about 50°F per hour. The recycle pump stroke was raised as needed to maintain a recycle temperature of 600°F.

Period 8A began at 0900 hours on October 24. At 0955 hours it was again necessary to use vacuum overhead from PDU Run 130-82 for the buffer pump charge. At 2100 hours a full set of PETC samples were taken. Unit shutdown was initiated at 0900 hours on October 25.

The catalyst bed was found in a settled position after shutdown. After adding anthracene oil on top of the catalyst bed and rodding lightly, the catalyst fell freely from the reactor. Yellow solids were found on the wall in the vapor space of the overhead separator. Approximately a week after sampling, the solids turned white. An analysis of this material is given below.

Carbon, W %	14.67
Hydrogen, W %	6.40
Sulfur, W %	0.66
Nitrogen, W %	0.30
Chlorine, W %	0.18

APPENDIX C

PETC SAMPLING PROGRAM

PETC SAMPLING PROGRAM

Samples of feed coal, liquid, slurry and solids products were collected for PETC during Runs 177-143 and 177-144. As PETC was interested in the trace element analysis of the samples, efforts were made to supply clean sample containers and to minimize the contact of samples with metal containers. Samples were taken in flint glass and polyethylene bottles washed in dilute nitric acid solution and rinsed in distilled water.

During Run 177-143, daily coal feed samples (approximately 50 grams) were taken and shipped to PETC as discrete daily samples rather than as a run composite. The vacuum bottoms samples were grab samples from a vacuum still run. However, the entire filter cake production and vacuum bottoms production from vacuum still and pressure filter runs in Period 6 were sampled and shipped to PETC.

By Period 3 of Run 177-144, too little vacuum overhead was being generated to meet the vacuum overhead recycle requirements. It was necessary to maintain the proper recycle rates to control the reactor slurry composition without the introduction of extraneous distillate oils. Accordingly, samples of separator bottoms, filter liquid and vacuum overhead was discontinued in Period 4. This permitted increased charges to the vacuum still and greater vacuum overhead production. However, separator bottoms, filter liquid and vacuum overhead samples were taken during Period 8, and the entire vacuum bottoms production and filter cake production were also sampled. As in Run 177-143, discrete daily coal samples were taken.

PETC samples taken in Runs 177-143 and 177-144 are shown in the following tables.

PETC SAMPLES TAKEN DURING RUN 177-143

Period	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>
Feed Coal, gms	73	72	68	65	72	69	68	63
Water, gms	100	100	100	100	100	100	100	100
Separator Overhead, gms	100	100	100	100	100	100	100	100
Separator Bottoms, gms	101	101	105	103	102	102	105	109
Filter Liquid, gms	100	100	101	100	100	100	101	100
Filter Cake, gms	-	-	-	-	-	231	-	-
Vacuum Overhead, gms	104	100	100	100	109	100	100	105
Vacuum Bottoms, gms	17	90	102	100	109	1181	92	100
Spent Catalyst, gms	100							

PETC SAMPLES TAKEN DURING RUN 177-144

Period	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>
Feed Coal, gms	50	54	51	50	51	51	51	51
Water, gms	100	100	100	100	100	100	100	100
Separator Overhead, gms	100	100	100	100	100	100	100	100
Separator Bottoms, gms	-	102	100	-	-	-	-	100
Filter Liquid, gms	-	100	100	-	-	-	-	100
Filter Cake, gms	-	-	-	-	-	-	-	575
Vacuum Overhead, gms	-	100	100	-	-	-	-	100
Vacuum Bottoms, gms	-	100	100	100	125	104	100	1014
Spent Catalyst, gms	100							

Miscellaneous

264 Grams HRI No.3830 catalyst, American Cyanamid HDS-1442A
 4 Sheets Whatman, Grade 42, Filter Paper
 2 Sheets Whatman, Grade 202, Reeve-Angel Filter Paper

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A P P E N D I X D

CALCULATION OF UNCONVERTED COAL AND BENZENE- INSOLUBLE RESIDUUM QUANTITIES FROM ANALYTICAL DATA

ANALYTIC INFORMATION

- A_V - Ash in vacuum bottoms, weight fraction
- I_V - Non-ash benzene-insolubles in vacuum bottoms, weight fraction
- Q - Vacuum bottoms, weight fraction of feed to still (filter)
-
- A_F - Ash in filter cake, weight fraction
- I_F - Non-ash benzene-insolubles in filter cake, weight fraction
- S_F - Soluble oil in filter cake (equals $1-A_F-I_F$)

DERIVED QUANTITIES

- F - Filter cake, weight fraction of feed to filter (still) forced ash basis equals $Q \times A_V/A_F$
- I - Weight fraction filterable benzene-insoluble in feed to still and filter
- U - Weight fraction unconverted coal in feed to still and filter
- A - Ash in feed to still (filter); equals $Q \times A_V$

$$I = A \times I_V - U$$

$$U = F \times I_F - F \times S_F \times \frac{I}{I-I-U-A} = F \times I_F - \frac{F \times S_F \times I}{1-Q(A_V+I_V)}$$

$$I = Q \times I_V - F \times I_F + \frac{F \times S_F \times I}{1-Q(I_V+A_V)}$$

$$I = \frac{Q \times I_V - F \times I_F}{1 - \frac{F \times S_F}{A_F(1-Q(I_V+A_V))}}$$

$$U = Q \times I_V - I$$

EXAMPLE	Analytical Data		Derived Data			
	Filter Cake	Vacuum Btms.	Filter (Still) Feed	Vacuum Btms.	Filter Cake	Filtered Liquid
Soluble Oil, %	32.02	72.98	88.60	72.98	32.02	95.13
Insoluble Oil, %	() (26.54)	() (16.86)	4.54	10.76	1.64	4.87
Unconverted Coal, %	()	()	.57	6.10	24.90	
Ash, %	41.44	10.16	4.28	10.16	41.44	
% of Feed		42.2		42.2	10.34	

APPENDIX E

CALCULATION OF NORMALIZED PRODUCT DISTRIBUTION

The normalized product distribution in these tables is calculated by the following procedure, which results in a total of normalized products equalling the amount of coal fed plus the amount of hydrogen consumed which is determined as part of the calculation. The calculation is iterative, and starts with calculating the hydrogen, nitrogen and sulfur contents of the collected products (excluding ammonia and hydrogen sulfide). The nitrogen deficiency of the collected products relative to the amount of nitrogen in the coal feed is used to calculate ammonia formation, and the sulfur deficiency is used to calculate hydrogen sulfide formation. The amount of hydrogen in the collected products plus that in the calculated ammonia and hydrogen sulfide formation less the amount of hydrogen in the coal fed is taken as the first estimate of hydrogen consumption. The amount of collected products plus ammonia and hydrogen sulfide (output total) is then compared with the amount of coal fed plus the hydrogen consumption calculated (input total). Since the two totals will probably not agree, a second calculation is carried out multiplying the collected product quantities by the ratio of coal plus hydrogen consumed divided by the total of the products. The adjusted, collected product quantities result in new values of ammonia, hydrogen sulfide, hydrogen consumption, output total and input total. The calculated input and output quantities are again compared and further iterations are carried out with new adjustment factors until they agree, which corresponds to the normalized totals sought. The adjusted product flows are then used with the product analyses to calculate the normalized product distribution, which is expressed on a dry coal basis.