DIRECT LIQUEFACTION PROOF OF CONCEPT FACILITY
Hydrocarbon Technologies, Inc., Lawrenceville, NJ

Final

Topical Report
POC Bench Run 05 (227-97)

Work Performed Under Contract No. AC22-92PC92148

For

U.S. Department of Energy
Pittsburgh Energy Technology Center
Performed by
Hydrocarbon Technologies, Lawrenceville, NJ
DIRECT LIQUEFACTION PROOF-OF-CONCEPT PROGRAM
Hydrocarbon Technologies, Inc., Lawrenceville, N.J.

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V.R. Pradhan
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APRIL 1997
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ABSTRACT

This report presents the results Bench Run PB-05, conducted under the DOE Proof of Concept - Bench Option Program in direct coal liquefaction at Hydrocarbon Technologies, Inc. in Lawrenceville, New Jersey. Bench Run PB-05 was the fifth of the nine runs planned in the POC Bench Option Contract between the U.S. DOE and Hydrocarbon Technologies, Inc. Bench Run PB-05 had multiple goals. These included the evaluation of the effect of using dispersed slurry catalyst in direct liquefaction of a high volatile bituminous Illinois No. 6 coal and in combined coprocessing of coal with organic wastes, such as heavy petroleum resid, MSW plastics, and auto-shredder residue. PB-05 employed a two-stage, back-mixed, slurry reactor system with an interstage V/L separator and an in-line fixed-bed hydrotreater.

Coproducts of waste plastics with Illinois No. 6 coal did not result in the improvement observed earlier with a subbituminous coal. In particular, decreases in light gas yield and hydrogen consumption were not observed with Illinois No. 6 coal as they were with Black Thunder Mine coal. The higher thermal severity during PB-05 is a possible reason for this discrepancy, plastics being more sensitive to temperatures (cracking) than either coal or heavy resid. The ASR material was poorer than MSW plastics in terms of increasing conversions and yields. HTI's new dispersed catalyst formulation, containing phosphorous-promoted iron gel, was highly effective for the direct liquefaction of Illinois No. 6 coal under the reaction conditions employed; over 95% coal conversion was obtained, along with over 85% residuum conversion and over 73% distillate yields.
EXECUTIVE SUMMARY

Bench Run PB-05 was the fifth 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 direct liquefaction and coprocessing of a high volatile bituminous coal, (Illinois No. 6), with organic wastes, such as MSW plastics (a mixture of HDPE, polypropylene, and polystyrene was used to simulate the MSW plastics), heavy resid, and auto-fluff. The unit was configured to have an interstage vapor-liquid separator and an in-line fixed-bed hydrotreating unit. The run consisted of 25 days of continuous operation, spanning six operating conditions. The effect of dispersed slurry catalyst activity and feed composition were evaluated at target operating conditions of reactor space velocity of 640 kg/h/m³ reactor and reactor temperatures of 449 and 460°C.

The first two reaction conditions were carried out using 'coal-only' feed, with the objective of studying the effect of 50 ppm molybdenum, added as Molyvan-A, in improving performance over use of only iron and phosphorous in the form of HTI’s gel catalyst (as in Condition 1 of PB-05). Conditions 3 through 6 focused on the coprocessing of organic wastes with coal; Condition 3 studied the coprocessing of coal with MSW plastics and Hondo VTB oil; Conditions 4 and 5 studied the coprocessing of coal with MSW plastics and auto-fluff material; and Condition 6 looked at coal/oil coprocessing at 50/50 w/w% feed concentration. The run was carried out in an “ashy-recycle” mode of operation, achieved by recycling part of the flash vessel, (O-6), bottoms back to the first stage reactor. The in-line hydrotreater operation was very successful (except for the last run condition); SOH product with a high H/C ratio and low heteroatom content was obtained.

Following were the highlights of Bench Run PB-05:

- HTI’s new dispersed catalyst formulation, containing phosphorous-promoted iron gel, was highly effective for the direct liquefaction of Illinois No. 6 coal under the reaction conditions employed; over 95% total coal conversion was obtained, along with over 85% residuum conversion and over 73% distillate yields.

- The addition of 50 ppm molybdenum, in the form of Molyvan-A, to HTI’s Fe/P gel catalyst did not bring about any significant improvement in process performance. It appears that the presence of phosphorous masks the effect of the molybdenum additive.
The coprocessing of equal amounts of coal, MSW plastics, and Hondo VTB resid resulted in improved performance, e.g., about 90% conversion of resid was obtained, along with 79% distillate yield and 3.9% chemical hydrogen consumption.

Coprocessing waste plastics with Illinois No. 6 coal did not result in the improvement observed earlier with a subbituminous coal. In particular, reduced light gas yields and hydrogen consumption were not observed with Illinois No. 6 coal as was the case with Black Thunder Mine coal. The higher thermal severity during PB-05 could be the reason for this discrepancy, plastics being more sensitive to temperatures (cracking) than either coal or heavy resid. The ASR material was poorer than MSW plastics in terms of contributing to conversion and yields.

The last run condition, which studied conventional coal/oil coprocessing with minimal recycle, resulted in the best performance, although material recovery balance for that condition was poor.

**BACKGROUND, OBJECTIVE, AND SCOPE OF WORK**

The POC Bench Option Project (PB-Series) is geared to evaluate different novel processing concepts in catalytic coal liquefaction and coprocessing of organic wastes, such as plastics, heavy resids, waste oils, and ligno-cellulose wastes, with coal. The long-term performance data at the bench scale of operations (30 kg/day) will be used eventually to complement the larger scale process demonstration "Proof-of-Concept" studies for the U.S. DOE. The new ideas being explored in this program include using novel dispersed slurry catalysts, combinations of dispersed and supported catalysts (hybrid mode), coprocessing of coal with waste plastics, low quality resids, waste oils, and ligno-cellulosic wastes, etc. Bench Run PB-05 was carried out for 25 operating days, spanning five process conditions (Table 1), to evaluate the effects of an all-dispersed catalyst system (iron and molybdenum based dispersed catalyst only in both reactor stages, K-1 and K-2), on the direct liquefaction of a bituminous Illinois No.6 coal and on the combined processing of Illinois No. 6 coal with waste plastics, heavy petroleum resid, and auto-fluff. The following were the primary objectives:
To examine the effects of iron, molybdenum, and phosphorous-based dispersed slurry catalysts on the direct liquefaction of Illinois No. 6 coal, using a high-low two-stage temperature sequence, with interstage product separation and in-line distillate hydrotreating.

To study the coprocessing of Illinois No. 6 coal with heavy petroleum resid, waste plastics, and auto-fluff in an "all-dispersed catalyst" two-stage hydroconversion reaction system.

**SYSTEM CONFIGURATION**

Bench Run PB-05 involved two equal volume back-mixed reactors with internal recirculation. No supported extrudate catalyst was employed in the hydroconversion reactors; only the in-line HTU treating the net product used 500 cc of Criterion C-411 catalyst. Direct liquefaction of coal and the coprocessing of vacuum resids, waste plastics, and auto-fluff with coal was studied using a coil preheater, interstage separation, and in-line hydrotreating of light products from both stages. The simplified schematic of the unit configuration is shown in Figure 1.

Unit 227 was used in this run. Coal, resid, auto-fluff (premixed with recycle PFL) and waste plastics were introduced through the feed system of Unit 227. Light products and gases from the first stage were removed from the interstage separator. After separating out the process water, the oil fraction was repressurized and combined with process knockouts and the second stage hot separator (O-1) overheads for feeding to the in-line hydrotreater. A slurry product was removed from the bottom of the hot separator. An off-line pressure filtration unit was used to recover a solids-free liquid (recycle solvent) from the slurry product. Two independent gas feed and discharge systems were required to handle the gas from the two-stage-interstage separation configuration.
MATERIALS USED

Feeds

Illinois No. 6 coal (HTI #6303) was used for PB-05 (227-97) Bench Run. Hondo VTB was the source of petroleum resid, while a 40/30/30 w/w/w% mixture of HDPE/PP/PS simulated the MSW co-mingled plastics, while ASR was used in the form of a pre-mixed with PFL. The analyses of these feedstocks are listed in Tables 2 and 3.

Start-up and Make-up Oil

L-814; analysis shown in Table 4.

Catalysts

Hydrotreater: Criterion C-411 Trilobe (HRI-6135)
K-1 & K-2: HTI’s phosphorous-modified iron catalyst and Molyvan-A, added to reactor K-1 by premixing with recycle PFL.

INTERSTAGE (Reactor K-1) SLURRY SAMPLES

Five interstage (reactor K-1) slurry samples, one for each condition, were collected. These samples were taken immediately after the completion of each of the work-up periods.

EXTERNAL SAMPLES

The following samples of different process streams were collected during work-up periods for Consol, Inc.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weight</th>
</tr>
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<tbody>
<tr>
<td>Feed Coal</td>
<td>100 g</td>
</tr>
<tr>
<td>O-6 Bottoms</td>
<td>300 g</td>
</tr>
<tr>
<td>First Stage Overhead Oil</td>
<td>300 g</td>
</tr>
<tr>
<td>PFL</td>
<td>250 g</td>
</tr>
<tr>
<td>Feed Slurry</td>
<td>200 g</td>
</tr>
<tr>
<td>First Stage Slurry</td>
<td>300 g</td>
</tr>
<tr>
<td>SOH Product</td>
<td>300 g</td>
</tr>
<tr>
<td>PFS</td>
<td>250 g</td>
</tr>
</tbody>
</table>

Five gallon SOH distillate samples were also collected for DOE/PETC during the HTU off-line conditions of PB-05.
SUMMARY OF OPERATIONS

Start-up Operations

The 500 cc of Criterion 411 catalyst (HTI-6292) that was charged to the hydrotreater had an outage before charging of 38% and an outage after charging of 12¾%.

The unit was mechanically complete and pressure checked for startup by 0500 hours August 6. Normal start-up commenced without incident. By August 8 the start-up was completed, and the unit was lined out and ready for run operations.

Condition 1 (Periods 1-5)

Condition 1 began at 0400 hours on August 8. The feed for the condition was a blend of 1384 g/hr of coal, 831 g/h of O-6 bottoms slurry, and 553 g/h of Pressure Filtered Liquid (PFL). 13.2 g/h of phosphoric enhanced iron catalyst was also part of the feed slurry. There were minor problems with the feed pumps, J-1 and J-2. Twice plugs developed in the feed lines that could not be cleared with high pressure. A pressure drop began to develop in the coil and in the first stage reactor (K-I) as early as Period 3. By the end of Condition 1 pressure drops in both the first (K-1) and second stage (K-2) were as high as 50 psi. The unit parameters were held until the end of the condition so as not to disturb the unit.

Condition 2 (Periods 6-9)

Condition 2, began at 0400 hours on August 13 with the addition of 0.23 g/h of Molyvan-A catalyst to the reactor feed. All other parameters remained the same. Due to a 300 psi pressure drop in the feed section (namely the coil), which started late in Condition 1, the unit was placed on wash at 1030 hours in subperiod 6A, (the condition transition period). The unit was back on-line by 1500 hours, before the end of subperiod 6A, with restrictions freed. During period 8, at 0600 hours the hydrotreater (HTU) was taken off-line for two hours so the HTU feed pump could be repacked. The hydrotreater was placed back on-line by 0800 hours without incident.

Condition 3 (Periods 10-13)

Condition 3 began at 0400 hours August 17 with the introduction of the plastics blend to the unit. The coal feed was lowered to 471 g/h, and Hondo oil was added to the
unit at 457 g/h. The plastics blend consisted of 183 g/h high density polyethylene (HDPE), 137 g/h Polypropylene (PP), and 137 g/h Polystyrene (PS). At 0800 hours during Period 11, the hydrotreater was taken off-line to obtain non-hydrotreated samples. The hydrotreater was returned to service at 1600 hours with the initiation of subperiod 12B. The unit ran smoothly throughout Condition 3.

**Condition 4 (Periods 14-17)**

Condition 4, commenced at the start of period 14 at 0400 hours on August 21. The Hondo oil feed was discontinued, and the plastics blend remained the same as in Condition 3. The coal feed rate was raised to 927 g/h, the O-6 bottoms slurry feedrate was raised to 996 g/h, and the pressure filter liquid feedrate was raised to 664 g/h. The viscosity of the feed rose to as high as 10,000 cps; in response the feed pot temperatures were raised from 425°F to 475°F in an effort to lower viscosity. The feed line temperatures were also raised incrementally. Viscosity, however, dropped only modestly. The high viscosity created no operational difficulties, and the unit continued smoothly.

**Condition 5 (Periods 18-21)**

Condition 5, began at 0400 hours on August 25. The feed composition changes included the introduction of Auto Shredder Residue (ASR) also called auto-fluff, at 229 g/h and a reduction of the plastics blend feed rate by exactly one half. Plastics feed was 92 g/h HDPE, 69 g/h PP, and 69 g/h PS. During Condition 5, the second stage left side buffer pump stopped on several occasions due to restrictions. The restrictions were cleared, pump checks were replaced, and the pump was placed back in service. Feed pump problems continued throughout the condition. During period 21 the hydrotreater plugged and could not be cleared. It was taken off-line for the remainder of the run.

**Condition 6 (Periods 22-25)**

Condition 6, began at 0400 hours on August 26. The four day condition experienced smooth operations feeding only three components, 692 g/h of coal, 692 g/h of Hondo oil, and 277 g/h of O-6 bottoms slurry.
Shut-down and Post Run Inspection

At 0400 hours on September 2 shut-down procedures were initiated as planned. Unit temperatures were lowered and the unit was washed as specified in the procedure.

Inspection of the unit after shutdown showed that the feedpot, P-2, was approximately half full of hard carbonaceous material. Five gallons of very heavy solids were removed from P-2. The circulation lines to P-2 were plugged. The preheater coil and the L/S buffer line both had plugs as well. The hydrotreater had to be rodded out to recover 684 g of catalyst. The hydrotreater catalyst was mostly black with some white pieces mixed in. Neither the first nor second stage bubble caps came out upon removal of the bottom heads and riser tubes. Both reactor top heads had to be cleaned by maintenance due to modest amounts of hard carbonaceous deposits. Both bottom heads were cleaned by operations to remove small amounts of hard carbonaceous material. The run chronology and statistics can be found in the following Tables. Run 227-98 had an online efficiency of 99.3% during the 25 day run.

**RUN 227-97 OPERATIONS-STATUS-CLASSIFICATION CHRONOLOGY**

<table>
<thead>
<tr>
<th>Operations Classification</th>
<th>Start Time</th>
<th>Start Date</th>
<th>End Time</th>
<th>End Date</th>
<th>Duration Hours</th>
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<td>I.D.</td>
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<tr>
<td>S/U</td>
<td>0500</td>
<td>8/6/96</td>
<td>0400</td>
<td>8/8</td>
<td>47</td>
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<tr>
<td>Cond. 1</td>
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<td>0400</td>
<td>8/13</td>
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<tr>
<td>Cond. 2</td>
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<td>8/13</td>
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<td>8/13</td>
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<td>Wash</td>
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<td>8/25</td>
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<td>Cond. 5</td>
<td>0400</td>
<td>8/25</td>
<td>0400</td>
<td>8/29</td>
<td>96</td>
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<tr>
<td>Cond. 6</td>
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<td>8/29</td>
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<td>9/2</td>
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<tr>
<td>S/D</td>
<td>0400</td>
<td>9/2</td>
<td>1400</td>
<td>9/3</td>
<td>34</td>
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## ON-LINE TIME AND DOWN TIME FOR RUN 227-97

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<td>81</td>
<td>4.5</td>
<td>681</td>
<td>4.5</td>
<td>99.2</td>
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**Definitions**
- **S/U = Startup.** Time between gas flow initiation and feedstock cut-in during which unit temperatures and/or pressures are being increased.
- **S/D = Shutdown.** Time between feedstock cut-out and liquid flow termination during which unit temperatures and/or pressures are being decreased.
- **On-Line Time.** The sum of S/Us, S/Ds, and Run-Periods.
- **D/T = Down-Time.** The time during which gases and liquids are not being charged to the unit. This is the same as the time between an intermediate shutdown and startup.
- **On-Line Efficiency =** On-Line Time / (On-Line Time + Down-Time + Unfinished Run-Time.)
OPERATING CONDITIONS AND MATERIAL RECOVERY BALANCES

Bench run operations were smooth and without any major problems. The overall material recovery balances were on the low side throughout Bench Run PB-05. An average material recovery balance (the daily material balance summary is attached in the Appendix) of about 94.5 W% was obtained (Figure 3) for the entire Bench Run PB-05. The operating summary and the process performance of individual periods during PB-05 are shown in Table 5. Figure 2 shows feed space velocities and reactor temperatures during PB-05. As shown in Figure 2, the increased reactor temperatures during Conditions 1 through 5 were uniform while the space velocity fluctuated around an average value of 640 kg/h/m². The feed composition, in terms of weight percents of different feedstocks, is shown in Figure 4.

PROCESS PERFORMANCE RESULTS

The conversions and yields of different products, process performance, and product quality for PB-05 are addressed in this Section. The calculation of daily material recovery balances, coal conversions, normalized product yields, and other process performance-related indicators was carried out using programs available in the microvax database. (Some programs were also modified as per the requirement of the process configuration for PB-05, for example, handling of three independent gas streams for material balance). Process performance during PB-05 is summarized in Table 5, depicted in Figures 5 through 10, and discussed in detail in the following sections.

Total Coal (mixed-feed) and 524°C+ Residuum Conversion

Typical coal (or mixed 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-05, are shown in Figure 5. Coal conversion (W% maf feed) varied between about 95.6 to 99% maf throughout the course of the run. Conversion was the highest for the condition studying the coprocessing of equal amounts of coal, Hondo resid, and MSW plastics. The conversion of 524°C+ residuum from coal is also plotted in Figure 5. Residuum conversion varied between about 82 to 90 W% (maf feed). For the operating conditions which processed either
coal alone or a mixture of coal and MSW plastic, the resid conversion was around 85 W% maf. Conversion increased to over 89 W% maf during Conditions 3 and 6 which processed the mixture of equal amounts of coal, Hondo resid, were MSW plastics and the mixture of equal amounts of coal and Hondo resid. During condition 5, which employed auto-fluff, resid conversion was the lowest of all, (81.6 W% maf). The addition of 50 ppm molybdenum as Molyvan-A during condition 2 did not have any major impact on coal and residuum conversions.

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

Distillate yields and 524°C+ residuum yields are shown in Figure 6. Distillate yields varied between 72 and 79 W% maf. The highest distillate yields of 79 W% maf were obtained during conditions 3 and 6, both coprocessing conditions. Distillate yields were around 73 W% maf for the first two ‘coal only’ conditions. The lowest distillate yield and the highest residuum yield were obtained during condition 5, in which part of the mixed MSW plastics were replaced by auto-fluff. Not much effect of the addition of 50 ppm molybdenum was observed in going from condition 1 to condition 2 of the ‘coal only’ operation.

**Distillate Yield/Selectivity**

The yields of distillate fractions, such as naphtha (IBP-177°C), middle distillates (177-343°C), and heavy distillates (343°C+), are shown in Figure 7. The numbers plotted in this figure, converted to a selectivity basis, are shown in Figure 8. Selectivities for different distillate fractions were similar for the first two condition. The addition of plastics to the feed mixture during conditions 3 and 5 increased selectivity to the lightest naphtha fraction. The addition of auto-fluff to the feed mixture during condition 5 reduced light fraction selectivity. The last run condition, condition 6, which studied the coprocessing of coal and Hondo resid, resulted in the highest selectivity/yield of the middle distillate fraction.

**Hydrogen Consumption and Light Gas (C₁-C₃) Yield**

Hydrogen consumption (Figure 9) based on maf coal (mixed feed) varied between 3.4 to 5.8 W%. In general, chemical hydrogen consumption during PB-05 tendency to be on the low side, primarily because no supported catalyst was used and space velocities were high; thus, the operating severity was on the low side. Hydrogen consumption was low (<4 W% MAF) for conditions 3 and 6, which both used mixed
feeds containing mixed plastics and Hondo resid with coal. Light gas (C₁-C₃) yields varied between 5.2 and 9.0 W% maf, with the highest gas yield during the coprocessing condition feeding (33% each of coal/Hondo/plastics).

Hydrogen Utilization

Hydrogen utilization during coal conversion is characterized by two indicators: hydrogen efficiency and C₁-C₃ gas selectivity. The former is defined as the amount of C₄-524°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 10, hydrogen efficiency varied between 12 to 24%. The lowest hydrogen efficiency of 12% was obtained when auto-fluff was used as part of the feed with coal and mixed plastics. The highest hydrogen efficiency and the lowest light gas selectivity (the most efficient hydrogen utilization) was obtained for the last run condition, condition 6, which studied the coprocessing of 50/50 w/w% coal and Hondo resid.
PRODUCT QUALITY

Different first-stage and second-stage product fractions (product gases, SOH, PFL, and PFS) from work-up periods 4, 9, 13, 17, 21, and 25 were analyzed in detail for their composition. These analyses fractions are listed in Tables 7 through 12.

Separator Overhead Product (SOH)

The SOH oil stream represents the net light distillate (IBP-343°C) from PB-05. While the hydrotreater unit was on-line (all the operating days except for Periods 20 through 25), the only major distillate stream out of the unit was the SOH stream, as the O-1 hot separator overheads, atmospheric flash overheads, first stage overhead oil, and the unit knockouts were being fed to the hydrotreater. The properties of SOH oil from both stages for the work-up periods are shown in Tables 6 and 7. The second stage SOH oils, which represent a major portion of the net process distillates, had a typical boiling range of 40-380°C. The API gravities were high (> 35), and the H/C atomic ratios were also high (> 1.70), especially during the operating periods when the HTU was online. Conditions 5 and 6, when the in-line HTU had to be by-passed due to plugging, produced a poorer quality SOH oil. The nitrogen and sulfur contents also increased significantly, as shown in Figure 11. The quality of the SOH process distillates (second stage), in terms of heteroatoms content and H/C ratios, is depicted in Figures 11 and 12. The first stage overhead oil, SOH, was understandably poorer in quality than the second stage overheads.

Pressure Filter Liquid (PFL) and Pressure Filter Solids (PFS)

The flash vessel (O-6) bottoms go through pressure filtrations for separation of solids from heavy liquid product and recycle oil. The liquid oil, called pressure filter liquid (PFL), is usually heavier than 343°C boiling point and contains unreacted heavy residuum material to a varying degrees. The solids from filtration, PFS, are oil-containing solids, normally extracted with toluene for oil recovery. The oil-free solids are then used for determining the extent of coal or total feed conversion based upon the solubility of the PFS material in a solvent such as quinoline. Detailed analyses of the PFL and PFS streams from PB-05 are listed in Tables 8 through 11. As shown in Tables 8 and 9, the PFL API gravity was low for most of the run conditions. The resid content of the second stage unit PFL varied between 29 and 36 W%, while resid in the interstage (first stage) PFL varied between 37 and 50 W%. In general, the asphaltene and the preasphaltene contents of the PFL from both reactor stages were
on the high side (Figure 13). The H/C ratios were higher for coprocessing conditions than for ‘coal-only’ conditions. As shown in Tables 10 and 11, the H/C ratios of the pressure filter solids (sampled and analyzed prior to extraction with toluene) were high (around 1.00) due to oil retention. The coal conversion, based upon the quinoline solubility of the pressure filter solids corrected to the ash content of feed coal, was around 96 W% maf throughout the run (almost all coal/feed conversion occurred in the first stage).
ANALYSIS OF PROCESS PERFORMANCE RESULTS

Effects of Feed Type

One of the primary objectives of Bench Run PB-05 was to study combined hydroconversion processing of mixed plastics, auto-fluff, and Hondo resid with a high volatile bituminous Illinois No. 6 coal. In previous runs, coprocessing of mixed feeds always resulted in better process performance than liquefaction of coal only. Distillate yields were higher for all of the coprocessing conditions, along with lower gas makes and mostly lower hydrogen consumptions, i.e., higher hydrogen efficiencies for coprocessing operations. The addition of plastics and Hondo resid, both separately and combined, had an overall positive effect on process performance, as shown in Figure 14. However, the addition of auto-fluff (ASR) had a negative impact on process performance, especially distillate yields and resid conversion, as shown in Table 5.

Effect of Coal Rank/Type on Coprocessing Performance

Coprocessing of waste plastics with Illinois No. 6 coal did not show the improvement observed earlier with a subbituminous coal. In particular the decrease in light gas yield and hydrogen consumption were not observed with Illinois No. 6 coal as was the case with Black Thunder mine coal. The higher reaction severity during PB-05 could be a reason for this discrepancy, plastics being more sensitive to temperature (cracking) than either coal or heavy resid.

It was well established during earlier HTI bench-scale runs that addition of mixed waste plastics improved overall hydrogen utilization in liquefaction of subbituminous Wyoming Black Thunder Mine coal. Mixed plastics addition at 25, 33, and 50 W% had resulted in an improved distillate yield and a lower light gas make. The hydrogen consumption was also lowered by the plastics addition. During PB-05, in which a high volatile bituminous Illinois No. 6 coal used, the effect of mixed plastics on distillate yields and quality was similar to that observed earlier with Black Thunder mine coal. There was an increase in distillate yield during the coal/plastics coprocessing operation compared to the 'coal only' case. However, surprisingly, the light C_1-C_3 gas makes and hydrogen consumptions were not lower during the coal/plastics coprocessing operations than they were with the 'coal only' operation. This indicates that the effect of plastics on coal conversion is either coal-dependent or reaction severity-dependent or both. It should be noted that the overall thermal severity during
PB-05, feeding Illinois No. 6 coal, was about 20% higher than the earlier runs conducted using Black Thunder Mine coal. Also, as shown in Figure 15, the coprocessing of Hondo resid had much more of an impact with Illinois No. 6 coal than with Black Thunder coal.
TECHNO-ECONOMIC ASSESSMENT

Feed to Bench Run PB-05 was automobile shredder residue (ASR) in combination with Illinois coal, residual oil and plastics. The run featured an interstage separator and in-line hydrotreater and used dispersed iron and phosphorus catalysts. Recycle concluded topped separator bottoms containing solids.

Feed compositions are presented in Table 12 for the periods analyzed in this assessment of the run. Reactor operating conditions were held nearly constant throughout the run as shown in Table 13, which also summarizes process performances and yields.

Table 14 presents material balances. Liquids production in coal-only operation was outstanding, even better than observed in PB-04. With residual oil coprocessing, liquids production increased, while with ASR coprocessing the liquid yield decreased. Table 15 summarizes hydrogen balances, utilities production, and thermal efficiencies for the run conditions analyzed.

Liquefaction plant investment details, listed in Table 16, show little difference among the PB-05 run conditions except for Periods 21 and 25. Total plant investment costs are shown in Table 17. Coal-only operation investments are more than ten percent below those in PB-04. Use of ASR increases the investment cost slightly, but lowers the cost on a daily barrel cost basis.

Economics are summarized in Table 18, and the breakdown of the equivalent crude oil price is shown in Table 19. All the periods investigated showed better economics than with coal-only operation. The equivalent crude oil price for coal-only operation is almost $30/B, even better than in PB-04. Table 20 shows comparison of the economics of the cases at maximum reactor throughput. Figure 16 compares economic results feeding subbituminous coal in PB-04 with bituminous coal in PB-05.

In summary, results of the technical assessment of PB-05 again show that use of Automobile Shredder Residue is economically justified when no charge is taken for this feed. The addition of phosphorus, as previously found, is economically beneficial, especially in coal-only operation. The use of oil and plastics is again shown economically attractive.
CONCLUSIONS

The following conclusions can be drawn based upon the data obtained from Bench Run PB-05:

- HTI's new dispersed catalyst formulation, containing phosphorous-promoted iron gel catalyst, was highly effective for the direct liquefaction of Illinois No. 6 coal under the reaction conditions employed; over 95% total coal conversion was obtained along with over 85% residuum conversion, and over 73% distillate yields.

- The addition of 50 ppm molybdenum, in the form of Molyvan-A, to HTI's Fe/P gel catalyst did not bring about any significant improvement in process performance. The presence of phosphorous appears to mask the effect of molybdenum.

- The coprocessing of equal amounts of coal, MSW plastics, and Hondo VTB resid resulted in improved performance, e.g., about 90% conversion of resid was obtained along with 79% distillate yield and 3.9% chemical hydrogen consumption.

- Coprocessing waste plastics with Illinois No. 6 coal did not bring about the improvement observed earlier with a subbituminous coal. In particular, decreases in light gas yield and hydrogen consumption were not observed with Illinois No. 6 coal as was the case with Black Thunder mine coal. The higher thermal severity during PB-05 could be the reason for this discrepancy, since plastics are more sensitive to temperatures (cracking) than either coal or heavy resid. The ASR material was poorer than MSW plastics in terms of contributing to conversions and yields.

- The last run condition, which studied conventional coal/oil coprocessing with minimal recycle resulted in the best performance, although material recovery balances for that condition were poor.
# Table 1. Run Plan for Bench Run PB-05

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Recycle Type

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Space Velocity, kg/h/m³ react

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Temperatures, °C

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Table 2. Analysis of Illinois No. 6 Feed Coal

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Table 3. Analysis of Heavy Oil, ASR, and Waste Plastics Feed

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Table 5. PB-05: Process Performance Summary

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**PROCESS CONDITIONS**

- **Space Velocity, mf fresh feed / reactor volume**
  - K-1 (kg/h/m³) 617 677 579 669 758 593
  - K-2 (kg/h/m³) 617 677 579 669 758 593

- **Reactor Temperatures, ºC**
  - K-1 444 447 447 447 448 447
  - K-2 454 458 457 458 458 457

- **Feed Composition, W%**
  - Coal (Ill. No. 6 Crown Il Mine) 100 100 33 67 67 50
  - Hondo VTB Oil 0 0 33 0 0 50
  - ASR 0 0 0 0 16 0
  - MSW Waste Plastics 0 0 33 33 17 0

- **Dispersed Catalyst Loading, ppm**
  - Iron 1000 1000 1000 1000 1000 1000
  - Molybdenum 0 50 50 50 50 50
  - Phosphorous 100 100 100 100 100 100

**NET NORMALIZED YIELDS, W% maf fresh feed**

- C₁-C₃ in Gases 7.23 6.74 9.00 8.23 7.09 5.19
- C₄-C₆ in Gases 2.33 2.17 5.06 4.51 2.65 2.29
- IBP-177°C (IBP-350°F) 16.90 16.23 27.63 25.21 17.63 15.06
- 177-260°C (350-500°F) 16.53 15.96 13.84 10.72 12.97 23.84
- 260-343°C (500-650°F) 16.05 16.31 14.49 12.30 12.46 18.73
- 343-454°C (650-850°F) 17.00 17.12 13.34 15.77 20.20 14.49
- 454-524°C (850-975°F) 4.35 5.58 4.47 6.05 6.49 4.63
- 524°C+ (975+°F) 10.25 10.97 9.53 12.83 14.59 8.68
- Unconverted Feed 4.44 3.78 0.91 2.91 3.81 2.29
- Water by Forced Oxygen Bal. 4.16 4.27 1.88 3.02 3.26 2.83
- COₓ 1.56 1.45 0.85 1.08 1.08 0.63
- NH₃ 1.40 1.34 0.61 0.79 1.08 0.76
- H₂S 3.32 3.33 2.29 2.01 2.52 3.91

**PROCESS PERFORMANCE, W% maf fresh feed**

- Material Recovery Balance 101.08 97.91 94.48 95.62 97.35 91.67
- Hydrogen Consumption 5.52 5.23 3.90 5.43 5.80 3.37
- Total Feed Conversion
  - (SO₂ Free) 95.6 96.2 99.1 97.1 96.2 97.7
- 524°C+ Conversion 85.3 85.3 89.6 84.3 81.6 89.0
- C₄ 524°C Distillates 73.2 73.4 78.8 74.6 72.4 79.1
Table 6. First Stage Separator Overhead (SOH) Properties

(Composite Sample)

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Elemental Analysis

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H/C Ratio

|        | 1.44 | 1.36 | 1.55 | 1.54 | 1.67 |

ASTM D-86 Distillation, W%

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<td>33.7</td>
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<tr>
<td>177-260°C</td>
<td>42.8</td>
<td>38.8</td>
<td>17.2</td>
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<td>32.91</td>
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<tr>
<td>260-343°C</td>
<td>27.1</td>
<td>36.8</td>
<td>25.1</td>
<td>28.1</td>
<td>30.51</td>
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<tr>
<td>343°C+</td>
<td>11.2</td>
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<td>14.29</td>
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<td>Loss</td>
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<td>0.3</td>
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<td>1.1</td>
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Table 7. Second Stage Separator Overhead (SOH) Properties

(Composite Sample)

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<td>FBP, °C</td>
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Elemental Analysis

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<td>Hydrogen, W%</td>
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<td>11.85</td>
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<td>Sulfur (Antek), ppm</td>
<td>98.14</td>
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<td>4724.5</td>
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<td>Nitrogen (Antek), ppm</td>
<td>2.82</td>
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<td>11000</td>
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<td>H/C Ratio</td>
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ASTM D-86 Distillation, W%

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<td>IBP-177°C</td>
<td>34.0</td>
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<td>39.07</td>
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<tr>
<td>260-343°C</td>
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<td>23.6</td>
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<tr>
<td>343°C+</td>
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<td>6.1</td>
<td>5.9</td>
<td>6.4</td>
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<tr>
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<td>1.1</td>
<td>2.2</td>
<td>1.3</td>
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Table 8. Pressure Filter Liquid (PFL) Properties

(Composite Sample)

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<td>Gravity, °API</td>
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<td>-8.5</td>
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<td>IBP, °C</td>
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Elemental Analysis, W%

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<td>87.87</td>
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<td>88.27</td>
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<td>Hydrogen</td>
<td>6.59</td>
<td>6.73</td>
<td>8.77</td>
<td>9.96</td>
<td>9.76</td>
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<td>Nitrogen</td>
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| H/C Ratio | 0.89 | 0.91 | 1.19 | 1.36 | 1.33 | 1.12 |

ASTM D-1160 Distillation, W%

<table>
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<td>343-454°C</td>
<td>42.09</td>
<td>35.91</td>
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<tr>
<td>454-524°C</td>
<td>12.82</td>
<td>14.35</td>
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<td>17.22</td>
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<td>15.56</td>
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<td>524°C+</td>
<td>30.32</td>
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<td>35.81</td>
<td>36.50</td>
<td>35.72</td>
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<tr>
<td>Loss</td>
<td>0.53</td>
<td>0.61</td>
<td>0.38</td>
<td>0.30</td>
<td>0.55</td>
<td>0.27</td>
</tr>
</tbody>
</table>

| Cyclohexane Insolubles | 82.28 | 88.5 | 66.34 | 60.46 | 74.57 | 70.16 |
| Toluene Insolubles     | 21.17 | 17.7 | 29.51 | 29.78 | 20.87 | 14.00 |
Table 9. Pressure Filter Liquid (PFL) Properties

(Interstage Sample)

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<td>25</td>
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<td>Gravity, °API</td>
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Elemental Analysis, W%

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<td>87.97</td>
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<td>8.04</td>
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<td>0.94</td>
<td>0.92</td>
<td>1.52</td>
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<td>1.05</td>
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ASTM D-1160 Distillation, W%

| IBP-343°C | 13.21 | 10.09 | 5.15 | 5.52 | 9.08 | 8.13 |
| 343-454°C | 31.91 | 32.16 | 30.93 | 27.46 | 37.24 | 26.13 |
| 524°C+    | 40.07 | 43.62 | 47.11 | 50.15 | 37.60 | 47.50 |
| Loss      | 0.89  | 0.16  | 0.52  | 0.75  | 0.46  | 0.11 |
| Cyclohexane Insolubles | 87.63 | 68.14 | 74.78 | 54.19 | 39.86 |
| Toluene Insolubles     | 26.71 | 23.14 | 28.73 | 19.40 | 9.87  |
Table 10. First Stage Pressure Filter Solid (PFS) Properties

(Composite Sample)

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Elemental Analysis, W%

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H/C Ratio

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Composition, W%

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<td>2.95</td>
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Coal Conversion,

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**Elemental Analysis, W%**

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<tr>
<td>Sulfur</td>
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<td>4.55</td>
<td>4.95</td>
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<td>Nitrogen</td>
<td>0.53</td>
<td>0.61</td>
<td>0.62</td>
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<td>H/C Ratio</td>
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<td>0.82</td>
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**Composition, W%**

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<td>Quinoline Insolubles</td>
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<td>68.9</td>
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<td>Ash (in Quinoline Filt.)</td>
<td>44.08</td>
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<td>1.77</td>
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<td>1.53</td>
<td>2.62</td>
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**Coal Conversion, W% MAF**

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Table 12. Composite Feed Analysis

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<td>Coal</td>
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<td>Residue</td>
<td></td>
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<td>Plastics</td>
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Ultimate Analysis, W%

<table>
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<tr>
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<td>Carbon</td>
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<td>8.93</td>
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<td>Ash</td>
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<td>10.24</td>
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Heating Value, BTU/lb

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<td>13,290</td>
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Table 13. Material Balance For A Feedrate of 12,000T/D

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<th>Period 13</th>
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<th>Period 25</th>
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<tbody>
<tr>
<td>Feed</td>
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<td></td>
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<tr>
<td>Coal, T/D</td>
<td>12,000</td>
<td>12,000</td>
<td>4,000</td>
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<td>Oil, B/D</td>
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<td>0</td>
<td>22,243</td>
<td>0</td>
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<td>33,365</td>
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<td>Auto Shredder Residue, T/D</td>
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<td>0</td>
<td>0</td>
<td>0</td>
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<td>Plastics, T/D</td>
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<tr>
<td>Total, T/D</td>
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<tr>
<td>Liquid Products, B/D</td>
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<td>Gasoline</td>
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<td>Barrels of Product/Ton Feed</td>
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<td>4.63</td>
<td>4.10</td>
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<td>4.76</td>
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<td>By-products</td>
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<td>Propane, B/D</td>
<td>2,702</td>
<td>2,526</td>
<td>3,378</td>
<td>3,670</td>
<td>3,646</td>
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<td>Butane, B/D</td>
<td>1,880</td>
<td>1,757</td>
<td>3,989</td>
<td>3,247</td>
<td>1,924</td>
<td>2,161</td>
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<td>Sulfur, T/D</td>
<td>413</td>
<td>402</td>
<td>274</td>
<td>257</td>
<td>278</td>
<td>439</td>
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<td>Ammonia, T/D</td>
<td>152</td>
<td>146</td>
<td>70</td>
<td>88</td>
<td>117</td>
<td>87</td>
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<td>Waste to disposal, T/D</td>
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<td>1,493</td>
<td>631</td>
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Table 14. Hydrogen Balance, Utilities & Thermal Efficiency

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<th>Period 9</th>
<th>Period 13</th>
<th>Period 17</th>
<th>Period 21</th>
<th>Period 25</th>
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<tr>
<td>Hydrogen Balance</td>
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<td>Hydrogen Consumption, MMSCFD</td>
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<td>Liquefaction</td>
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<td>-32.0</td>
<td>-36.3</td>
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<td>Solution &amp; Purge losses</td>
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<td>9.2</td>
<td>11.0</td>
<td>10.2</td>
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<td>Total</td>
<td>296.3</td>
<td>293.5</td>
<td>187.8</td>
<td>229.9</td>
<td>254.6</td>
<td>211.5</td>
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<td>Hydrogen Production, MMSCFD</td>
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<td>Partial Oxidation</td>
<td>142.4</td>
<td>157.9</td>
<td>132.3</td>
<td>181.8</td>
<td>190.6</td>
<td>139.2</td>
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<td>Steam Reforming</td>
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<td>135.6</td>
<td>55.5</td>
<td>48.1</td>
<td>64.0</td>
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<td>293.5</td>
<td>187.8</td>
<td>229.9</td>
<td>254.6</td>
<td>211.5</td>
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<tr>
<td>Utilities Purchased or Produced</td>
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<td>Power, MW</td>
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<td>221</td>
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<td>224</td>
<td>227</td>
<td>168</td>
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<td>Steam (600 psig) 1000 lb/hr</td>
<td>357</td>
<td>310</td>
<td>341</td>
<td>208</td>
<td>223</td>
<td>339</td>
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<td>Cooling water, 1000 GPM</td>
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<td>185</td>
<td>169</td>
<td>154</td>
<td>158</td>
<td>173</td>
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<td>Purchased Natural Gas, MMBTU/D</td>
<td>88</td>
<td>82</td>
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<td>52</td>
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<td>Inputs, MMBTU/Day</td>
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<td>454.3</td>
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<tr>
<td>Gasoline</td>
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<td>Diesel Fuel</td>
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<td>203.4</td>
<td>228.6</td>
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<td>Propane &amp; Butane</td>
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<td>30.4</td>
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<td>22.4</td>
<td>19.1</td>
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<td>6.4</td>
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<td>4.8</td>
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<td>Total</td>
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<td>307.9</td>
<td>353.5</td>
<td>315.1</td>
<td>295.9</td>
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<td>Thermal Efficiency, %</td>
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<td>77.8</td>
<td>76.4</td>
<td>80.3</td>
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32
Table 15. Liquefaction Plant Investment Details

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<th>Period 9 coal</th>
<th>Period 13 coal/oil/pl</th>
<th>Period 17 coal/plastic</th>
<th>Period 21 coal/ASR/p</th>
<th>Period 25 coal/oil</th>
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<tr>
<td>Pumps</td>
<td>24,282</td>
<td>24,331</td>
<td>23,483</td>
<td>24,924</td>
<td>21,800</td>
<td>18,693</td>
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<td>Reactors &amp; Hydrotreater</td>
<td>46,380</td>
<td>44,090</td>
<td>48,084</td>
<td>44,649</td>
<td>39,945</td>
<td>42,745</td>
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<td>Fired heaters</td>
<td>13,095</td>
<td>13,480</td>
<td>13,375</td>
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<td>12,289</td>
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<td>21,152</td>
<td>21,113</td>
<td>21,267</td>
<td>21,851</td>
<td>19,585</td>
<td>16,589</td>
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<td>Drums</td>
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<td>31,062</td>
<td>31,441</td>
<td>32,133</td>
<td>29,882</td>
<td>24,314</td>
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<td>Towers</td>
<td>8,609</td>
<td>8,685</td>
<td>8,442</td>
<td>8,826</td>
<td>8,051</td>
<td>6,378</td>
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<td>Compressors</td>
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<td>33,127</td>
<td>34,648</td>
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<td>15,277</td>
<td>17,436</td>
<td>16,491</td>
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<td>12,144</td>
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<td><strong>191,322</strong></td>
<td><strong>192,363</strong></td>
<td><strong>197,047</strong></td>
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<td><strong>154,837</strong></td>
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**Plant Investment, $MM**

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<th>Period 17</th>
<th>Period 21</th>
<th>Period 25</th>
</tr>
</thead>
<tbody>
<tr>
<td>Materials &amp; Equipment</td>
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<td>346.6</td>
<td>348.5</td>
<td>357.0</td>
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<td>280.5</td>
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<td>128.5</td>
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<tr>
<td><strong>Total</strong></td>
<td><strong>632.0</strong></td>
<td><strong>621.4</strong></td>
<td><strong>624.8</strong></td>
<td><strong>640.0</strong></td>
<td><strong>585.7</strong></td>
<td><strong>502.9</strong></td>
</tr>
</tbody>
</table>
### Table 16. Total Plant Investment Summary

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

<table>
<thead>
<tr>
<th></th>
<th>Period 4</th>
<th>Period 9</th>
<th>Period 13</th>
<th>Period 17</th>
<th>Period 21</th>
<th>Period 25</th>
</tr>
</thead>
<tbody>
<tr>
<td>coal</td>
<td>57.6</td>
<td>57.6</td>
<td>23.9</td>
<td>41.8</td>
<td>41.8</td>
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<tr>
<td>Oil Storage &amp; Handling</td>
<td>0.0</td>
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<td>0.0</td>
<td>29.9</td>
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<tr>
<td>Auto Shredder Residue Prep'n</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>17.2</td>
<td>0.0</td>
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<tr>
<td>Plastics Preparation</td>
<td>0.0</td>
<td>0.0</td>
<td>16.8</td>
<td>16.5</td>
<td>9.8</td>
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<tr>
<td>Liquefaction</td>
<td>632.0</td>
<td>621.4</td>
<td>624.8</td>
<td>640.0</td>
<td>585.7</td>
<td>502.9</td>
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<td>Hydrogen Manufacture</td>
<td>268.7</td>
<td>266.0</td>
<td>187.3</td>
<td>212.8</td>
<td>231.7</td>
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<td>Oxygen Plant</td>
<td>69.4</td>
<td>74.6</td>
<td>65.9</td>
<td>82.2</td>
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<td>68.4</td>
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<td>Treating</td>
<td>276.0</td>
<td>265.4</td>
<td>302.9</td>
<td>282.4</td>
<td>260.1</td>
<td>239.2</td>
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<td>Product Upgrading</td>
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<td>105.3</td>
<td>99.3</td>
<td>106.0</td>
<td>104.1</td>
<td>105.4</td>
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<td>Utilities</td>
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<td>309.0</td>
<td>315.2</td>
<td>285.9</td>
<td>289.5</td>
<td>311.6</td>
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<td>Tankage, Waste Handling</td>
<td>167.5</td>
<td>166.1</td>
<td>172.6</td>
<td>162.8</td>
<td>176.4</td>
<td>177.4</td>
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<td>General Offsites</td>
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<td>211.0</td>
<td>211.0</td>
<td>211.0</td>
<td>211.0</td>
<td>211.0</td>
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<tr>
<td>Subtotal</td>
<td>2,123.3</td>
<td>2,076.4</td>
<td>2,042.2</td>
<td>2,041.5</td>
<td>2,012.1</td>
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<td>Contingency &amp; Fee</td>
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<td>407.9</td>
<td>402.1</td>
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<td>Total Plant Investment</td>
<td>2,547.4</td>
<td>2,491.1</td>
<td>2,450.2</td>
<td>2,449.4</td>
<td>2,414.2</td>
<td>2,261.2</td>
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<tr>
<td>$/BPSD of Product</td>
<td>51,016</td>
<td>50,396</td>
<td>44,110</td>
<td>49,770</td>
<td>51,805</td>
<td>39,600</td>
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</table>
### Table 17. Product Cost Calculation

<table>
<thead>
<tr>
<th></th>
<th>Period 4</th>
<th>Period 9</th>
<th>Period 13</th>
<th>Period 17</th>
<th>Period 21</th>
<th>Period 25</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>coal</td>
<td>coal</td>
<td>coal/oil/pl</td>
<td>coal/plastic</td>
<td>coal/ASR/pl</td>
<td>coal/oil</td>
</tr>
<tr>
<td><strong>Operating Costs, $MM/yr</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Coal, as received ($20.50/Ton)</td>
<td>89.79</td>
<td>89.79</td>
<td>29.93</td>
<td>60.16</td>
<td>60.16</td>
<td>44.90</td>
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<tr>
<td>Oil ($8.00/B)</td>
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<td>0.00</td>
<td>58.46</td>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>Auto Shredder Residue (no cost)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Plastics (no cost)</td>
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<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>Natural Gas ($2.00/MMBTU)</td>
<td>57.82</td>
<td>53.58</td>
<td>32.57</td>
<td>34.43</td>
<td>32.45</td>
<td>34.48</td>
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<td>River Water ($0.10/Mgal)</td>
<td>0.30</td>
<td>0.29</td>
<td>0.29</td>
<td>0.27</td>
<td>0.27</td>
<td>0.29</td>
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<tr>
<td>Waste Disposal ($5.00/Ton)</td>
<td>2.47</td>
<td>2.45</td>
<td>1.04</td>
<td>1.97</td>
<td>6.01</td>
<td>1.16</td>
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<tr>
<td>Catalysts &amp; Chemicals</td>
<td>13.49</td>
<td>12.43</td>
<td>7.63</td>
<td>7.18</td>
<td>8.18</td>
<td>8.92</td>
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<tr>
<td>Dispersed Catalyst</td>
<td>6.35</td>
<td>6.35</td>
<td>6.35</td>
<td>6.35</td>
<td>6.35</td>
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<tr>
<td>Labor</td>
<td>23.07</td>
<td>23.07</td>
<td>23.71</td>
<td>23.39</td>
<td>23.71</td>
<td>23.39</td>
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<td>Capital-related costs</td>
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<td>373.43</td>
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<td><strong>Total</strong></td>
<td>598.06</td>
<td>584.61</td>
<td>554.65</td>
<td>524.75</td>
<td>522.32</td>
<td>575.99</td>
</tr>
<tr>
<td><strong>By-product Credits, $MM/yr</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Propane ($12.50/B)</td>
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<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>Butane ($14.50/B)</td>
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<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Sulfur ($52.00/Ton)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>Ammonia ($120.00/Ton)</td>
<td>0.00</td>
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<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td><strong>Net Product cost, $MM/year</strong></td>
<td>598.06</td>
<td>584.61</td>
<td>554.65</td>
<td>524.75</td>
<td>522.32</td>
<td>575.99</td>
</tr>
<tr>
<td>, $/B</td>
<td>34.44</td>
<td>34.07</td>
<td>28.19</td>
<td>30.08</td>
<td>31.81</td>
<td>29.03</td>
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<tr>
<td><strong>Equivalent Crude Oil Price, $/B</strong></td>
<td>28.38</td>
<td>28.02</td>
<td>22.43</td>
<td>24.20</td>
<td>25.83</td>
<td>23.21</td>
</tr>
</tbody>
</table>


Table 18. Breakdown of Equivalent Crude Oil Price

<table>
<thead>
<tr>
<th></th>
<th>Period 4</th>
<th>Period 9</th>
<th>Period 13</th>
<th>Period 17</th>
<th>Period 21</th>
<th>Period 25</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal</td>
<td>4.51</td>
<td>4.55</td>
<td>1.31</td>
<td>2.99</td>
<td>3.18</td>
<td>1.91</td>
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<tr>
<td>Oil</td>
<td>0.00</td>
<td>0.00</td>
<td>2.55</td>
<td>0.00</td>
<td>0.00</td>
<td>3.74</td>
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<tr>
<td>Auto Shredder Residue</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Plastics</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Natural Gas</td>
<td>2.90</td>
<td>2.71</td>
<td>1.42</td>
<td>1.71</td>
<td>1.71</td>
<td>1.47</td>
</tr>
<tr>
<td>River Water</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Waste Disposal</td>
<td>0.12</td>
<td>0.12</td>
<td>0.04</td>
<td>0.10</td>
<td>0.32</td>
<td>0.05</td>
</tr>
<tr>
<td>Catalysts &amp; Chemicals</td>
<td>0.68</td>
<td>0.63</td>
<td>0.33</td>
<td>0.36</td>
<td>0.43</td>
<td>0.38</td>
</tr>
<tr>
<td>Dispersed Catalyst</td>
<td>0.32</td>
<td>0.32</td>
<td>0.28</td>
<td>0.32</td>
<td>0.34</td>
<td>0.27</td>
</tr>
<tr>
<td>Labor</td>
<td>1.16</td>
<td>1.17</td>
<td>1.03</td>
<td>1.16</td>
<td>1.25</td>
<td>1.00</td>
</tr>
<tr>
<td>Maintenance</td>
<td>1.04</td>
<td>1.05</td>
<td>0.93</td>
<td>1.04</td>
<td>1.12</td>
<td>0.89</td>
</tr>
<tr>
<td>By-product credits</td>
<td>-1.66</td>
<td>-1.58</td>
<td>-1.76</td>
<td>-1.91</td>
<td>-1.77</td>
<td>-1.34</td>
</tr>
<tr>
<td>Equivalent Crude Oil Price, $/B</td>
<td>28.38</td>
<td>28.02</td>
<td>22.43</td>
<td>24.20</td>
<td>25.83</td>
<td>23.21</td>
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</tbody>
</table>
Table 19. Comparison of Cases at Maximum Reactor Throughput

<table>
<thead>
<tr>
<th></th>
<th>Period 4</th>
<th>Period 9</th>
<th>Period 13</th>
<th>Period 17</th>
<th>Period 21</th>
<th>Period 25</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Total Feed to Liquefaction, T/D</strong></td>
<td>12,000</td>
<td>12,000</td>
<td>12,000</td>
<td>12,000</td>
<td>12,940</td>
<td>15,188</td>
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<tr>
<td><strong>Number of Liquefaction Trains</strong></td>
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<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td><strong>Total Liquid Product, B/D</strong></td>
<td>49,933</td>
<td>49,431</td>
<td>55,547</td>
<td>49,214</td>
<td>50,432</td>
<td>72,220</td>
</tr>
<tr>
<td><strong>Liquefaction Plant Cost, $MM</strong></td>
<td>632.0</td>
<td>621.4</td>
<td>624.8</td>
<td>640.0</td>
<td>620.6</td>
<td>603.6</td>
</tr>
<tr>
<td><strong>Total Plant Investment, $MM</strong></td>
<td>2,547.4</td>
<td>2,491.1</td>
<td>2,450.2</td>
<td>2,449.4</td>
<td>2,550.8</td>
<td>2,702.6</td>
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<tr>
<td><strong>Net Operating Cost, $MM/year</strong></td>
<td>565.0</td>
<td>553.3</td>
<td>514.3</td>
<td>486.3</td>
<td>515.9</td>
<td>654.8</td>
</tr>
<tr>
<td><strong>, $/B</strong></td>
<td>34.44</td>
<td>34.07</td>
<td>28.19</td>
<td>30.08</td>
<td>31.14</td>
<td>27.60</td>
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<tr>
<td><strong>Equivalent Crude Oil Price, $/B</strong></td>
<td>28.38</td>
<td>28.02</td>
<td>22.43</td>
<td>24.20</td>
<td>25.20</td>
<td>21.89</td>
</tr>
</tbody>
</table>
Figure 1. Simplified Schematic of HTI's Bench Unit Configured for Run PB-05
Figure 2. PB-05: Daily Operating Conditions

![Graph showing Reactor Temperature and Kg Feed from Reactor Volume over Operating Days (Period Number).]

- ■ K-1 Temp
- ○ K-2 Temp
- ◆ Space Velocity

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Figure 4. PB-05: Feed Composition

![Diagram showing feed composition for different run conditions. The x-axis represents run conditions (1-5, 6-9, 10-13, 14-17, 18-21, 22-25), and the y-axis shows W% Dry Feed. Legend includes: Coal, ASR, Hondo Resid, and MSW Plastics.]
Figure 6. PB-05: C_4-524°C Distillate Yield and 524°C Residuum Yield
Figure 7. PB-05: Distillate Fraction Yield

Operating Conditions

Weight % Distillates

343 C+  177-343 C  C4-177 C
Figure 8. PB-05: Distillate Fraction Selectivity

![Distillate Fraction Selectivity Chart]

- 343 C+
- 177-343 C
- C4-177 C

Operating Conditions:
1 (1-5)
2 (6-9)
3 (10-13)
4 (14-17)
5 (18-21)
6 (22-25)
Figure 9. PB-05: Hydrogen Consumption and Light C\textsubscript{1}-C\textsubscript{3} Gas Yield
Figure 10. PB-05: Hydrogen Efficiency and C₁-C₃ Gas Selectivity
Figure 11. PB-05: Quality of SOH Distillates
Figure 13. PB-05: Solubility of PFL Product
Figure 14. PB-05: Effect of Feed Type vs. Process Performance
Figure 15. PB-05: Effect of Feed Type on Process Performance: Illinois No. 6 Coal vs. Black Thunder mine Coal
Figure 16. PB-05: Economics of Bituminous vs. Subbituminous Coals
APPENDIX [Daily Material Recovery Balance Data]

The material balance summary and the analysis of product gas streams are listed in appendix. The material balance summary lists all the input streams to the unit, including recycle, and all the output streams, along with the material recovery balance (page 1). The temperatures of various fractionators and separators and pressure filtration data are summarized on page 2. The hourly feed, recycle, and product stream rates are summarized on page 3, while the yield of product stream, based upon W% moisture-free feed, are listed on page 4. The GC analyses of vent and bottom gases is summarized on page 5. The PFL fed to the buffer includes the PFL material pumped to the ebullating pump hot checks. The normalized yields of individual light gases are also included in the appendix.
RUN 227-97 (POC P8-05) MATERIAL BALANCE

Illinois Coal-Waste Coprocessing using Dispersed Slurry Catalysts

FEED: Illinois No. 6 Crown II Mine: POC-01 Coal (HTI-6303)
Hondo VTB Oil and Waste Plastics
CATALYSTS: HTI'S Iron GelCat (1000 ppm Fe/100 ppm P)

<table>
<thead>
<tr>
<th>Number</th>
<th>01T</th>
<th>02T</th>
<th>03T</th>
<th>04T</th>
<th>05T</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Start of Period)</td>
<td>08/08/96</td>
<td>08/09/96</td>
<td>08/10/96</td>
<td>08/11/96</td>
<td>08/12/96</td>
</tr>
<tr>
<td>of Run (End of Period)</td>
<td>24.</td>
<td>48.</td>
<td>72.</td>
<td>96.</td>
<td>120.</td>
</tr>
</tbody>
</table>

**G, GRAMS**

| Total Feed | 27143.0 | 29305.0 | 33352.0 | 30621.0 | 36673.0 |
| Makeup Oil to Charge (L-814/extracted oil) | 0.0 | 803.0 | 4662.0 | 0.0 | 1182.0 |
| Makeup Oil to Buffer (L-814/extracted oil) | 0.0 | 5739.0 | 0.0 | 0.0 | 0.0 |
| Fl Recycled to Charge+K-2 Cat Addition | 22236.0 | 9416.0 | 10569.0 | 12235.0 | 13471.0 |
| AS Btms Recycled to Charge | 13607.0 | 13490.0 | 18121.0 | 18385.0 | 22020.0 |
| Filter Cake Recycled to Charge | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Fl to Stage 1 Buffer | 761.0 | 864.0 | 827.0 | 5041.0 | 2733.0 |
| Fl to Stage 2 Buffer | 744.0 | 846.0 | 1038.0 | 3307.0 | 833.0 |
| Water to Hot Separators | 10627.0 | 8778.0 | 10183.0 | 9998.0 | 9057.0 |
| Total Sulfur Added | 1016.3 | 976.2 | 995.0 | 920.0 | 1042.2 |
| Additive (Fe/P-Mo Catalysts) | 299.4 | 279.5 | 318.1 | 292.1 | 349.8 |
| 2 to 1st Stage | 4638.7 | 4643.9 | 4625.1 | 4637.6 | 4630.0 |
| 2 to 2nd Stage | 2057.7 | 2057.8 | 2057.8 | 2057.8 | 2058.8 |
| Hydrogen Bleed | 678.6 | 694.8 | 694.8 | 695.2 | 695.1 |
| TOTAL GRAMS IN | 83888.6 | 77892.1 | 87442.8 | 88190.6 | 94744.9 |

**TS, GRAMS**

| Hydrogen Out | 4351.4 | 4055.0 | 3717.2 | 3806.5 | 3654.4 |
| Total Gas Product (N2,H2 Free) | 3303.4 | 4295.2 | 4316.5 | 4483.9 | 5037.2 |
| Hot Knocksouts | 37.0 | 65.0 | 41.0 | 44.0 | 52.0 |
| Separator Overhead (HTU) Product | 22178.0 | 20888.0 | 24696.0 | 26135.0 | 24450.0 |
| Atmospheric Overhead Product (Sample) | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| AS Bottoms | 50084.0 | 35269.0 | 48241.0 | 54675.0 | 49359.0 |
| Feed + Interstage Slurry Sample | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| TOTAL GRAMS OUT | 79961.9 | 65572.1 | 81011.6 | 89144.4 | 82552.5 |
| Total Material Recovery (Gross) | 95.41 | 84.18 | 92.65 | 101.08 | 87.13 |
### UNIT MATERIAL BALANCE (CON’T)

#### CHARGE, PRODUCT, AND RECYCLE RATES

#### D RATES, GRAMS/HOUR

<table>
<thead>
<tr>
<th></th>
<th>01T</th>
<th>02T</th>
<th>03T</th>
<th>04T</th>
<th>05T</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbonaceous Feed</td>
<td>1131.0</td>
<td>1221.0</td>
<td>1389.7</td>
<td>1275.9</td>
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<td>Makeup Oil Rate</td>
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<td>1179.6</td>
<td>1342.6</td>
<td>1232.6</td>
<td>1476.2</td>
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<td>Water to Hot Separator</td>
<td>272.6</td>
<td>194.3</td>
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<tr>
<td>2 to 1st Stage</td>
<td>424.3</td>
<td>416.6</td>
<td>377.4</td>
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<tr>
<td>2 to 2nd Stage</td>
<td>193.3</td>
<td>193.5</td>
<td>192.7</td>
<td>193.2</td>
<td>192.9</td>
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<tr>
<td>2 to 1st Stage Buffer</td>
<td>85.7</td>
<td>85.7</td>
<td>85.7</td>
<td>85.8</td>
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#### CYCLE RATES TO REACTOR, GRAMS/HOUR

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<tbody>
<tr>
<td>FL Recycled to Slurry + Pretreater Buffer</td>
<td>926.5</td>
<td>392.3</td>
<td>440.4</td>
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<td>AS Bottoms Recycled</td>
<td>567.0</td>
<td>562.1</td>
<td>755.0</td>
<td>766.0</td>
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<td>2 to 1st Stage Buffer</td>
<td>31.7</td>
<td>36.0</td>
<td>34.5</td>
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<td>113.9</td>
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<td>2 to 2nd Stage Buffer</td>
<td>31.0</td>
<td>35.3</td>
<td>43.3</td>
<td>137.8</td>
<td>34.7</td>
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#### COLLECTED PRODUCTS (INCLUDING SAMPLES), GRAMS/HOUR

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<tr>
<td>Total Gas (incl. N2)</td>
<td>356.4</td>
<td>362.6</td>
<td>347.1</td>
<td>358.1</td>
<td>354.1</td>
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<tr>
<td>(N2 free)</td>
<td>319.3</td>
<td>347.9</td>
<td>334.7</td>
<td>345.4</td>
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<tr>
<td>OH</td>
<td>388.5</td>
<td>406.3</td>
<td>502.4</td>
<td>550.7</td>
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<tr>
<td>SOH-H2O</td>
<td>535.6</td>
<td>464.0</td>
<td>526.6</td>
<td>538.3</td>
<td>547.3</td>
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<tr>
<td>OH-NET WATER</td>
<td>92.8</td>
<td>98.3</td>
<td>102.3</td>
<td>121.7</td>
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<td>Knockouts</td>
<td>1.5</td>
<td>2.7</td>
<td>1.7</td>
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<tr>
<td>Filter Cake</td>
<td>32.0</td>
<td>77.7</td>
<td>204.3</td>
<td>206.2</td>
<td>204.2</td>
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<tr>
<td>Filter Liquid</td>
<td>210.6</td>
<td>71.8</td>
<td>450.3</td>
<td>290.7</td>
<td>75.9</td>
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<tr>
<td>SOH + KO</td>
<td>1.5</td>
<td>2.7</td>
<td>1.7</td>
<td>1.8</td>
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<tr>
<td>Total CAS Bottoms</td>
<td>2086.8</td>
<td>1511.2</td>
<td>2010.0</td>
<td>2278.1</td>
<td>2056.6</td>
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<tr>
<td>Reactor 1 Liquid Sample</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
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<tr>
<td>Separator Bottoms to CAS</td>
<td>2086.8</td>
<td>1511.2</td>
<td>2010.0</td>
<td>2278.1</td>
<td>2056.6</td>
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<tr>
<td>Total Asoh</td>
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<td>2.7</td>
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<tr>
<td>AS Bottoms to Pressure Filter</td>
<td>1231.8</td>
<td>613.0</td>
<td>1172.6</td>
<td>1354.5</td>
<td>990.0</td>
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<tr>
<td>Total Filter Cake</td>
<td>32.0</td>
<td>77.7</td>
<td>204.3</td>
<td>206.2</td>
<td>204.2</td>
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<tr>
<td>Total Filter Liquid</td>
<td>1199.8</td>
<td>535.3</td>
<td>968.4</td>
<td>1148.3</td>
<td>785.8</td>
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#### PRESSURE FILTER

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<tr>
<td>Vg Duration, min</td>
<td>29564.0</td>
<td>14713.0</td>
<td>28143.0</td>
<td>32508.0</td>
<td>23759.0</td>
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<tr>
<td>Vg Pressure, psig</td>
<td>769.0</td>
<td>1865.0</td>
<td>4902.0</td>
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<td>4900.0</td>
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<tr>
<td>Vg Final Top Temp, deg-f</td>
<td>28101.0</td>
<td>12397.0</td>
<td>22422.0</td>
<td>26601.0</td>
<td>18096.0</td>
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<tr>
<td>Pressure Filter Charge, g</td>
<td>694.0</td>
<td>451.0</td>
<td>819.0</td>
<td>958.0</td>
<td>763.0</td>
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<tr>
<td>Pressure Filter Loss, g</td>
<td>2.60</td>
<td>12.68</td>
<td>17.42</td>
<td>15.22</td>
<td>20.62</td>
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### UNIT MATERIAL BALANCE (CON’T)  

**GAS RATES:**

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<td>2 to 1st Stage, scfh</td>
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<td>2 to 1st Stage Recycle, scfh</td>
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<td>40.1</td>
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<td>40.1</td>
<td>40.1</td>
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<tr>
<td>2 to 2nd Stage, scfh</td>
<td>35.6</td>
<td>35.6</td>
<td>35.6</td>
<td>35.6</td>
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<tr>
<td>Feed H2, scfh</td>
<td>11.7</td>
<td>12.0</td>
<td>12.0</td>
<td>12.0</td>
<td>12.0</td>
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<tr>
<td>Vent Gas, Scfh (N2-Free)</td>
<td>79.3</td>
<td>75.1</td>
<td>69.4</td>
<td>70.9</td>
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**NET ADJ. PRODUCTS, % DRY COAL:**

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</thead>
<tbody>
<tr>
<td>CO + CO2</td>
<td>1.16</td>
<td>1.43</td>
<td>1.27</td>
<td>1.48</td>
<td>1.76</td>
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<tr>
<td>Total C1-C3</td>
<td>6.12</td>
<td>7.02</td>
<td>6.74</td>
<td>6.87</td>
<td>6.91</td>
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<td>Total C4-C7</td>
<td>2.26</td>
<td>2.27</td>
<td>1.61</td>
<td>2.22</td>
<td>2.06</td>
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<tr>
<td>STH + KO Water</td>
<td>8.49</td>
<td>8.33</td>
<td>7.62</td>
<td>9.87</td>
<td>11.51</td>
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<tr>
<td>STH + KO Oil</td>
<td>35.56</td>
<td>34.44</td>
<td>37.42</td>
<td>44.68</td>
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<tr>
<td>F</td>
<td>44.95</td>
<td>30.96</td>
<td>38.61</td>
<td>34.42</td>
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<tr>
<td>Fs</td>
<td>3.62</td>
<td>10.20</td>
<td>16.28</td>
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<td>AS Bottoms</td>
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<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>Reactor 1 Liquid Sample</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
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**HYDROTREATIER OPERATION:**

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<tbody>
<tr>
<td>Hydrotreater Top Temp, F</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Hydrotreater Middle Temp, F</td>
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<td></td>
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<tr>
<td>Hydrotreater Bottom Temp, F</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Hydrotreater DP, psi</td>
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**OPERATING CONDITIONS:**

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<tbody>
<tr>
<td>1st Stage Est Reaction Temp, F</td>
<td>805</td>
<td>835</td>
<td>833</td>
<td>831</td>
<td>834</td>
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<tr>
<td>2nd Stage Est Reaction Temp, F</td>
<td>810</td>
<td>854</td>
<td>854</td>
<td>853</td>
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<tr>
<td>Charge Pot Internal Temp, F</td>
<td>199</td>
<td>211</td>
<td>245</td>
<td>283</td>
<td>314</td>
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<tr>
<td>1st Separator Liquid (Stage I) Temp, F</td>
<td>654</td>
<td>648</td>
<td>651</td>
<td>659</td>
<td>652</td>
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<tr>
<td>2nd Separator Liquid (Stage II) Temp, F</td>
<td>106</td>
<td>105</td>
<td>105</td>
<td>102</td>
<td>101</td>
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<td>Unit Back Pressure, psig</td>
<td>2509</td>
<td>2509</td>
<td>2504</td>
<td>2511</td>
<td>2516</td>
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<td>1st Stage Reactor DP, psi</td>
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<td>5.5</td>
<td>7.5</td>
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<td>2nd Stage Reactor DP, psi</td>
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<td>3.1</td>
<td>3.2</td>
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<td>3.6</td>
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<tr>
<td>1st Stage Internal Recycle Rate, cc/hr</td>
<td>47640</td>
<td>47640</td>
<td>47640</td>
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<tr>
<td>2nd Stage Internal Recycle Rate, cc/hr</td>
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<td>47640</td>
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<tr>
<td>1st Stage Dry Coal SV, Lb Feed/Hr/Ft3</td>
<td>34.1</td>
<td>36.8</td>
<td>41.9</td>
<td>38.5</td>
<td>46.1</td>
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<tr>
<td>2nd Stage Dry coal SV, Lb Feed/Hr/Ft3</td>
<td>34.1</td>
<td>36.8</td>
<td>41.9</td>
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<td>46.1</td>
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<td>1st Stage Space Velocity, Lb/Hr/Ft3</td>
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<tr>
<td>1st Stage Slurry Residence Time, min</td>
<td>34.6</td>
<td>37.1</td>
<td>32.8</td>
<td>33.3</td>
<td>29.1</td>
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<tr>
<td>2nd Stage Slurry Residence Time, min</td>
<td>34.2</td>
<td>36.5</td>
<td>32.2</td>
<td>31.7</td>
<td>28.7</td>
</tr>
</tbody>
</table>

**Moisture in Coal, %:**

|       | 4.30 | 4.18 | 4.18 | 4.18 | 4.18 |

**Total Dry Coal Fed to Date, g:**

|       | 26223. | 53675. | 85867. | 115450. | 150880. |

**2 Consumed--Meter, % Dry Coal:**

|       | 11.5  | 11.8  | 11.4  | 12.1  | 10.5  |
### Illinois Coal-Waste Coprocessing using Dispersed Slurry Catalysts

**FEED:** Illinois No. 6 Crown II Mine: POC-01 COAL (HTI-6303)

**Hondo VTB Oil and Waste Plastics**

**CATALYSTS:** HTI'S Iron GelCat (1000 ppm Fe/100 ppm P)

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<th>07T</th>
<th>08T</th>
<th>09T</th>
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<tbody>
<tr>
<td>(Start of Period)</td>
<td>08/13/96</td>
<td>08/14/96</td>
<td>08/15/96</td>
<td>08/16/96</td>
<td>08/17/96</td>
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<tr>
<td>of Run</td>
<td>144.</td>
<td>168.</td>
<td>192.</td>
<td>216.</td>
<td>240.</td>
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<tr>
<td><strong>Total Feed</strong></td>
<td>27766.0</td>
<td>32377.0</td>
<td>32452.0</td>
<td>33588.0</td>
<td>27164.0</td>
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<td><strong>Makeup Oil to Charge (L-814/extracted oil)</strong></td>
<td>7307.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>1168.0</td>
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<tr>
<td><strong>Makeup Oil to Buffer (L-814/extracted oil)</strong></td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td><strong>1 Recycled to Charge &amp; 2 Cat Addition</strong></td>
<td>10328.0</td>
<td>12936.0</td>
<td>12967.0</td>
<td>13421.0</td>
<td>9848.0</td>
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<tr>
<td><strong>Slts Recycled to Charge</strong></td>
<td>16672.0</td>
<td>19440.0</td>
<td>19485.0</td>
<td>20168.0</td>
<td>16149.0</td>
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<tr>
<td><strong>Filter Cake Recycled to Charge</strong></td>
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<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
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<tr>
<td><strong>Fl to Stage 1 Buffer</strong></td>
<td>1136.0</td>
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<td>725.0</td>
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<td><strong>Fl to Stage 2 Buffer</strong></td>
<td>846.0</td>
<td>776.0</td>
<td>740.0</td>
<td>1070.0</td>
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<td><strong>Water to Hot Separators</strong></td>
<td>8863.0</td>
<td>9860.0</td>
<td>9560.0</td>
<td>9717.0</td>
<td>9432.0</td>
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<tr>
<td><strong>Total Sulfur Added</strong></td>
<td>969.0</td>
<td>938.5</td>
<td>938.5</td>
<td>957.4</td>
<td>860.7</td>
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<tr>
<td><strong>Additive (Fe/P+Mo Catalysts)</strong></td>
<td>269.4</td>
<td>314.2</td>
<td>314.9</td>
<td>325.9</td>
<td>263.4</td>
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<tr>
<td><strong>2 to 1st Stage</strong></td>
<td>4636.7</td>
<td>4641.3</td>
<td>4643.3</td>
<td>4642.7</td>
<td>4644.0</td>
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<tr>
<td><strong>2 to 2nd Stage</strong></td>
<td>2058.9</td>
<td>2058.9</td>
<td>2057.8</td>
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<tr>
<td><strong>Hydrogen Bleed</strong></td>
<td>695.3</td>
<td>695.2</td>
<td>695.2</td>
<td>695.2</td>
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<tr>
<td><strong>TOTAL GRAMS IN</strong></td>
<td>81547.3</td>
<td>84882.1</td>
<td>84578.7</td>
<td>87936.7</td>
<td>75298.6</td>
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<tbody>
<tr>
<td><strong>Hydrogen Out</strong></td>
<td>3425.9</td>
<td>3399.4</td>
<td>3416.0</td>
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<tr>
<td><strong>Total Gas Product (N2,H2 Free)</strong></td>
<td>12616.0</td>
<td>9421.0</td>
<td>9997.1</td>
<td>9583.1</td>
<td>6488.0</td>
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<tr>
<td><strong>Nit Knockouts</strong></td>
<td>37.0</td>
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<td>61.0</td>
<td>42.0</td>
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<tr>
<td><strong>Separator Overhead (HTU) Product</strong></td>
<td>21807.0</td>
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<td><strong>Atmospheric Overhead Product (Sample)</strong></td>
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<tr>
<td><strong>AS Bottoms</strong></td>
<td>43327.0</td>
<td>45195.0</td>
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**Total Material Recovery (Gross)** | 100.07 | 92.62 | 94.24 | 98.12 | 100.75
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<td>188.5</td>
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<td>% Pfs</td>
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GAS RATES:-----

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NET ADJ. PRODUCTS, W% DRY COAL -----

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HYDROTREATHER OPERATION-----

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<td>Hydrotreater DP, Psi</td>
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OPERATING CONDITIONS-----

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# Illinois Coal-Waste Coprocessing using Dispersed Slurry Catalysts

**FEED:** Illinois No. 6 Crown II Mine: POC-01 Coal (HTI-6303)

**Catalysts:** HTI's Iron GelCat (1000 ppm Fe/100 ppm P)

## Material Balance

### Illinois Coal-Uaste Coprocessing using Dispersed Slurry Catalysts

#### FEED:
- Illinois No. 6 Crown II Mine: POC-01 Coal (HTI-6303)
- Hondo VTB Oil and Waste Plastics

#### CATALYSTS:
- HTI's Iron GelCat (1000 ppm Fe/100 ppm P)

### Table: Material Balance

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### GRAMS

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### Total Material Recovery (Gross)

<p>| Source                  | 101.00    | 94.92     | 94.48     | 100.75    | 91.74     |</p>
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<td><strong>CHARGE, PRODUCT, AND RECYCLE RATES-----</strong></td>
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<td>Hydrotreater DP, Psi</td>
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# Illinois Coal-Waste Coprocessing using Dispersed Slurry Catalysts

**FEED:** Illinois No. 6 Crown II Mine: POC-01 COAL (HT1-6303)

**Waste:** Hondo VTB Oil and Waste Plastics

**CATALYSTS:** HT1's Iron GelCat (1000 ppm Fe/100 ppm P)

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<td>of Run (End of Period)</td>
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**TOTAL GRAMS IN**

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<td>Additive (Fe/Mo Catalysts)</td>
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<td>L to 1st Stage</td>
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**TOTAL GRAMS OUT**

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**Total Material Recovery (Gross)**

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<td>8.91</td>
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**UNIT MATERIAL BALANCE (CON'T)**

**ARGE, PRODUCT, AND RECYCLE RATES-----

**RATES, GRAMS/HOUR**

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<td>Carbonaceous Feed</td>
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<td>Makeup Oil Rate</td>
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<td>417.7</td>
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<td>Recycled to 2nd Stage</td>
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**CLE RATES TO REACTOR, GRAMS/HOUR**

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<td>Recycled to Slurry + Pretreater Buffer</td>
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<td>663.9</td>
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**COLLECTED PRODUCTS (INCLUDING SAMPLES), GRAMS/HOUR**

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<tr>
<td>Gas (incl. N2) (N2 free)</td>
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<td>264.1</td>
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<td>Knockouts</td>
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<td>SOH-H2O</td>
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<td>H-Net W2O</td>
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<td>80.2</td>
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<td>Filter Cake</td>
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<td>124.0</td>
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<td>167.9</td>
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<td>Filter Liquid</td>
<td>132.0</td>
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<td>Ash + KO</td>
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<tr>
<td>Filter Cake</td>
<td>2013.5</td>
<td>2237.0</td>
<td>2252.0</td>
<td>2262.4</td>
<td>2067.3</td>
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<tr>
<td>Filter Liquid</td>
<td>2013.5</td>
<td>2237.0</td>
<td>2252.0</td>
<td>2262.4</td>
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<tr>
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<td>2.4</td>
<td>2.9</td>
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**SURE FILTER**

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<td>g Duration, min</td>
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<td>Pressure, psig</td>
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<td>2975.0</td>
<td>4294.0</td>
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<td>3084.0</td>
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<td>g Final Top Temp, deg-f</td>
<td>20794.0</td>
<td>21544.0</td>
<td>26285.0</td>
<td>23947.0</td>
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<td>g Filter Charge</td>
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<td>939.0</td>
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<td>1083.0</td>
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<td>g Filter Loss</td>
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<td>11.69</td>
<td>13.49</td>
<td>13.86</td>
<td>12.52</td>
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**UNIT MATERIAL BALANCE (CON'T)**

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<tr>
<td>2 to 1st Stage, scfh</td>
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<td>40.1</td>
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<td>2 to 2nd Stage, scfh</td>
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<td>35.6</td>
<td>35.6</td>
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<td>Feed H2, scfh</td>
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<td>12.0</td>
<td>12.0</td>
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<td>Vent Gas, Scfh (N2-Free)</td>
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<td>72.4</td>
<td>72.2</td>
<td>72.1</td>
<td>73.6</td>
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</table>

**NET ADJ. PRODUCTS, W% DRY COAL -----**

| Total CO + CO2 | 0.45 | 0.56 | 0.66 | 0.61 | 0.89 |
| Total C1-C3 | 2.99 | 3.27 | 4.22 | 3.95 | 5.31 |
| Total C4-C7 | 1.59 | 1.56 | 1.29 | 1.18 | 1.70 |
| SOH + KO) Water | 0.46 | 4.96 | 5.24 | 5.59 | 4.82 |
| SOH + KO) Oil | 43.17 | 43.54 | 41.54 | 32.72 | 44.64 |
| Fl | 2.78 | 27.51 | 36.89 | 46.44 | 49.97 |
| Fs | 8.64 | 10.85 | 11.18 | 10.07 | 12.69 |
| AS Bottoms | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| Reactor 1 Liquid Sample | 0.00 | 0.00 | 0.92 | 0.00 | 0.00 |

**HYDROTREATER OPERATION-----**

| Hydrotreater Top Temp, F |     |     |     |     |     |
| Hydrotreater Middle Temp, F |     |     |     |     |     |
| Hydrotreater Bottom Temp, F |     |     |     |     |     |
| Hydrotreater DP, Psi |     |     |     |     |     |

**OPERATING CONDITIONS-----**

| 1st Stage Est Reaction Temp, F | 837. | 836. | 836. | 837. | 834. |
| 2nd Stage Est Reaction Temp, F | 854. | 857. | 856. | 855. | 856. |
| Charge Pot Internal Temp, F | 476. | 477. | 477. | 477. | 472. |
| 1st Separator Liquid (Stage II) Temp, F | 671. | 671. | 672. | 671. | 664. |
| 2nd Separator Liquid (Stage I) Temp, F | 114. | 110. | 110. | 110. | 106. |
| Unit Back Pressure, psig | 2495. | 2495. | 2496. | 2496. | 2488. |
| 1st Stage Reactor DP, psi | 11.9 | 11.8 | 11.5 | 11.7 | 11.6 |
| 2nd Stage Reactor DP, psi | 2.5 | 2.6 | 2.6 | 2.7 | 3.0 |

| 1st Stage Internal Recycle Rate, cc/hr | 47640. | 47640. | 47640. | 52933. | 78077. |
| 2nd Stage Internal Recycle Rate, cc/hr | 47640. | 47640. | 47640. | 47640. | 47640. |
| 1st Stage Dry Coal SV, Lb Feed/Hr/Ft3 | 45.0 | 41.7 | 47.7 | 54.9 | 41.0 |
| 2nd Stage Dry coal SV, Lb Feed/Hr/Ft3 | 45.0 | 41.7 | 47.7 | 54.9 | 41.0 |
| Wt Stage Space Velocity, Lb/Hr/Ft3 | 22.5 | 20.9 | 23.9 | 27.5 | 20.5 |
| 1st Stage Slurry Residence Time, min | 36.8 | 39.6 | 39.8 | 40.1 | 50.1 |
| 2nd Stage Slurry Residence Time, min | 36.5 | 39.2 | 39.1 | 39.6 | 44.3 |

| Moisture in Coal, W% | 4.21 | 4.19 | 4.08 | 3.99 | 3.94 |
| Total Dry Coal Fed to Date, g | 477193. | 509279. | 545990. | 588224. | 619780. |
| 2 Consumed--Meter, W% Dry Coal | 9.7 | 10.5 | 9.3 | 8.1 | 10.6 |
### Illinois Coal-Waste Coprocessing using Dispersed Slurry Catalysts

**FEED:** Illinois No. 6 Crown II Mine: POC-01 COAL (HTI-6303)

**CATALYSTS:** HTI's Iron GelCat (1000 ppm Fe/100 ppm P)

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<tr>
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<tr>
<td>Total Gas Product (N2,H2 Free)</td>
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<td>4213.8</td>
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<td>3896.3</td>
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<td>2858.5</td>
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<td>3099.8</td>
</tr>
<tr>
<td>Knockouts</td>
</tr>
<tr>
<td>118.0</td>
</tr>
<tr>
<td>39.0</td>
</tr>
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<td>20.0</td>
</tr>
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<td>7.0</td>
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<tr>
<td>9.0</td>
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<td>Distillates Overhead (HTU) Product</td>
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<td>26443.0</td>
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<td>Atmospheric Overhead Product (Sample)</td>
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<td>TOTAL GRAMS OUT</td>
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<td>84449.2</td>
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<td>63841.9</td>
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<td>54095.8</td>
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**Total Material Recovery (Gross):**

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### Material Balance (Con't)

#### Charge, Product, and Recycle Rates

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<td>Total Makeup Oil Rate</td>
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<td>Solvent Makeup Oil</td>
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<td>to 1st Stage</td>
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<td>193.5</td>
<td>193.5</td>
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<td>85.8</td>
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#### Recycle Rates to Reactor, Grams/Hour

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<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
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<tr>
<td>Bottoms Recycled</td>
<td>861.5</td>
<td>367.2</td>
<td>331.1</td>
<td>302.1</td>
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<td>34.1</td>
<td>40.2</td>
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#### Collected Products (Including Samples), Grams/Hour

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<td>Total Gas (incl. N2)</td>
<td>346.9</td>
<td>329.9</td>
<td>321.7</td>
<td>275.4</td>
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<td>N (N2 free)</td>
<td>348.6</td>
<td>341.3</td>
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<td>288.7</td>
<td>310.5</td>
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<tr>
<td>H</td>
<td>603.8</td>
<td>554.8</td>
<td>614.6</td>
<td>654.8</td>
<td>620.5</td>
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<td>SOH+H2O</td>
<td>452.5</td>
<td>467.7</td>
<td>465.1</td>
<td>447.0</td>
<td>458.8</td>
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<td>H-Net WATER</td>
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<td>65.8</td>
<td>58.8</td>
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<tr>
<td>Knockouts</td>
<td>4.9</td>
<td>1.6</td>
<td>0.8</td>
<td>0.3</td>
<td>0.4</td>
</tr>
<tr>
<td>Filter Cake</td>
<td>223.5</td>
<td>227.7</td>
<td>140.8</td>
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<td>Filter Liquid</td>
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<td>OH + KO</td>
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<td>0.4</td>
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<tr>
<td>Total CAS Bottoms</td>
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<td>1254.0</td>
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<td>Reactor 1 Liquid Sample</td>
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<td>Parator Bottoms to CAS</td>
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<tr>
<td>Total Asoh</td>
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<td>Bottoms to Pressure Filter</td>
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<tr>
<td>Total Filter Cake</td>
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<td>227.7</td>
<td>140.8</td>
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<td>60.2</td>
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<tr>
<td>Total Filter Liquid</td>
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<td>1088.9</td>
<td>724.2</td>
<td>411.2</td>
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#### Pressure Filter

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<tbody>
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<td>g Duration, min</td>
<td>15.00</td>
<td>17.29</td>
<td>16.28</td>
<td>19.44</td>
<td>17.41</td>
</tr>
<tr>
<td>g Pressure, psig</td>
<td>35745.0</td>
<td>31597.0</td>
<td>20760.0</td>
<td>12251.0</td>
<td>8295.3</td>
</tr>
<tr>
<td>g Final Top Temp, deg-f</td>
<td>3563.0</td>
<td>5464.0</td>
<td>3380.0</td>
<td>2382.0</td>
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<tr>
<td>Pressure Filter Liquid, g</td>
<td>29050.0</td>
<td>24862.0</td>
<td>16105.0</td>
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<td>Pressure Filter Loss, g</td>
<td>1332.0</td>
<td>1271.0</td>
<td>1275.0</td>
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<td>782.0</td>
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<tr>
<td>Pfs</td>
<td>15.00</td>
<td>17.29</td>
<td>16.28</td>
<td>19.44</td>
<td>17.41</td>
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GAS RATES:-----

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<tr>
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<td>40.2</td>
<td>40.2</td>
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<tr>
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<td>2nd Stage scfh</td>
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<td>Feed H2, scfh</td>
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<td>12.0</td>
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<td>Recycle Gas, Scfh (N2-Free)</td>
<td>69.8</td>
<td>73.3</td>
<td>71.8</td>
<td>73.4</td>
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NET ADJ. PRODUCTS, W% DRY COAL -----

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<tr>
<td>CO + CO2</td>
<td>1.01</td>
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<td>0.54</td>
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<td>0.60</td>
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<tr>
<td>C1-C3</td>
<td>6.69</td>
<td>4.36</td>
<td>3.97</td>
<td>3.42</td>
<td>4.88</td>
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<tr>
<td>C4-C7</td>
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<td>2.02</td>
<td>1.56</td>
<td>2.16</td>
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<tr>
<td>OH + KO Water</td>
<td>3.62</td>
<td>3.71</td>
<td>3.68</td>
<td>3.64</td>
<td>4.51</td>
</tr>
<tr>
<td>OH + KO Oil</td>
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<td>31.30</td>
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<tr>
<td>H1</td>
<td>40.94</td>
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<td>Reactor 1 Liquid Sample</td>
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HYDROTREATER OPERATION-----

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<tbody>
<tr>
<td>Hydrotreater Top Temp, F</td>
<td>838.</td>
<td>834.</td>
<td>833.</td>
<td>835.</td>
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<td>Hydrotreater Middle Temp, F</td>
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<td>854.</td>
<td>854.</td>
<td>854.</td>
<td>854.</td>
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<td>661.</td>
<td>650.</td>
<td>642.</td>
<td>620.</td>
<td>607.</td>
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<td>Hit Back Pressure, psig</td>
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<td>Hit Back Pressure, psig</td>
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OPERATING CONDITIONS-----

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<td>59.8</td>
<td>65.9</td>
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</table>

Moisture in Coal, W%            | 4.00| 4.19| 4.19| 4.19| 4.19|

Total Dry Coal Fed to Date, g   | 655756. | 698295. | 736653. | 771649. | 800105. |

2 Consumed--Meter, W% Dry Coal  | 10.1 | 8.0 | 9.1 | 9.5 | 11.8 |
### BLACK THUNDER MINE-POC-02 COAL (HRI 6213)

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**BLACK THUNDER MINE-POC-02 COAL (HRI 6213)**

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