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Direct Liquefaction Proof-Of-Concept Program

**Topical Report
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EXECUTIVE SUMMARY

The Bench Run PB-07 was the seventh of the nine runs planned in the POC Bench Option Contract between the U.S. DOE and Hydrocarbon Technologies, Inc. The primary goal of this bench run was to evaluate the effects of varying levels of iron and various promoters in a dispersed catalyst on hydroliquefaction of a high volatile bituminous Illinois No. 6 coal. The bench run PB-07 (HTI 227-102) was completed at the end of Period 20 (0400 HRS on 4/1/97). During the first 16 days of operation, a total of five run conditions were studied. Only four of these five conditions have been worked up for yields, because the original Condition 4, that employed the dispersed catalysts containing no iron, but only promoters, encountered plugging problems in the unit and resulted in a pre-mature unit shut-down. The last four days of this run were planned to produce the coal-derived heavy oil material, suitable for making carbon-based products, for West Virginia University, at the request of FETC. In general, the following observations were made during the run:

- Coal conversions were very high (96-97 % maf) and so were the resid conversions (87-89% maf); the C₄-524°C distillate yields were lower (67-68 % maf) than expected (72-75 % maf), from the high resid and coal conversion levels.
- With about 2500 ppm of iron, in the form of GelCat™, under the prevailing reaction conditions, neither promoter was found to have any significant impact on the overall process performance.
- Substantial hydrocracking was observed during the run, as exemplified by the abnormally high gas yields (15-16 % maf), high yields of light distillates, C₄-343°C (50-55 % maf), and low heavy VGO yields (2-3 % maf). The high gas yields were mainly due to high temperatures employed during the run. Lower reaction temperature or reduced fresh Fe catalyst loading may help minimize gas formation.
- In the absence of iron catalyst, with promoters only, the resid levels in the recycle solvent increased up to 50 w%, creating significant pumping problems and forcing a premature unit shut-down.
- During the last four days of operations, about 55 kg of the coal-derived O-6 bottoms material was produced for West Virginia University.

BACKGROUND, OBJECTIVE, AND SCOPE OF WORK

The POC Bench Option Project (PB-Series) is geared to evaluate different novel processing concepts in catalytic direct coal liquefaction and coprocessing of organic wastes such as plastics, heavy resids, waste oils, and ligno-cellulose wastes with coal. The new ideas being explored in this program include using novel dispersed slurry catalysts and combinations of dispersed and supported catalysts (hybrid mode), and coprocessing of coal with waste plastics, low quality resids, waste oils, and ligno-cellulosic wastes etc.

The primary objective of bench run PB-07 was to study the impact of dispersed catalyst composition and loading upon the direct liquefaction performance of a high volatile bituminous Illinois No. 6 coal.

The run was carried out for 20 operating days (including the four days used for the production of O-6 bottoms material for West Virginia University), spanning over five process conditions.

SYSTEM CONFIGURATION

Bench Run PB-07 involved two equal volume back-mixed reactors with internal recirculation. The HTI Unit 227 was used in this run. No pretreater/preheater was used. A small mixing/charge pot was employed for the feed (two-hour feed batches). Interstage V/L separator was employed along with an in-line fixed-bed hydrotreater. Coal, catalyst, and recycle oil were introduced through the feed system. After separating out the process water, the oil fraction (SOH) from first stage reactor and flashed product from the hot separator bottoms were re-pressurized and combined with the second stage hot separator (O-1) overheads for feeding to the in-line hydrotreater. A slurry stream was removed from the bottom of the flash vessel (O-6) bottoms. An off-line pressure filtration unit was used to recover a solid-free liquid (recycle solvent) from the slurry stream.

FEED MATERIALS AND SPECIAL SAMPLES

Coal Feed

Illinois No. 6 coal (HTI 6157), which was employed during Run PB-05 (227-97), was used for Run PB-07 (227-102).

Start-up and Make-up Oil

L-814

Catalysts

HTU:	Criterion C-411 Trilobe (HRI-6135)
K-1:	HTI's GelCat™ containing Fe and promoters

Interstage (Reactor K-1) Slurry Samples

Five interstage (reactor K-1) slurry samples were planned, one for each of the Run Conditions. These samples were taken immediately after the completion of each of the indicated Work-up Periods.

Special Samples for Consol

Samples of different process streams were collected during Work-up Periods for Consol, Inc., as per the following plan:

COLLECTION PERIODS - 4, 7, 10, 13, and 16

<u>SAMPLE STREAM</u>	<u>AMOUNT, gram</u>
SOH-1st Stage	250
SOH-2nd Stage	250
Pressure Filter Liquid (PFL)	350
Pressure Filter Cake (PFC)	350
O-6 BOTTOMS	350
FEED SLURRY	250
INTERSTAGE SAMPLE	250

Special Samples for West Virginia University

Upon DOE/FETC's request, The bench run PB-07 was extended for four operating periods (17 through 20) to produce O-6 bottoms material for West Virginia University. Approximately 55 kg of the required material was generated during the extension of the run and was shipped to West Virginia University for producing special carbon materials.

OPERATIONAL DETAILS

Unit Configuration

For Run PB-07, the bench scale unit 227 was set up to process bituminous coal using two 2,000 ml reactors with interstage separation. Both stages contained no supported catalyst, but slurry catalysts added with the feed. Hydrogen sulfide, fed to the second stage, provided sulfur to maintain activity of the slurry catalyst and the supported catalyst in a fixed-bed in-line hydrotreater.

Figure 1, Drawing 227SF041, is a simplified flow diagram of Unit 227. The prepared feed blend was charged to the feed tank (P-2) and pumped to the first-stage reactor (K-1). The effluent from K-1 was flashed in the interstage hot separator (O-1A), removing gases and 343°C- distillate before the liquid phase was fed to the second-stage reactor (K-2). The products from K-2 were separated in a hot separator (O-1). The O-1 bottoms stream was depressurized, sent to the atmospheric flash vessel (O-3), and the atmospheric bottoms stream was withdrawn from the bottoms receiver (O-6). Overheads from O-1 and O-1A flowed through the in-line hydrotreater (K-3), and unhydrotreated knockouts were also collected and fed to the hydrotreater. The hydrotreated liquids and water were separated from the gases in the high-pressure cold separator (O-2). Part of O-6 bottoms stream was recycled directly and the rest was pressure filtered (PF) to remove the solids and to produce PF liquid for recycle and for the internal-recycle purges (buffers).

Run Conditions

Run PB-07 included five conditions, spanning 16 operating periods as shown in Table 1. The only variable was the type and loading of slurry catalysts. The K-1 and K-2 temperatures were held constant at 449 °C and 460 °C, respectively. The Illinois No. 6 coal (Table 2) feed rate was maintained at 1360 g/hr, except for the extension period (periods 17-20) of the run for the production of O-6 bottoms material.

Startup

Startup consisted of establishing the proper flows of oil and gases, presulfiding the catalyst in the hydrotreater, setting vessel temperatures, and increasing the reactor temperatures to 413 °C. Filtered L-814, a heavy gas oil, was used as a startup oil for this run (Table 3). The O-6 bottoms was recycled for feed when it became available.

The startup proceeded smoothly.

Condition 1 (Periods 1 through 4)

Period 1 started with the introduction of coal feed at 0400 hours March 10, 1997 (Each 24 hour period started and ended at 0400 hours). The coal feed rate and reactor

temperatures were gradually increased, reaching full coal rate at 1600 hours and run temperatures of 449 °C and 460 °C at 0100 hours. The slurry catalyst was HTI GelCat™ (L-873).

Charge pump stoppages occurred in each period of Condition 1. Usually solids had to be cleared from the suction line and the suction check valves had to be replaced. In the process of flushing the charge pump check valves in Period 4, the L-814 wash oil was accidentally transferred from P-1 to P-2.

There were some problems with the O-2 level control, requiring brief manual valve operation. In Period 1 the O-1A level could not be lowered until the back pressure was decreased to 2400 psig. An automatic valve "stroker" on the O-1A level controller helped maintain a consistent level, but a failed solenoid valve on the "stroker" had to be replaced in Period 3. The O-3 control valve and discharge had to be blown clear in Period 2. In Period 2 a seal ruptured on the O-2A water pump, causing a large loss of water, but no pressure disruption.

The pressure drop across the first stage system was slowly increasing during Period 2, apparently due to a restriction in the O-1A overhead line, which disappeared after the water injection rate was increased from 250 to 300 g/hr. In Period 4 the H₂S feed rate was corrected from 66 g/hr to the specified 40 g/hr. A 419 g first stage slurry sample was successfully obtained at the end of Period 4.

Condition 2 (Periods 5 through 7)

In Condition 2 the slurry catalyst was changed to HTI Fe GelCat™ with 0.4 W% promoter (L-921). Occasional charge pump stoppages continued. At 0000 hours Period 6 the circulator pump stopped and could not be restarted. All the circulating lines were disassembled and cleaned and the circulator pump was replaced. The coal was out for 7 hours and the reactor temperatures were lowered to 399°C. The coal feed rate was gradually increased, reaching the full coal rate at 1600 hours during Period 7. The reactor temperatures were brought back to 449 and 460 °C, respectively by 2100 hours. Also in Condition 2, the K-2 right-side buffer pump check became clogged and had to be replaced.

Condition 3 (Periods 8 through 10)

Starting at 1000 hours during Period 8, the feed included 0.45 g/hr of the second promoter in addition to the L-921. Occasionally, there were brief losses of feed, usually due to plugged checks. On three occasions (Periods 8 and 9) the outage was one or two hours, and L-814 oil was charged for reactor feed.

In Period 10 there was difficulty maintaining a steady level in O-1A. Twice, the O-1A level could only be lowered by decreasing the back pressure. Also, the K-2 right-side internal-recycle check and surge line had to be flushed because of restrictions. A 515 grams first stage slurry sample was successfully obtained at the end of Period 10.

Condition 4 (Periods 11 through 12)

In Condition 4 the 33 g/hr of HTI GelCat™ Fe catalyst was discontinued. Only 0.45 g/hr of promoter 1 and 0.50 g/hr of promoter 2 were charged. The slurry in P-2 started getting very viscous because of the heavier bottoms recycle in this condition. There were numerous pumping problems associated with the viscous slurry. The feed system temperature was increased to 163 °C, but the pumping problems continued, and P-2 erupted from sudden vaporization. At 1330 hours Period 12, the reactor temperatures were lowered to 399 °C while the feed system was flushed. This wash was continued until 0400 hours when a new condition was started.

Condition 5 (Periods 13 through 16)

HTI GelCat Fe catalyst without promoters were again added to the feed slurry. The coal feed rate was gradually increased, but a restriction developed in the feed line at 0830 hours. The reactor temperatures were lowered to 399 °C, and the unit was washed using the sample-system flush pump. After an 8-hour wash, the reactor was cooled. A plug of hard, dry solids was found in the feed line near the junction with the internal recycle.

Liquid feed was resumed at 1445 hours March 23, and coal feed was resumed at 0400 hours March 24, starting another Period (13). Promoter 1 was discontinued at 1300 hours, leaving the L-897 at 33 g/hr as the only catalyst added. The specified reactor temperatures of 449 and 460 °C were reached at 2400 hours. The K-2 internal recycle was lost at the end of Period 13, causing the reactor temperature to increase to 471 °C. When buffer oil was charged through the pulse lines, the recycle recovered and the K-2 temperature returned to 460 °C.

In Period 14 the K-2 internal recycle was lost again. At 1400 hours the spread between the 85"- and 42"-down thermocouples had increased to 22°C. As soon as the internal recycle rate was increased from 60/60% to 75/75% in an attempt to make the temperature distribution more uniform, the recycle was lost. With lower flow, the reactor temperature increased to 481 °C. The K-2 heaters were turned off and the buffer pumps were turned on to cool the reactor. The recycle was not regained until 1640 hours when the pressure was lowered in the pulse line. To avoid losing the recycle again, the reactor temperature was slowly returned to the specified levels. By 2100 hours the K-2 temperature had lined out at 460 °C. The feed became somewhat more viscous in Period 15, and there were about six feed stoppages. The feed circulation was also lost for about 30 minutes, requiring the addition of PFL to the slurry and flushing with L-814 oil. The H₂S feed line to K-2 became plugged at the end of Period 15. It was rerouted to K-1. At 1100 hours Period 16 the H₂S was discontinued and TNPS was charged to K-2 with the buffer oil at 108 g/hr.

The feed was steady through Period 16, except for an approximately 20 % rate decrease followed by a 10% overcorrection in the last two hours. A 405 g first stage sample was successfully obtained at the end of Period 16.

Run 227-102 Extension

Unit Configuration and Run Conditions

The run was extended by four days (designated Run 227-102E) to produce 55 kg of O-6 bottoms. The unit configuration was changed by bypassing the hydrotreater, discontinuing the pressure filters, and recycling separator overheads (O-2A and O-2 bottoms).

227-102E (Periods 17 through 20)

The reactor temperatures and feed rates were lowered starting at 0400 hours Period 17 (March 17, 1997). The specified 399 °C reactor temperatures and 680 g/hr coal feed rate were reached by 1400 hours. There were brief feed pump stoppages in Periods 17, 19, and 20. In Period 17 approximately three hours of feed slurry were lost through the circulating pump packing.

Shutdown and Inspection

With the reactors still at 399 °C, two 6-hour batches of O-6 bottoms were collected after the coal feed was removed. During the shutdown occasional feed pumping problems continued as the solids in the circulation system were flushed out.

The reactor internals were clear and easily removed, but there was a moderate amount of scale on the reactor walls. The downstream vessels were generally clean. There were three hard-to-explain 1/16"-diameter erosion holes above the recycle cup in the otherwise-unmarked internal-recycle tube.

On-Line Time Summary

The chronology of the operations for Run 227-102 is listed in Appendix along with a summary of the total time in startup, run, shutdown, and down modes. Unit 227 operated for 587 hours with one interruption.

RUN 227-102 OPERATIONS-STATUS-CLASSIFICATION CHRONOLOGY

Operations Classification	<u>Start</u>		<u>End</u>		<u>Duration</u>
<u>I.D.</u>	<u>Time</u>	<u>Date</u>	<u>Time</u>	<u>Date</u>	<u>Hours</u>
S/U	1200	3/8/97	0400	3/10	40
1-12	0400	3/10	1300	3/21	273
Wash	1300	3/21	0400	3/22	15
13	0400	3/22	0800	3/22	4
S/D	0800	3/22	0400	3/23	20
D/T	0400	3/23	1500	3/23	11
S/U	1500	3/23	0400	3/24	13
13-16	0400	3/24	0400	3/28	96
17-20	0400	3/28	0400	4/1	96
S/D	0400	4/1	1000	4/2	30

OPERATING CONDITIONS AND MATERIAL BALANCES

The bench run PB-07 proceeded with interruptions in Condition 4, which employed a combination of two promoters, with no iron. The unit plugged up during the Condition 4, as the percentage of residuum material in the recycle solvent increased to about 50 w%. The operating summary and the process performance of individual periods during PB-07 are shown in *Table 4*. *Figure 2* shows the operating conditions during PB-07 in terms of feed space velocity and reactor temperature. The reactor temperatures during Conditions 1 through 5 were uniform, but the space velocity encountered operational problems or difficulties. An average material recovery balance (the daily material balance summary is attached in the Appendix) of about 98 W% was obtained (*Figure 3*) for the entire Bench Run PB-07. The loadings of dispersed slurry catalysts, that were changed during the course of the run are listed in *Table 4*. The material balances were calculated using a PC-based spreadsheet, while the yield calculations (normalization) were conducted using the VAX-routines.

PROCESS PERFORMANCE RESULTS

The process performance, conversions, and product yields and quality for Run PB-07 are addressed in this section. The calculation of coal conversions, normalized product yields, and other process performance-related indicators were carried out using programs written in HTI's VAX database. The overall process performance during Run PB-07 is summarized in *Table 4*, depicted in *Figures 4 through 10* and discussed in details in the following sections.

Total Coal and 524°C+ Residuum Conversion

Typical combined feed conversions (based on the solubility of pressure filter solids in quinoline and expressed on an SO₃-free basis), obtained during equilibrated Periods of different Conditions of PB-07 are shown in *Figure 4*. The coal conversions (W% maf) were about 96 % throughout the course of the Run. The conversion of 524°C+ residuum material varied between 86 to 89 W% maf. The highest resid conversion was obtained during the last condition of the run when only an iron containing dispersed catalyst was employed.

C₄-524°C Distillate and 524°C+ Residuum Yields

Distillate yields and 524°C+ residuum yields (including the unconverted resid material in the feed as well), are shown in *Figure 5*. The distillate yields varied between about 67 and 71 W% maf coal. The residuum yield varied between about 9 to 13 W% maf coal. Neither distillate yield nor residuum yield was very sensitive to the catalyst loading/type as long as iron-containing GelCat™ was employed.

Distillate Fraction Yield/selectivity

The yields of distillate fractions such as naphtha (IBP-177°C), middle distillate (177-343°C), and heavy distillate (343°C+) are shown in *Figure 6*. The numbers plotted in this Figure, converted to selectivity basis, are shown in *Figure 7*. The yields of light fractions, namely naphtha and mid-distillate, were high under all of the run conditions.

Hydrogen Consumption and Light Gas (C₁-C₃) Yield

Hydrogen consumption (*Figure 8*), based upon maf feed, varied between about 7 to 8.5 W%. The light gas yields varied between about 12-15 W% maf coal, which were abnormally high, and seemed to be the effect of a combination of the high reaction severity (high reactor temperatures) and the iron-promoter combination in the dispersed catalyst.

Hydrogen Utilization

The hydrogen utilization during coal conversion is characterized by two indicators: hydrogen efficiency and C_1 - C_3 gas selectivity. The former is defined as the amount of C_4 - 524°C distillates obtained per unit weight of hydrogen consumed while the latter is the amount of light hydrocarbon gases produced per unit weight of distillates. As shown in *Figure 9*, the hydrogen efficiency varied between about 8 and 9 kg distillates per kg H_2 . The light gas selectivity was between 17 and 26 %. Both of these numbers were not good from the standpoint of hydrogen utilization, due primarily to high gas formation and hydrogen consumption in Run PB-07.

PRODUCT QUALITY

Different product fractions (product gases, first and second stage SOH products, PFL, and PFC) from the Work-up Periods 3,4,7,8,10, and 16 were analyzed in detail for their composition. These analyses for different product fractions are listed in Tables 5 through 7.

Separator Overhead Product (SOH)

The properties of SOH oil (the second stage separator) for the Work-up Periods are shown in *Table 5*. The API gravities of the SOH product were high all across (33-38 °API), with over 1.7 atomic H/C ratio. The second stage SOH oils had a typical boiling range of 50-370°C. The nitrogen and sulfur heteroatom contents were typically low during Run PB-07, due to in-line fixed-bed hydrotreating. The quality of second stage SOH process distillate, in terms of heteroatoms content and H/C ratios, is depicted in *Figures 10 and 11*.

Pressure Filter Liquid (PFL) and Pressure Filter Cake (PFC)

The flash-vessel separator bottoms (O-6) go through pressure filtrations for separation of solids from heavy liquid product and the recycle oil. The liquid oil, called pressure filter liquid (PFL), is usually higher than 343°C in boiling point and contains the unreacted heavy residuum material to a varying degree. The residuum content of the PFL varied between 33 and 37 w%; the H/C ratio (as shown in *Figure 11*) varied between about 0.92 and 1.05. The properties of the PFL did not vary substantially with changes in the reaction conditions. Similar was the case with pressure filter cake. Based upon the PFC analysis, coal conversion values of above 96 W% maf were obtained.

TECHNO-ECONOMIC ASSESSMENT

Technical assessment on the results from Run PB-7 has been completed. This run used Illinois #6 coal in an all-dispersed catalyst mode direct liquefaction operation. Interstage separation and on-line hydrotreating were used throughout the run.

Coal analysis was the same as in Run PB-5. Four periods from the run were analyzed, and the operating conditions and performances for the four periods are summarized in Table 8. Reactor conditions were substantially constant throughout the run, and were nearly the same as in the coal-only portion of Run PB-5. The major difference between the four conditions in Run PB-7 was the dispersed catalyst levels, as shown in Table 8. The Iron levels were 2-1/2 times higher than previously used in Run PB-5. Comparison with Run PB-5 shows that $C_1 - C_3$ gas yields were twice as high in this run, at the expense of distillate yields. Hydrogen consumption was over 1/3 higher in this run than that in Run PB-5.

Material balances are summarized in Table 9, showing a 4 percent reduction, compared with PB-5, in liquid yield, expressed in barrels of product per ton of feed. As expected, LPG production substantially exceeded that in Run PB-5. Hydrogen balances, utilities requirements and thermal efficiencies are listed in Table 10. As shown, Hydrogen consumption is 20 percent higher per barrel of product than in Run PB-5. Capacities of process units and off-site are listed in Table 11.

Liquefaction plant investment details are summarized in Table 12. Costs are about 10 percent higher than in Run PB-5, primarily due to increases in compressors and high-pressure gas-handling equipment. Overall plant costs are listed in Table 13. These costs are again substantially higher than in Run PB-5, especially in gas treating.

Overall economics are summarized in Table 14, showing more than a 20 percent increase in equivalent crude oil price over Run PB-5. Table 15 gives the breakdown by category for the equivalent crude oil price.

In conclusion, the increased iron catalyst usage and high reaction temperature had a rather severe adverse economic impact, owing to the decreased distillate yields and to the increases in both the gas yield and the hydrogen consumption.

CONCLUSIONS

The following conclusions can be drawn based upon the data obtained from Bench Run PB-7:

- In the catalyst concentration range employed during the bench run PB-7, the overall process performance was not significantly affected by variations in the catalyst type and/or loadings; in the case where only promoters were employed without GelCat, the residuum in the recycle solvent increased to an extent that the continuous unit operations became difficult.
- Coal conversions were very high (96-97 % maf) and so were the resid conversions (87-89% maf); the C₄-975°F distillate yields were lower (67-68 % maf) than expected (72-75 % maf), based upon the high resid and coal conversion levels.
- With about 2500 ppm iron, employed as GelCat™, and under the prevailing reaction conditions, neither of the promoters at 100 ppm level were found to have any significant impact on the overall process performance.
- Substantial hydrocracking was observed during this run, as exemplified by the abnormally high gas yields (15-16 % maf), high yields of light distillates, C₄-343°C (50-55 % maf), and low heavy VGO yields (2-3 % maf). This is probably due to the high reaction temperatures and/or relatively high Fe concentration. It is recommended that in the future runs, both K1 and K2 temperatures should not exceed 450°C, especially when promoter 1 is present. Fe catalyst loading should be maintained in the range of 1000-1500 ppm if Illinois No. 6 coal is used in liquefaction.

Table 1. Run Plan for Bench Run PB-07*

Condition	1	2	3	4	5	Extension
Period Number	1-4	5-7	8-10	11-12	13-16	17-20
Work-Up Period	3/4	7/8	10	n/a	16	
Space Velocity, mf fresh feed / reactor volume						
K-1 (kg/h/m ³)	640	640	640	640	640	**
Reactor Temperatures, °C						
K-1	449	449	449	449	449	
K-2	460	460	460	460	460	
Catalyst Loading, ppm						
Iron	2500	2500	2500	0	2500	
Promoter 1	250	100	100	100	0	
Promoter 2	0	33	100	100	0	
Recycle to Feed Ratio	1.0	1.0	1.0	1.0	1.0	

*Condition 4 could not be completed as planned, due to the plugging problems in the unit. A four-day long extension of PB-07 was also carried out to produce approximately 120 pounds of the coal-derived O-6 bottoms material for West Virginia University.

**Production of O-6 bottoms.

Table 2. Analysis of Illinois No. 6 Feed Coal

HTI Designation	HTI-6157
Moisture Content, W%	3.39
Proximate Analysis, W% Dry	
Volatile Matter	49.86
Fixed Carbon	57.95
Mineral Matter	10.03
Ultimate Analysis, W% Dry	
Carbon	71.20
Hydrogen	5.25
Nitrogen	1.42
Sulfur	4.02
Ash	10.24
Oxygen (Diff.)	7.87
H/C Atomic Ratio	0.89

Table 3. Analysis of Start-up/Make-up Oil

HTI Designation	Filtered L-814
API Gravity, °	0.40
ASTM D-1160 Distillation, °C	
IBP	309
5 V%	351
10 V%	374
20 V%	394
30 V%	409
40 V%	426
50 V%	437
60 V%	449
70 V%	467
80 V%	507
84 V%	524
Weight Percents	
IBP-343°C	5.47
343-454°C	53.99
454-524°C	22.18
524°C ⁺	18.36
Elemental Analysis, W%	
Carbon	88.96
Hydrogen	8.25
Sulfur	2.22
Nitrogen	0.19
NMR Data	
W% Aromatic Carbon	88.03
W% Cyclic Hydrogen	44.36

Table 4. Process Performance Results

Condition	1	2	3	4	5
Catalyst Loading, ppm					
Iron	2500	2500	2500	0	2500
Promoter 1	250	100	100	100	0
Promoter 2	0	33	100	100	0
<i>Mass Balance Period</i>	3	7/8	10	n/a	16
Material Recovery, %	99.3	98.8	94.9	n/a	104.7
Normalized Yields, % MF Coal					
C ₁ -C ₃	14.6	11.9	15.1		16.2
C ₄ -C ₇	7.0	8.0	5.9		12.3
IBP-177°C	12.7	11.7	12.8		14.1
177-260°C	16.1	12.2	13.5		12.6
260-343°C	13.5	15.7	13.9		11.6
343-454°C	10.2	13.4	11.2		8.3
454-524°C	3.0	3.3	3.3		2.7
524°C+	8.4	9.7	8.1		6.9
Unconverted Coal	3.3	3.0	3.2		3.2
H ₂ O	3.3	3.6	4.3		4.1
CO _x	1.1	1.3	1.5		1.7
NH ₃	1.3	1.4	1.4		1.4
H ₂ S	3.0	3.1	3.0		3.0
Process Performance, % MAF Coal					
Coal Conversion	96.3	97.0	96.3		96.4
Residuum Conversion	87.1	86.0	87.5		88.8
C ₄ -524°C Yield	69.0	71.1	67.0		68.1
H ₂ Consumption	7.9	8.6	7.5		8.3
H ₂ Efficiency, kg dist//kg H ₂	8.7	8.3	8.9		8.2
C ₁ -C ₃ Gas Selectivity*	23.5	18.5	24.9		26.3

*Defined as a percentage of C₁-C₃ light gas yield relative to the C₄-524°C distillate yield.

Table 5. Properties of Second Stage Separator Overhead (SOH)

Condition	1	2	3	5
Period	4	7	10	16
Gravity, ° API	35.6	34.7	33.0	37.8
IBP, ° C	78	50	72	51
FBP, ° C	370	378	363	358
Elemental Analysis				
Carbon, W%	86.33	86.75	87.48	86.66
Hydrogen, W%	12.90	13.05	12.52	13.12
Sulfur (Antek), ppm	118.8	136.5	145.9	67.0
Nitrogen (Antek), ppm	6.0	8.2	23.0	1.0
H/C Ratio	1.79	1.81	1.72	1.82
ASTM D-86 Distillation, Composition, W%				
IBP-177°C	31.38	29.41	31.53	37.5
177-260°C	36.73	28.54	31.74	31.8
260-343°C	27.40	33.83	28.95	26.3
343°C+	4.49	8.22	7.79	4.4

Table 6. Properties of Second Stage Pressure Filter Liquid (PFL)

Condition	1	2	3	5
Period	4	7	9/10	16
Gravity, °API	-6.8	-4.4	-7.6	-8.2
IBP, °C	230	253	245	245
Elemental Analysis, W%				
Carbon	89.56	89.09	88.87	89.17
Hydrogen	6.83	7.67	6.97	7.03
Sulfur	1.23	0.84	0.73	0.70
Nitrogen	0.63	1.00	1.26	1.40
H/C Ratio	0.92	1.03	0.94	0.95
ASTM D-1160 Distillation, Composition, W%				
IBP-343°C	15.8	12.3	12.6	13.1
343-454°C	36.4	41.6	36.2	35.5
454-524°C	13.0	13.5	14.9	14.3
524°C+	34.8	32.6	36.3	37.1
CCR, W%	22.6	20.5	21.6	23.9
Cyclohexane Insolubles	26.5	25.8	25.6	22.6
Toluene Insolubles	6.44	6.25	5.19	6.94

Table 7. Properties of Second Stage Pressure Filter Cake (PFC)

Condition	1	2	3	5
Period	4	7	10	16

Elemental Analysis, W%

Carbon	43.7	50.1	42.1	42.1
Hydrogen	2.9	3.6	2.8	2.9
Sulfur	5.1	4.7	5.0	5.7
Nitrogen	0.6	0.6	0.7	0.7

H/C Ratio	0.80	0.87	0.81	0.81
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Composition, W%

Quinoline Insolubles	62.2	54.4	61.0	62.0
Ash (in Quinoline Filt.)	48.6	40.6	47.6	48.4
ASTM Ash	48.4	41.1	48.3	48.6

Table 8 Operating Conditions & Process Performance

	Period 3	Period 8	Period 10	Period 16	PB-5 Period 9
Operating Conditions	1	2	3	5	1
Space Velocity, kg/h/m ³	640	608	630	609	677
Temperature, ° C					
Stage 1	446	446	446	446	447
Stage 2	456	458	457	451	458
Recycle oil					
Rate, kg/kg MF feed	0.92	0.93	0.91	0.89	1.00
Resid, W%	36.5	39.7	36.3	37.1	32.6
Solids, W%	0.0	0.0	0.0	0.0	7.3
Catalyst rate, ppm MF feed					
Promoter 1	0	33	100	0	0
Promoter 2	250	100	100	0	100
Iron	2500	2500	2500	2500	1000
Process Performance					
Resid Conversion, W% MAF	87.1	86.0	87.5	88.8	85.1
Coal Conversion, W% MAF	96.4	96.6	96.4	96.5	96.2
Process Yields, W% MF feed					
H ₂ S	3.00	3.09	2.98	2.98	3.02
NH ₃	1.34	1.36	1.42	1.34	1.22
H ₂ O	3.28	3.54	4.27	4.01	3.87
CO + CO ₂	1.09	1.24	1.48	1.68	1.32
C ₁ - C ₃	14.52	11.78	15.02	16.07	6.11
C ₄ - 177°C	19.50	19.57	18.55	26.20	16.69
177 - 343°C	29.34	27.71	27.20	24.10	29.27
343 - 454°C	10.14	13.32	11.11	8.21	15.53
454 - 524°C	2.99	3.26	3.29	2.63	5.06
524+ °C	8.35	9.59	8.00	6.85	9.95
Unconverted feed	3.26	3.01	3.19	3.18	3.43
Ash	10.24	10.24	10.24	10.24	10.24
Total	107.05	107.71	106.75	107.49	105.71
C ₄ -524°C	61.97	63.86	60.15	61.14	66.56

Table 9 Economic Analysis-Material Balance

	Period 3	Period 8	Period 10	Period 16	PB - 5 Period 9
Feed to Liquefaction, T/D					
Coal	12,000	12,000	12,000	12,000	12,000
Liquid Products, B/D					
Gasoline	13,860	14,051	13,399	13,568	14,819
Diesel Fuel	32,747	33,200	31,659	32,059	35,013
Total	46,607	47,251	45,058	45,627	49,832
Barrels of Products/Ton Feed	3.88	3.94	3.75	3.80	4.15
By-products					
Propane, B/D	6,442	5,492	6,526	7,161	2,526
Butane, B/D	3,064	2,974	3,100	3,596	1,757
Sulfur, T/D	358	362	362	362	402
Ammonia, T/D	161	163	171	161	146
Waste to Disposal, T/D	1,241	1,243	1,242	1,240	1,245

**Table 10 Hydrogen Balance, Utilities Production
& Thermal Efficiency**

	Period 3	Period 8	Period 10	Period 16	PB - 5 Period 9
Hydrogen Balance					
Hydrogen Consumption, MMSCFD					
Liquefaction	319.2	349.2	305.2	339.3	258.4
Product Upgrading	12.1	-29.0	24.1	0.1	26.0
Solution & Purge Loss	15.4	13.8	15.7	16.8	9.3
Total	346.7	334.0	345.0	356.2	293.7
H ₂ consumed, SCF/Bbl product	7,440	7,070	7,660	7,810	5,890
Hydrogen production, MMSCFD					
via Partial Oxidation	106.7	124.9	112.7	92.6	150.5
via Steam Reforming	240.0	209.1	232.3	263.6	143.2
Total	346.7	334.0	345.0	356.2	293.7
Utilities Produced or Purchased					
Power, MW	252	253	253	260	215
Steam (600 Psig), 1000 lb./hr	794	673	817	875	388
Cooling water, 1000 GPM	288	261	293	306	195
Purchased Natural Gas, MMMBTU/D	132.7	126.9	129.1	139.0	108.8
Raw water, 1000 Gal/D	13,671	12,479	14,084	14,399	9,411
Thermal Efficiency					
Inputs, MMMBTU/D					
Feed	319.0	319.0	319.0	319.0	319.0
Natural Gas	147.2	140.7	143.2	154.2	120.7
Total	466.2	459.7	462.2	473.2	439.7
Outputs, MMMBTU/D					
Gasoline	75.9	77.0	73.4	74.3	81.2
Diesel	190.1	192.8	183.8	186.1	203.4
Propane	24.8	21.1	25.1	27.5	9.7
Butane	13.4	13.0	13.5	15.7	7.7
Sulfur & Ammonia	6.3	6.4	6.5	6.3	6.4
Total	310.5	310.3	302.3	309.9	308.4
Thermal Efficiency (HHV), %	66.6	67.5	65.4	65.5	70.1

Table 11 Capacities of Process Units & Offsites

							PB - 5
Area	Description	Sizing Basis	Period 3	Period 8	Period 10	Period 16	Period 9
100	Feed Preparation						
	Coal	T/D coal feed	12,000	12,000	12,000	12,000	12,000
200	Liquefaction	T/D total feed	12,000	12,000	12,000	12,000	12,000
300	H₂ Manufacture						
	Steam Reforming	MMSCFD H ₂	240	209	232	264	143
	Partial Oxidation	MMSCFD H ₂	107	125	113	93	151
400	Oxygen Plant	T/D Oxygen	1,323	1,546	1,398	1,148	1,865
500	Product Treating						
	Sour water treating	gpm water	3,113	3,132	3,134	3,126	2,939
	Sulfur recovery	T/D Sulfur	358	362	362	362	402
	Gas plant	lb/hr C ₁ -C ₃	154,240	117,830	150,200	160,740	61,140
600	Upgrading						
	Catalytic Reforming	B/D gasoline	13,860	14,051	13,399	13,568	14,819
	Hydrotreating	B/D liquids	32,747	33,200	31,659	32,059	35,013
700	Utilities						
	Steam generation	M lb/hr	796	674	818	877	388
	Power generation	MW	252	253	253	260	315
	Cooling water	M GPM	288	261	293	306	195
800	Tankage						
	Product Liquids	B/D liquids	46,607	47,251	45,058	45,627	49,832
	Propane & Butane	B/D	9,506	8,466	9,626	10,757	4,283
	Solids handling	T/D	1,241	1,243	1,242	1,240	1,245
900	General Offsites	T/D total feed	12,000	12,000	12,000	12,000	12,000

Table 12 Liquefaction Plant Investment Details

	Period 3	Period 8	Period 10	Period 16	PB - 5 Period 9
Major Equipment Cost, \$M					
Pumps	23,525	23,820	23,500	23,270	21,361
Reactors & Hydrotreater	46,535	47,680	46,535	47,680	39,614
Fired Heaters	13,932	13,943	14,076	13,937	12,709
Exchangers	21,982	21,914	22,062	22,492	19,267
Drums	35,156	34,796	35,425	37,326	29,310
Towers	9,491	9,425	9,526	9,847	7,843
Compressors	38,312	39,401	37,736	39,950	29,414
HPU	22,737	20,843	22,963	24,222	14,215
Total	211,670	211,822	211,823	218,724	173,733
 <u>Plant Investment, \$MM</u>					
Materials & Equipment	383.5	383.7	383.7	396.2	314.8
Labor & Subcontracts	165.9	166.1	166.1	171.5	136.2
Indirects	138.1	138.2	138.2	142.7	113.3
Total Investment, \$MM	687.5	688.0	688.0	710.4	564.3

Table 13 Total Plant Investment Summary

(Plant Investment in \$MM, 1994 US Gulf Coast basis)

Plant Section	Period 3	Period 8	<u>Period 10</u>	Period 16	PB - 5
					Period 9
Coal Preparation	57.6	57.6	57.6	57.6	57.6
Oil Storage & Handling	0.0	0.0	0.0	0.0	0.0
Liquefaction	687.5	688.0	688.0	710.4	564.3
Hydrogen Manufacture	301.3	294.0	300.6	305.6	266.6
Oxygen Plant	57.2	63.7	59.4	51.7	72.7
Treating	424.8	376.9	433.6	451.2	265.4
Product Upgrading	114.8	116.9	112.8	111.9	120.7
Utilities	524.7	476.8	533.4	556.4	345.6
Tankage, Waste Handling	155.6	157.3	151.7	153.0	163.8
General Offsites	211.0	211.0	211.0	211.0	211.0
Subtotal	2,534.5	2,442.2	2,548.1	2,608.8	2,067.7
Fee, Contingency	506.3	487.9	509.1	521.4	413.0
Total Plant Investment	3,040.8	2,930.1	3,057.2	3,130.2	2,480.7
\$/BPD of Product	65,240	62,010	67,850	68,600	49,780

Table 14 Product Cost Calculation

	Period 3	Period 8	Period 10	Period 16	PB - 5 Period 9
Operating Costs, \$MM/year					
Coal, as received (\$20.50/T)	89.79	89.79	89.79	89.79	89.79
Natural gas (\$2.00/MMBTU)	87.21	83.39	84.83	91.30	71.45
River Water (\$0.10/Mgal)	0.45	0.41	0.46	0.47	0.31
Catalysts & Chemicals	19.27	17.31	18.96	20.69	12.96
Dispersed Catalyst	15.87	16.59	18.17	15.77	6.35
Ash disposal(\$5.00/T)	2.04	2.04	2.04	2.04	2.05
Labor	22.75	22.75	22.75	22.75	22.99
Maintenance	20.38	20.38	20.38	20.38	20.59
Capital-Related	451.07	435.95	452.58	463.10	374.46
Total	708.83	688.61	709.96	726.29	600.95
By-product credits, \$MM/year					
Propane (\$12.50/B)	26.45	22.55	26.80	29.40	10.37
Butane (\$14.50/B)	14.59	14.17	14.76	17.13	8.37
Sulfur (\$52.00/T)	6.12	6.18	6.18	6.18	6.87
Ammonia (\$120.00/T)	6.34	6.44	6.72	6.34	5.75
Total	53.50	49.34	54.46	59.05	31.36
Net Product Cost, \$MM/year	655.33	639.27	655.50	667.24	569.59
Net Product Cost, \$/B	42.80	41.19	44.29	44.52	34.80
Equivalent Crude Oil Price, \$/B	36.87	35.18	38.45	38.69	28.72

Table 15 Breakdown of Equivalent Crude Oil Price

	PB - 5				
	Period 3	Period 8	Period 10	Period 16	Period 9
Product, B/D	46,607	47,251	45,058	45,627	49,832
Contribution to Price, \$/B					
Coal	5.05	4.94	5.26	5.21	4.53
Natural Gas	4.91	4.59	4.98	5.30	3.60
Water	0.03	0.02	0.03	0.03	0.02
Dispersed Catalyst	0.89	0.91	1.07	0.91	0.32
Other Catalysts & Chemicals	1.08	0.95	1.11	1.20	0.65
Waste disposal	0.11	0.11	0.12	0.12	0.10
Labor	1.28	1.25	1.33	1.32	1.16
Maintenance	1.15	1.12	1.20	1.18	1.04
Capital Related Costs	25.38	24.00	26.54	26.85	18.88
By-product Credit	-3.01	-2.71	-3.19	-3.43	-1.58
Equivalent Crude Oil Price, \$/B	36.87	35.18	38.45	38.69	28.72

FIGURES

Figure 1. Process Flow Diagram of Bench-Scale-Liquefaction Unit (HTI Unit 227)

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ISS	DRAWN	CHKD	APPR	ISS	DRAWN	CHKD	APPR	ISS	DRAWN	CHKD	APPR	ISS	DRAWN	CHKD	APPR
4				3				2				1			
Hydrocarbon Technologies, Inc. R & D Center Lawrenceville, N.J.												TITLE: UNIT 227 WITH INTERSTAGE SEPARATION AND IN-LINE HYDROTREATER FOR RUN 102			
												JOB NO. HTI DRAWING NUMBER 227SF041 ISSUE 1			

Figure 2. Daily Operating Conditions

Figure 3. Daily Material Balance

Figure 4. Coal and Residuum Conversion

Figure 5. C₄-524°C Distillate Yield & 524°C + Yield

Figure 6. Distillate Fraction

Figure 7. Distillate Selectivity

Figure 8. Hydrogen Consumption & Light C₁-C₃ Gas Yield

Figure 9. Hydrogen Efficiency & Light C₁-C₃ Gas Selectivity

Figure 10. Quality of SOH Distillate

Figure 11. Product H/C Ratio

Figure 12. Solubility of PFL Product

APPENDIX

Summary of Material Balance for Run 227-102, PB-07

PERIOD DATE	1 3/10/97	2 3/11/97	3 3/12/97	4 3/13/97	5 3/14/97	6 3/15/97	7 3/16/97	8 3/17/97	9 3/18/97	10 3/19/97	11 3/20/97	12 3/22/97	13 3/23/97	14 3/24/97	15 3/25/97	Overall
FEEDS, gms																
Illinois No 6 Coal	27486	31855	31734	30536	27687	24667	25899	30125	27135	31266	28674	25283	31399	30027	30207	434211
TNPS	3181	0	0	0	0	0	0	0	0	0	0	0	0	0	1718	4961
H2S	1047	1472	1531	1025	971	974	974	979	880	962	951	971	883	863	269	14750
L-814	15088	0	0	2313	0	11226	489	1943	2981	0	0	0	0	0	0	26454
HTI Fe Catalyst	781	773	770	740	703	599	710	731	658	759	0	774	763	729	706	10190
Molyvan-A	0	0	0	0	0	0	0	8	9	10	9	4	0	0	0	40
PFS	0	0	4131	5608	6012	5350	1227	4395	4455	5597	5276	4459	5224	5217	5518	62762
O-6 btms	16540	21244	0	0	0	0	0	0	0	0	0	0	0	0	0	37140
PFL	5455	12089	28107	28017	23966	20760	33728	27039	28619	27436	25362	36721	28989	27023	26008	379732
Water	14339	13663	13688	13704	13133	14152	14098	14387	14982	14146	13238	11790	13752	14703	15118	208893
Hydrogen Feed	7387	7390	7390	7390	7394	7393	7392	7397	7393	7404	7389	7429	7189	7356	7355	110647
TOTAL FEED	91304	88485	87351	89333	79866	85120	84517	87004	87113	87580	80899	87430	88199	85918	86899	1289780
PRODUCTS, gms																
Hydrogen in Product Gases	6243	5550	5228	5171	5136	5777	5771	5144	5554	5253	5422	5908	4643	5699	5673	82242
Product Gas (H & N free)	11770	9050	9376	9754	7668	4184	9699	7709	7528	8230	8287	9783	9747	8730	11731	133510
PFS	0	0	10350	10992	12229	12963	3382	10115	10139	10754	10864	6673	11028	9770	11538	130797
PFL	49519	39191	32633	28984	25318	26904	38604	32492	31048	29994	28509	38689	33220	27747	31180	494032
ST 1 SOH sample	0	0	232	204	0	100	100	0	0	629	0	0	0	0	220	1485
ST 2 SOH sample	0	0	0	227	105	102	203	201	223	335	208	226	263	209	280	2582
Knockouts	0	5	0	10	10	4	25	0	33	12	1	24	15	3	0	142
SOH-OIL	8317	13364	11852	12352	11438	10038	10234	11472	8362	11249	10725	6993	12778	11127	11310	161611
SOH-H2O	14208	16951	17865	18215	16157	17925	18048	17848	19763	17530	15822	12296	17869	18747	19277	258519
	90055	84111	87536	85909	78061	77997	86066	84982	82650	83986	79838	80593	89563	82032	91209	1264920
TOTAL PRODUCTS	90055	84111	87536	85909	78061	77997	86066	84982	82650	83986	79838	80593	89563	82032	91209	1264920
RECOVERY, W%																
Liquid Closure, W%	85.83	85.71	91.21	89.14	90.04	87.53	91.54	92.87	90.66	87.94	89.96	81.12	92.79	86.05	92.79	88.98
HYDROGEN CONSUMPTION																
Metered Cons., W% maf coal	4.77	6.61	7.80	8.32	9.34	7.50	7.17	8.56	7.76	7.88	7.85	6.89	9.28	6.32	6.38	7.49
Metered Cons., W% maf frsh fd	4.77	6.61	7.80	8.32	9.34	7.50	7.17	8.56	7.76	7.88	7.85	6.89	9.28	6.32	6.38	7.49

Summary of Material Balance by Condition, Run 227-102, PB-07

CONDITION DATE	Condition 1	Condition 2	Condition 3	Condition 5	Overall
FEEDS, gms					
Illinois No 6 Coal	121611	78253	88526	116916	434211
TNPS	3181	0	0	1718	4961
H2S	5074	2918	2822	2986	14750
L-814	15088	11715	0	0	26454
HTI Fe Catalyst	3064	2012	2148	2971	10190
Molyvan-A	0	0	27	4	40
PFS	9739	12589	14448	20419	62762
O-6 btms	37784	0	0	0	37140
PFL	73668	78453	83094	118742	379732
Water	55394	41383	43515	55363	208893
Hydrogen Feed	29556	22180	22194	29329	110647
TOTAL FEED	354159	249503	256773	348447	1289780
PRODUCTS, gms					
Hydrogen in Product Gases	22192	16684	15952	21924	82242
Product Gas (H & N free)	39950	21551	23467	39990	133510
PFS	21342	28574	31008	39009	130797
PFL	150327	90826	93534	130836	494032
ST 1 SOH sample	436	200	629	220	1485
ST 2 SOH sample	227	410	759	978	2582
Knockouts	15	39	45	42	142
SOH-OIL	45885	31710	31083	42208	161611
SOH-H2O	67237	52130	55141	68189	258519
	347611	242124	251618	343396	1264920
TOTAL PRODUCTS	347611	242124	251618	343396	1264920
RECOVERY, W%					
	98.16	97.04	97.99	98.54	98.07
Liquid Closure, W%	87.98	89.70	90.49	88.19	88.98

Detailed Gas Normalized Yield

Period Number	3	8	10	16
Period Start	03/12/97	03/17/97	03/19/97	03/25/97
Normalized Yield, W% Dry Coal				
C ₁	5.13	4.14	4.77	5.53
C ₂	4.60	3.57	5.39	5.20
C ₃	4.89	4.15	4.96	5.45
C ₄	2.45	2.37	2.49	2.91
C ₅	1.21	1.03	1.81	1.31
C ₆ -C ₇	3.29	4.56	1.61	8.03
Normalized Yields, W% Dry Coal				
C ₁ -C ₃	16.18	13.12	16.72	17.90
C ₄ -C ₇	7.70	8.80	6.54	13.56