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# DIRECT LIQUEFACTION PROOF-OF-CONCEPT PROGRAM

## Topical Report #12
## Bench Run PB-10 (HTI 227-109)

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## *EXECUTIVE SUMMARY*

This report presents the results of the bench-scale test PB-10 (HTI Run No. 227-109), performed at HTI's facilities under DOE contract. The objective of this test was to demonstrate the feasibility of direct coal liquefaction on Shangwan coal utilizing various backend processing and recycle schemes. This bench test continues the work that was started in PDU testing 260-007. Additionally, this test collected all available data to allow for the best scale-up process design possible from this particular unit. Previous bench test (227-106, PB-09) was performed on different seams of Chinese coal (Shenhua Coal #2 & #3).

HTI's continuous Bench Scale Unit No. 227 was used for this run, spanning 24 days including start-up and shutdown. This unit employs two backmixed liquefaction reactors and a closely integrated fixed-bed hydrotreater. The coal/oil slurry is premixed offline and charged to a feed tank on a periodic basis. The reactors are close-coupled with the effluent from the first stage flowing directly to the second stage. The effluent from the second reactor was separated in a hot separator and the bottoms stream was sent to an atmospheric flash. The overhead from the 2nd stage hot separator was sent directly to the in-line hydrotreater. The hydrotreater outlet was sent to a cold separator and separated into a vent gas stream and a separator overhead stream (SOH). The vent gases were metered, sampled, and sent to flare. The overhead from the atmospheric flash was also sent to the hydrotreater as part of the feed charge. The bottoms stream from the atmospheric flash was separated offline by various processes depending on the particular recycle scheme being used for that condition. These streams were then analyzed. Part of the mid-distillate generated is used as a process oil in the buffer pumps for the first and second stage reactors. TNPS was used during startup to pre-sulfide the hydrotreater catalyst and TNPS and $H_2S$ were used during the run to activate the dispersed catalyst. During this test four different recycle schemes were used.

HTI's proprietary iron-based dispersed catalyst GelCat™ in different formulations were used in the entire run. The run includes 8 separate operating conditions with four different recycle schemes. Condition 1A (Periods 1-3A) used the base coal, Shangwan coal, which was used for the PDU testing (260-007). Part of the O-6 bottoms was recycled and the rest separated by pressure filtration. The pressure filter liquids were vacuum distilled and the bottoms recycled as well as a portion of the overheads. The pressure filter solids was solvent extracted and the toluene extracted oil was recycled while the toluene extracted solids was taken as a product. For Condition 1B this was changed to decreasing the total amount of O-6 bottoms recycled, recycling the pressure filter liquid directly and recycling the toluene extracted oil. For Condition 2A the same scheme was followed except the coal was changed from that used in the PDU program to L-1007 which was a smaller lot of the same coal that was ground by HTI. For Condition 2B the recycle of the pressure filter liquids was stopped and the vacuum still bottoms recycle was restarted. For Condition 3A a portion of the pressure filter solids was also recycled so as to resume solids recycle and the feed coal was changed back to that used in the PDU program. For Condition 3B the hydrotreater was bypassed to determine the quality and quantity of material flowing directly from the overhead of the hot separator to the hydrotreater. For Condition 4A the hydrotreater was placed back online and the O-6 bottoms was vacuum distilled twice recycling a 399°C+ fraction and a 399-538°C+ fraction. For Condition 4B the cut point of the first distillation was reduced and the overall recycle ratio was changed from 1.6 to 1.25.

The small lot of coal that was prepared by HTI does result in a slightly higher coal conversion of 1.8 W% maf ff than the bulk coal sample used for the PDU testing. The reactor performance as measured by reactor (single-pass) resid conversion shows some variation based on the recycle scheme being used. This follows the pattern that the greater the recycle of resid material the lower the reactor performance. This is due to the recycle resid becoming more concentrated in refractory materials which are very difficult or impossible to convert. The process performance, as measured by the process (overall) resid conversion, shows a very strong impact from the recycle scheme. Even though the reactor resid conversion decreases with higher levels of resid recycle, the process resid conversion increases sharply as recycle ratio increases. The maximum process performance is achieved with the maximum recycle of resid. The performance achieved for the design basis 93.5 W% maf ff coal conversion, 85.9 W% maf ff resid conversion, and 67.2 W% $C_4$-524°C distillate yield with a hydrogen consumption of 8.8 W% maf ff. The hydrotreater had a significant impact on final product quality; increasing the hydrogen content by 1.8 W%, increasing the H/C Atomic ratio from 1.48 to 1.85, increasing the API gravity from 21.3 to 36.0 and lowering the sulfur content from 1119 wppm to 59 wppm and the nitrogen content from 695 wppm to <1 wppm.

## BACKGROUND

This report presents the results of the bench-scale test, PB-10, performed at HTI's facilities under DOE contract (HTI Run No. 227-109). This bench test continues the work that was started in PDU testing 260-007. Previous bench test (PB-09, HTI 227-106) was performed on different seams of Chinese coal (Shenhua Ningtiaota Coal #2 & #3). Since another coal, Shangwan coal was selected for the liquefaction plant, PB-10 was made as approved by DOE/COR.

The objective of this test was to evaluate the liquefaction performance of Shangwan coal utilizing various backend processing and recycle schemes. Additionally, this test was to collect available process data to allow for the best scale-up process design possible from this particular unit.

## SCOPE OF WORK

A 20-day test was scheduled. Various recycle schemes were to be studied, while system pressure, reactor temperatures, space velocity, and catalyst loading, etc. were set prior to the run, allowing only small adjustments.

### Unit Description

HTI's continuous Bench Scale Unit No. 227 was used for this run (Figure 1), which employs two backmixed liquefaction reactors and an integrated fixed-bed hydrotreater. The coal/oil slurry is premixed offline and charged to a feed tank on a periodic basis. The reactors are close coupled with the effluent from the first stage flowing directly to the second stage. The effluent from the second reactor was separated in a hot separator and the bottoms stream was sent to an atmospheric flash. The overhead from the 2nd stage hot separator was sent directly to the hydrotreater. The hydrotreater outlet was sent to a cold separator and separated into a vent gas stream and a separator overhead stream (SOH). The vent gases were metered, sampled, and sent to flare. The overhead from the atmospheric flash was sent to the hydrotreater. The bottoms stream from the atmospheric flash was separated offline by various processes depending on the particular recycle scheme being used for that condition. These streams were then analyzed. Part of the mid-distillate generated is used as a process oil in the buffer pumps for the first and second stage reactors. TNPS was used during startup to pre-sulfide the hydrotreater catalyst and TNPS and $H_2S$ were used during the run to activate the dispersed catalyst.

### Recycle Schemes

During this run four different recycle schemes were tested. The first recycle scheme was used in Condition 1B & 2A (Periods 3B-9A). A portion of the bottoms from the atmospheric flash was separated by pressure filtration into a pressure filter liquid (PFL) and a pressure filter cake or solid (PFC) and the rest of the bottoms was recycled to the feed tank. Most of the PFL was then recycled to the unit with a small portion taken as a product (due to losses in handling). The PFC was further processed with the solvent extraction producing a toluene extracted oil (TEO) and a toluene extracted solid (TES). The TEO was also recycled to the feed tank (See Figure 2).

The second recycle scheme was used during Condition 2B (Periods 9B-10B). All of the bottoms from the atmospheric flash was sent to pressure filtration producing a PFL and a PFC. The PFL was then vacuum distilled at a nominal cut point of 399°C producing a vacuum still overhead (VSOH) and a vacuum still bottoms (VSB). The VSOH was withdrawn as a product. The VSB was recycled to the feed tank. The PFC was further processed by solvent extraction producing a TEO and a TES. The TEO was then recycled to the reactor system (See Figure 3).

The third recycle scheme was used during Condition 3A (Period 11A-15B). All of the bottoms from the atmospheric flash was sent to pressure filtration producing a PFL and PFC. The PFL was then vacuum distilled at a nominal cut point of 371°C producing a vacuum still overhead (VSOH) and a vacuum still bottoms (VSB). The VSOH was taken as a product. The VSB was recycled to the feed tank. A portion of the PFC was recycled to the feed tank and the rest of it was solvent extracted producing a TEO and TES. The TEO was then recycled to the unit (See Figure 4).

The last recycle scheme used was used during Condition 4B (Periods 17B-20A). All of the bottoms from the atmospheric flash was vacuum distilled at a nominal cut point of 371°C producing an atmospheric still overhead and an atmospheric still bottoms. The atmospheric still overhead was taken as product. The atmospheric still bottoms was partially recycled to the feed tank and the rest was further vacuum distilled at 538°C producing a vacuum still overhead and a vacuum still bottoms. The vacuum still overhead was recycled to the feed tank while the vacuum still bottoms was taken as a product (See Figure 5).

## Run Plan

The run plan (Table 1) includes 9 separate operating conditions, 8 of which were actually operated. Condition 1A (Periods 1-3A) used the base coal, Shangwan coal, which was the same shipment used for the PDU run (260-007). 1A was a startup condition to establish flows and material for recycle. Part of the O-6 bottoms was recycled and the rest separated by pressure filtration. The PFL were vacuum distilled at a nominal cut point of 454°C and the bottoms recycled as well as a portion of the overheads. The PFC was solvent extracted and the TEO was recycled while the TES was taken as a product. For Condition 1B this was changed to decreasing the total amount of O-6 bottoms recycled, recycling the pressure filter liquid directly and recycling the toluene extracted oil. For Condition 2A the same scheme was followed except the coal was changed from that used in the PDU program to L-1007 which was a smaller lot of the same coal that was ground by HTI. For Condition 2B the recycle of the PFL was stopped and the VSB recycle was restarted. For Condition 3A a portion of the PFC was also recycled so as to resume solids recycle and the feed coal was changed back to that used in the PDU program. For Condition 3B the hydrotreater was bypassed to determine the quality and quantity of material flowing directly from the overhead of the hot separator to the hydrotreater. For Condition 4A the hydrotreater was placed back online and the O-6 bottoms was vacuum distilled twice (producing a 399°C- fraction, a 399°C+ fraction, a 399-538°C fraction and a 538°C+ fraction) recycling a 399°C+ fraction and a 399-538°C+ fraction. For Condition 4B the cut point of the first distillation was reduced to 371°C and the overall recycle ratio was changed from 1.6 to 1.25. The final condition, Condition 5, would have tested the benefit of hydrotreating the 371-538°C recycle oil before it was recycled to improve it as a hydrogen donor solvent.

## OPERATING SUMMARY

### Overview of Run PB-10 (HTI 227-109)

Run 227-109 was conducted on Unit 227 which was set up in a two-stage (backmixed) reactor configuration with the use of HTI's dispersed GelCat™ catalyst. The hydrotreater (HTU) was online throughout this run except for a short period for by-pass sampling. The interstage separation system was not used. TNPS was used during startup to pre-sulfide the hydrotreater catalyst and TNPS and $H_2S$ were used during the run to activate the dispersed catalyst. Two-hour feeds were sent to the small feed tank (P-2). From P-2, the feed was pumped to the backmixed first stage reactor (K-1). The effluent from K-1 was sent to the back-mixed second stage reactor (K-2). The products from K-2 were then separated using a hot separator (O-1). The O-1 bottoms stream was depressurized, sent to the atmospheric flash vessel (O-3), and then finally withdrawn from the bottoms receiver (O-6). The O-6 bottoms (product) was recycled and sent to the pressure filter or vacuum stills. Overheads from O-1 flowed through the HTU K-3 and the cold separator (O-2), producing separator overhead (SOH) and vent gas streams. The toluene extractor was also used during some conditions of this run.

### Start-up

The start-up procedures for run 227-109 were initiated on October 25, 1998 at 1500 hours. The hydrotreater (HTU) had been charged with 1,000 cc (884gms) of Criterion 411, Trilobe Catalyst, HTI-6292.

### Condition 1A

Coal cut-in at 1200 hours, 10/28 marked the start of the first condition. Recycle oils were O-6 Bottoms, Toluene Extracted Oil (TEO), and Vacuum Still Overheads and Bottoms (VSOH 454°C- and VSB 454 °C+, respectively). GelCat™ batch L-1019 was used as the catalyst. Di-Tertiary Nonyl Polysulfide (TNPS), 37% S, was injected as the sulfiding agent. K-1 temperatures were held at 440°C, K-2 was held at 450°C, and the HTU was online at 379°C.

### Condition 1B (Periods 3B-5)

For the second condition, beginning 1,400 hours, 10/30, Pressure Filter Liquid (PFL) replaced VSB, VSO, and half of the O-6 bottoms. Catalyst is changed from GelCat™ batch L-1019 to L-1010, a higher grade chemical batch.

### Condition 2A (Periods 6-9A)

Condition 2A began at 1200 hours, 11/2, when the coal source was switched to L-1007. All other parameters remained the same.

### Condition 2B (Periods 9B-10)

Condition 2B began at 1700 hours on November 5th, Period 9B, where vacuum distillation of the PFL was re-implemented, now at 399°C. Recycle oils consisted of TEO and VSB. The sulfiding agent was switch from TNPS to $H_2S$ (94 wt. % S).

### Condition 3A (Periods 11-15)

Condition 3A began when PDU coal was re-established as the coal source fed to the unit. The VSB feedrate was lowered by 70 g/hr to 1,300 g/hr and PFC feed was initiated at 70 g/hr. TEO and $H_2S$ feedrates remain the same. The catalyst was changed to a liquid version of GelCat™, batch L-1064.

### Condition 3B (Periods 16A)

The purpose of this nine-hour condition was to obtain an unhydrotreated separator overhead oil sample. The HTU was off-line by 0515 hours. The system was purged until 0740 hours. This two-hour accumulation was saved as S-497. The desired unhydrotreated sample was accumulated from 0740 until 1340 hours. The sample, weighing 5,032 grams, was saved as S-498 and was a combined sample of both oil and water.

### Condition 4A (Periods 16B-17A)

Condition 4A began when first the HTU was brought back online at 1340 hours. The new condition saw recycle streams of only 280 g/hr solids containing VSB 399°C+ and 1,160 g/hr solids free VSOH 399-538°C.

### Condition 4B (Periods 17B-20A)

At 1400 hours, Period 17 the feed composition was changed to 380 g/hr of VSB and 745 g/hr of VSOH, generated as described above in Condition 4A.

### Condition 5 (Periods 20B-23A)

Condition 5A began when the new feed blend was added to the feed pot at 2000 hours, Period 20B. The new blend returned to GelCat™ batch L-1010 at 60 g/hr. Also implemented was the combining of the Separator Overheads Oil to the O-6 bottoms material before taking the Batch Vacuum Still cuts. The HTU temperature is lowered to 354°C at 1,400 hours, Period 21. Additional HTU feed of S-499 is implemented at 0930 hours, Period 22. S-499 is a VSOH material of HTI-6825 cut at 510°C. This material is to be fed to the HTU at 350 g/hr but the pump is not sized to reach this flowrate, so less than 300 g/hr is achieved. The HTU pump is taken off line at 1510 hours, Period 22, to increase pumping flow rate capabilities and accommodate increased demand of S-499 feed. The HTU pump is back on line at 1630 hours.

The Continuous Vacuum Still (CVS) is used at about 2200 hours, Period 22, to cut the SOH oil at 700°F. The O-6 Btms material continues to be cut by the Batch Vacuum Stills at 371°C. CVS Bottoms are fed to the unit for the first time at 0400 hours, Period 23.

### Shutdown and Inspection

The run was terminated due to a fire in the hood where the feed material was prepared. At 1245 hours on Thursday, November 19, the unit began shutting down. By 1600 hours the reactor temperatures were at 400°C and an 8-hour wash was performed. By 1400 hours, 11/20, all temperatures were below 150°C and all controllers and pumps were turned off. The unit was depressured and opened for inspection.

Both of the reactor's riser tubes, top heads, and bubble caps were removed with relative ease. The reactors were relatively clean.

# UNIT OPERATION

## Test Materials

The coal feedstock and catalyst were characterized prior to the test run.

### Feed Coal

Two lots of Chinese coals were used during this program. The base coal was the Shangwang coal used for the PDU program (260-007). An alternate lot (L-1007, which was pulverized and screened from HTI-6932, an earlier small shipment of Shangwan coal) was also used. These two coals are considered to have only minor variations in composition. (Table 2).

### Catalyst

Three different formulations of GelCat™ catalysts were used in this program. L-1019 was a gel form of GelCat™ produced from low-cost domestically available chemicals, L-1010 was a gel form of GelCat™ produced from high-cost imported chemicals and L-1064 was a liquid form of GelCat™.

## Reactor Conditions

Reactor pressure was kept at 17.2 MPa (2,500 psi) throughout the entire run. The first stage reactor temperature was set at 440°C for the entire test and held very steady at this value (Figure 6). The second stage reactor temperature was initially set at 450°C for Conditions 1A-4A and 453°C for Conditions 4B-5 and also held very steady at the set values (Figure 6). The space velocity for this run was to be held steady at 450 kg/hr/m$^3$/stage and as can be seen from Figure 7 this was achieved except for Periods 14 & 15. In these periods the space velocity varied by approximately 10% from the specified space velocity due to level changes in the feed tank.

## Material Recovery

The material recovery for the run is presented in Figure 8. Recoveries were generally good and averaged 98.1 W%. The recycle ratio (recycle solvent/maf feed) is presented in Figure 9. The recycle solvent includes all material recycled through the slurry mixing tank (SMT) including makeup oil but does not include material recycle to other parts of the unit (i.e. buffer oil to the reactors). Figure 10 present the loading of catalyst used during the run. The Fe catalyst was kept constant at approximately 5,000 wppm maf ff while the promoter was raised during the run from 100 to 150 wppm maf ff.

## PROCESS PERFORMANCE

### Methodology

The process performance in terms of conversions, product yields and qualities is discussed in this section. Detailed laboratory analysis of the products were performed on a daily basis to provide timely process evaluation. The daily material balance, coal conversion, normalized yields and other process performance related indicators were calculated using the programs developed at HTI. The overall material balance and process performance is summarized in Tables 3 to 5. The material balance was calculated in four different ways. The first method closes the balance at the O-6 bottoms. This is the balance that is used for all normalized yield calculations. The second method closes the balance after all bottoms processing and separation has occurred. For this case the losses which occur in the batch processes used are pro-rated based on the products from those processes. The final two methods merely present the same data as the first two but on a 100% maf coal basis as opposed to the actual 24 hour period collection weights.

Five periods were selected for the purpose of comparison of product quality and process performance. These periods represent five different operating conditions. Due to the changes that were frequently made to the unit in order to "tune" the performance and due to the unit rarely being in solvent balance, these periods represent the best approximation of what true steady state operation would have been and an excellent point of comparison among the operating parameters used in this test.

Comparing Condition 1B with Condition 2A shows the effect of the particular batch of coal that was used. Condition 1B was performed with the bulk coal shipment used for the PDU and Condition 2A was performed with a smaller batch of coal which was shipped early and ground by HTI. Condition 2B is also performed with the smaller batch of coal but the recycle is changed from that used in Conditions 1B & 2A. Instead of recycling the solids containing O-6 Bottoms, TEO and the entire PFL it was changed to recycling the TEO and the 750°F+ portion of the PFL. For Condition 3A the coal was changed back to the large lot used for the PDU, and this was used for the rest of the test. Also for Condition 3A the recycling scheme was changed to recycling a portion of the solids containing PFC, the TEO and the 700°F+ portion of the PFL. For Condition 4B the use of pressure filtration was eliminated. The O-6 bottoms were first distilled at 700°F producing a light oil product and a heavy oil/solids containing bottoms. This bottoms stream was then partially recycled and the rest vacuum distilled at 1,000°F producing a heavy oil for recycle and a resid stream as a product.

### Coal Conversion

The total coal conversion was calculated on the basis of the solubility of O-6 bottoms in quinoline. The coal conversions for different conditions are shown in Figure 11 and Table 5. Comparing Conditions 1B and 2A shows an increase in coal conversion of 1.8 W% maf fresh feed, from 90.9% to 92.7%. This would indicate that the coal used for the PDU program might have been partially oxidized prior to this bench-scale testing resulting in lower coal conversion for the PDU feed coal. The coal conversion for Condition 2B, also the smaller lot of coal, shows a conversion of 92.3 W% maf fresh feed showing little impact from the change in the recycle scheme. Condition 3A shows the highest coal conversion of 93.5 W% maf fresh feed with the PDU coal. This could indicate that the change in the recycle scheme resulted in a higher coal conversion or that the Condition 1B coal conversion which was lower was not representative of the performance of that lot of coal. This would indicate that the recycle scheme is responsible for the changes in the coal conversion. For Condition 4B the coal conversion drops to 89.7 W% maf fresh feed. This is a significant drop in conversion and might indicate that the recycle scheme was also responsible for the decrease. Condition 4B was too short to draw a strong conclusion on, though.

## Resid Conversion

The 524°C+ resid conversion represents the ability of the process to convert heavy (high boiling) fractions contained in the feedstocks. The resid conversion is an important measure of process performance and the effect of various recycle schemes. For the purpose of calculation, all of the maf portion of the feed coal is considered as 524°C+ resid while the resid content of any recycle oils has been measured by distillation. Two separate types of resid conversion are discussed. The first is the overall process resid conversion, calculated based on the resid conversion around the entire process and accounting for the recycle streams as internal recycles. The second is a single-pass reactor resid conversion, calculated based on the resid conversion just around the reactor and accounting for the recycle streams as feed and product streams from the reactor. The process resid conversion measures process performance and the reactor resid conversion measures reactor performance (Figures 11 & 12).

For Conditions 1B, 2A, 2B, & 3A the reactor resid conversion varied from 45.0 to 51.2 W% maf ff while the process resid conversion varied from 76.3 to 88.6 W% maf fresh feed. The highest process resid conversion, 88.6 W% maf ff, was for the same condition as the second lowest reactor resid conversion, 48.2 W% maf ff for Condition 2B. Condition 1B and 2A were performed with identical recycle schemes and different lots of coal. Even though Condition 1B had a slightly lower coal conversion than Condition 2A it has a higher resid conversion for both the process (79.9 vs 76.3 W% maf ff) and the reactor (51.2 vs 45.0 W% maf ff). This difference might not be very significant as Condition 1B was only 2 1/2 days long and Condition 2A was only 3 days long. For Conditions 2B and 3A the recycle scheme was significantly changed. While Condition 3A recycled a small portion of the PFC for recycling the solids and Condition 2B doesn't, the most important feature of these two recycle schemes was that the only resid material removed from the process is that which can not be extracted by the solvent extraction unit. As all other material is recycled it results in a very high resid conversion, limited by what is removed with the solvent extracted bottoms. Condition 4B recycles part of the resid material in the bottoms product from the first distillation, at 371°C; however, the bottoms from the second distillation, at 538°C, is taken as a product. This results in a much lower process resid conversion of only 64.8 W% maf ff, while the reactor resid conversion is actually the highest at 58.0 W% maf ff due to much reduced recycle ratio.

## Hydrogen Consumption

The hydrogen consumption for this run is presented in Figure 13 and Table 5. Hydrogen consumption shows little variation for Condition 1B, 2B, and 3A, varying from 8.2 to 8.8 W% maf ff. The hydrogen consumption for Condition 2A stands out as lower at 6.8 W% maf ff. This is the condition with the lowest light gas yield, 10.7 W% maf ff, as well as the lowest $C_1$-$C_7$ gas yield, 16.1 W% maf ff. The lower gas yield would account for a major portion of the decrease in hydrogen consumption. This condition also had a low process resid conversion. Condition 4B, which had the lowest process resid conversion, but had a significant gas yield, 12.4 W% maf ff for the light gas yield and 19.6 W% maf ff for the total gas yield, had a higher hydrogen consumption of 7.8 W% maf ff.

## Product Distribution

### Gas Yield

The $C_1$-$C_2$ gas yield varied from 7.4 to 9.7 W% maf ff for this program (Figure 14). The lowest dry gas yield was seen in Condition 2A and the highest in Condition 2B. The $C_3$-$C_4$ (LPG components) gas yield varied from 6.5 to 8.9 W% maf ff (Figure 15). For the three conditions with the lowest process resid conversion, Conditions 1B, 2A and 4B, the $C_3$-$C_4$ gas yield only varied from 6.5 to 7.2 W%, while for the two conditions with high process resid conversion, Conditions 2B and 3A, the $C_3$-$C_4$ yield varied from 8.0 to 8.9 W% maf ff.

### Total Distillate Yield

Due to the use of makeup oil to balance out the solvent requirements of the unit, some of the individual yields for the product fraction were negative. As this is not possible, all the liquid fractions have been combined into a single product yield, $C_4$-524°C (Figure 16). This represent the success of the process in converting the coal to useful products as opposed to light gases and resid. As would be expected the product yield matches the process resid conversion fairly closely. The first two conditions, Condition 1B and 2A, had comparable process resid conversion of 79.9 and 76.3 W% maf ff, respectively, and also comparable total distillate yields of 58.2 and 58.5 W% maf ff, respectively. Conditions 2B and 3A, with the highest resid conversions of 88.6 and 85.9 W% maf ff, respectively, also had the highest total distillate yields at 67.5 and 67.2 W% maf ff respectively. Condition 4B had both the lowest resid conversion, 64.8 W% maf ff, as well as the lowest total distillate yield, 45.6 W% maf ff.

### Product Quality

The two main product streams which were used for all calculations of material balance and normalized yields were the HTU product and the O-6 Bottoms. The quality of these two streams is discussed below. The other process streams varied more widely in quality as the specification for the recycle schemes changed from condition to condition so that some streams are only used for one condition and can't be compared across conditions. Appendix A includes a list of analysis performed on all the product streams.

Separator Overhead (SOH)

The HTU product (SOH) shows a decrease in quality as the run progresses. As seen in Figure 17 both the H/C atomic ratio and the API decreases as the run proceeds. From Figure 18 it can also be seen that both the Sulfur and Nitrogen content of the HTU product shows an increase as the run progresses. The decline in hydrogenation function as well as in HDS and HDN efficiencies of the hydrotreater may be attributed to the initial activity loss of the fresh catalyst loaded to the hydrotreater. The activity decline may also be related with changes in recycle scheme, where recycle streams with higher end points were fed to the HTU under certain conditions.

O-6 Bottoms

The O-6 Bottoms properties are presented in Figure 19. As the recycle schemes was changed throughout the run the quality of the O-6 Bottoms also changed throughout the run as it is very dependent on the recycle material. The solids content (as measured by the quinoline insolubles) varied from 9.0 to 11.4 W% throughout the test. The oil resid content of the bottoms (not including the solids) varied from 19.9-44.5 W% throughout the test.

### Product Characterization

The SOH and VSOH products from Period 13 were characterized by True Boiling Point distillation followed by crude assay tests according to ASTM fuel specifications. These results are summarized in Table 6.

### Hydrotreater Performance

The hydrotreater performance is presented in Table 7. During the run the primary feed to the hydrotreater was the overhead stream from the hot separator after the second reactor. This stream, which contained hydrocarbon gases and vapors, and non-hydrocarbon gases including hydrogen, was fed directly to the hydrotreater with no letdown in pressure and cooled slightly to approximately 371°C. Also fed to the HTU was the overhead stream from the atmospheric column.

The hydrotreater was by-passed at the end of the Condition 3A. This allowed a sample of unhydrotreated second stage SOH oil stream to be drawn and analyzed. The rate into the hydrotreater was measured during the previous 24 hours of operation. The rate exiting from the hydrotreater was based on the relative rates of the various components as measured during a two hour long collection period. This was then normalized to give a 100% material balance across the hydrotreater.

The hydrocarbon material (gases and liquids) flowing through the hydrotreater pick up 1.8% of their own weight in hydrogen. This results in an increase in the H/C ratio from 1.48 to 1.85. The API of the final product is increased to 36.0 from 21.3. The most dramatic improvement is to the sulfur and nitrogen content. The sulfur content 1119 wppm is reduced to 59 wppm. The nitrogen content of 695 wppm is reduced to <1 wppm. The hydrotreater was very successful at improving the quality of the final product. As can be seen from the data in Table 7, the hydrotreater also had a hydrocracking effect besides hydrotreating. From this it can be seen that the hydrotreater has a very significant contribution to the final quality of the product.

# CONCLUSIONS

The small lot of Shangwan coal that was processed by HTI does result in a slightly higher coal conversion of 1.8W% maf ff than the large lot of Shangwan coal used for the PDU program. The reactor performance as measured by reactor resid conversion shows some variation based on the recycle scheme being used. This follows the pattern that the greater the recycle of resid material the lower the reactor performance. This is due to the recycle resid becoming more concentrated in refractory materials which are very difficult or impossible to convert. The process performance, as measured by the process resid conversion, shows a very strong impact from the recycle scheme. Even though the reactor resid conversion decreases with high levels of resid recycle, the process resid conversion increases sharply as these materials are not taken as product. The maximum performance is achieved with the maximum recycle of resid. The performance achieved for the design basis 93.5 W% maf ff coal conversion, 85.9 W% maf ff resid conversion, and 67.2 W% $C_4$-975°F distillate yield with a hydrogen consumption of 8.8 W% maf ff. The hydrotreater had a significant impact on final product quality; increasing the hydrogen content by 1.8 W%, increasing the H/C Atomic ratio from 1.48 to 1.85, increasing the API gravity from 21.3 to 36.0. The HTU had a HDS efficiency of >95% and a HDN efficiency nearly 100.

# TABLE 1
# CHINA COAL BENCH TEST 227-109 RUN PLAN

| Condition | 1A | 1B | 2A | 2B | 3A | 3B | 4A | 4B | 5 |
|---|---|---|---|---|---|---|---|---|---|
| Purpose | Proposed PDU Condition | PDU Condition | Original Coal | Change Recycle | Optimize P-2 Coal | Hydrotreater Bypass | New Recycle | Adjust Recycle | Adjust Separation |
| Periods | 1-3A | 3B-5 | 6-9a | 9b-10 | 11-15 | 16a | 16b-17a | 17b-20a | 20b-24a |
| Solids Separation | See Note | See Note | See Note | See Note | See Note | See Note | See Note | See Note | See Note |
| Hydrotreater | YES | YES | YES | YES | YES | Bypass | Yes | Yes | Yes |
| Interstage Separation | NO | NO | NO | NO | NO | NO | NO | NO | NO |
| Coal | P-2 | P-2 | L-1007 | L-1007 | P-2 | P-2 | P-2 | P-2 | P-2 |
| VS Cutpoint | 800 | na | na | 750 | 750 | 750 | 750 | 700 | 700 |
| MAF Fresh Feed SV, kg/h/m3 | | | | | | | | | |
| One Reactor Volume | 450 | 450 | 450 | 450 | 450 | 450 | 450 | 450 | 450 |
| Total Reactor Volume | 225 | 225 | 225 | 225 | 225 | 225 | 225 | 225 | 225 |
| Pressure, psig | | | | | | | | | |
| K-1 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 |
| K-2 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 | 2500 |
| Temperature, F | | | | | | | | | |
| K-1 | 824 | 824 | 824 | 824 | 824 | 824 | 824 | 824 | 824 |
| K-2 | 842 | 842 | 842 | 842 | 842 | 842 | 842 | 847 | 847 |
| K-3 | 715 | 715 | 715 | 715 | 715 | 715 | 715 | 715 | 715 |
| Catalyst Concentration, Fe ppm ff | 5000 | 5000 | 5000 | 5000 | 5000 | 5000 | 5000 | 5000 | 5000 |
| Promoter (total Concentration) | 100 | 100 | 100 | 100 | 150 | 150 | 150 | 150 | 200 |
| Sulfur Addition, W% coal | 2 | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| Recycle to the SMT, recycle / maf coal | | | | | | | | | |
| O-6 Bottoms | 1.0 | 0.5 | 0.5 | 0 | 0 | 0 | 0 | 0 | 0 |
| TEO | 0.1 | 0.1 | 0.1 | 0.08 | 0.08 | 0.08 | 0 | 0 | 0 |
| VSB | 0.25 | 0 | 0 | 1.52 | 1.44 | 1.44 | 0 | 0 | 0 |
| VSOH | 0.25 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| PFL | 0 | 1.0 | 1.0 | 0 | 0 | 0 | 0 | 0 | 0 |
| PFC | 0 | 0 | 0 | 0 | 0.08 | 0.08 | 0 | 0 | 0 |
| VSB 750+F | 0 | 0 | 0 | 0 | 0 | 0 | 0.31 | 0.42 | 0.42 |
| VSB 750-1000F | 0 | 0 | 0 | 0 | 0 | 0 | 1.29 | 0.83 | 0.83 |
| Feeds, gms/hr | | | | | | | | | |
| Coal (from P-2, as is) | 1046 | 1046 | 0 | 0 | 1046 | 1046 | 1046 | 1046 | 1046 |
| Coal (L-1007, as-is) | 0 | 0 | 1046 | 1046 | 0 | 0 | 0 | 0 | 0 |
| Molyvan-A | 0 | 0.132 | 0.132 | 0.132 | 0.158 | 0.158 | 0.158 | 0.158 | 0.44 |
| O-6 Bottoms | 900 | 450 | 450 | 0 | 0 | 0 | 0 | 0 | 0 |
| TEO | 90 | 90 | 90 | 70 | 70 | 70 | 0 | 0 | 0 |
| VSB | 230 | 0 | 0 | 1370 | 1300 | 1300 | 0 | 0 | 0 |
| TNPS | 50 | 75 | 75 | 0 | 0 | 0 | 0 | 0 | 0 |
| H2S | 0 | 0 | 0 | 29 | 29 | 29 | 29 | 29 | 29 |
| GelCat L-1019 (7.5 W% Fe) | 60 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| GelCat L-1010 (7.5W% Fe) | 0 | 60 | 60 | 60 | 0 | 0 | 0 | 0 | 60 |
| Liquid Gelcat L-1064, Fe=8.6 | 0 | 0 | 0 | 0 | 52 | 52 | 52 | 54 | 0 |
| VSOH | 230 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| PFL | 0 | 900 | 900 | 0 | 0 | 0 | 0 | 0 | 0 |
| PFC | 0 | 0 | 0 | 0 | 70 | 70 | 0 | 0 | 0 |
| VSB 750F+ (16% solids) | 0 | 0 | 0 | 0 | 0 | 0 | 280 | 380 | 380 |
| VSOH 750-1000F | 0 | 0 | 0 | 0 | 0 | 0 | 1160 | 745 | 745 |
| Total Recycle | 1440 | 1440 | 1440 | 1440 | 1440 | 1440 | 1440 | 1125 | 1125 |

# TABLE 2
# SHANGWAN COAL ANALYSIS

| | | PDU Coal | L-1007 |
|---|---|---|---|
| As-Is Moisture, W% | 9.83 | | 9.40, 9.78 |
| | | | |
| Proximate Analysis | W% | mf W% | |
| Moisture | 8.95 | | |
| Ash | 4.90 | 5.38 | |
| Volatile Matter | 32.23 | 35.40 | |
| Fixed Carbon | 53.90 | 59.20 | |
| | | | |
| Ultimate Analysis, mf W% | External | Internal | |
| Carbon | 77.08 | 75.52, 75.31 | 77.33 |
| Hydrogen | 3.80 | 4.30, 4.15 | 4.80 |
| Sulfur | | 0.34, 0.36 | 0.37 |
| Nitrogen | 0.99 | 0.90, 0.85 | 0.86 |
| Ash | 6.05 | 5.99 | 5.64, 5.86 |
| Sulfur in Ash, W% | | | 4.78 |
| Oxygen by Difference, W% | 11.74 | 12.95 | 10.78 |
| | | | |
| Sulfur Forms, mf W% | | | |
| Sulfate | 0.01 | | |
| Pyritic | 0.22 | | |
| Organic | 0.08 | | |
| | | | |
| Mineral Analysis | W% ash | mf W% | |
| Silicon | 12.064 | 0.723 | |
| Aluminum | 5.631 | 0.337 | |
| Titanium | 0.252 | 0.015 | |
| Iron | 5.693 | 0.341 | |
| Calcium | 28.016 | 1.678 | |
| Magnesium | 0.507 | 0.030 | |
| Potassium | 0.299 | 0.018 | |
| Sodium | 1.714 | 0.103 | |
| Sulfur | 3.973 | 0.238 | |
| Phosphorus | 0.035 | 0.002 | |
| Strontium | 0.296 | 0.018 | |
| Barium | 0.125 | 0.008 | |
| Manganese | 0.303 | 0.018 | |
| Molybdenum | | | |
| | | | |
| Miscellaneous | | | |
| Heating Value (dry), btu/lb | 12934 | | |
| Heating Value (maf), btu/lb | 13767 | | |
| Silica Value | 34.88 | | |
| Chloride, mf W% | 0.01 | | |
| | | | |
| Sieve Analysis, W% | | Ash, W% | |
| +45 | 3.9, 4.3 | 7.68 | |
| 45-70 | 10.8, 12.1 | 4.98 | |
| 70-140 | 21.6, 28.8 | | |
| 140-200 | 24.6, 15.2 | | |
| 200-325 | 27.0, 25.9 | | |
| -325 | 10.8, 12.5 | | |
| Losses | 1.3, 1.2 | | |

# TABLE 3
# MATERIAL BALANCE, 100 PARTS MAF COAL BASIS

| DATE | 11/01/98 | 11/04/98 | 11/06/98 | 11/09/98 | 11/16/98 |
|---|---|---|---|---|---|
| CONDITION | 1B | 2A | 2B | 3A | 4B |
| PERIOD | 5 | 8 | 10 | 13 | 20A |
| Coal Description | P-2 Sample | L-1007 | L-1007 | P-2 Sample | P-2 Sample |
| Hydrotreater (yes/no) | Yes | Yes | Yes | Yes | Yes |
| Catalyst Type | L-1010 | L-1010 | L-1010 | L-1064 | L-1064 |
| K-1 Temperature, °F | 825.5 | 824.6 | 823.5 | 824.3 | 823.6 |
| K-2 Temperature, °F | 841.5 | 842.5 | 842.6 | 842.7 | 846.4 |
| Space Velocity maf (lb/hr/ft3 stage 1) | 27.74 | 27.90 | 25.94 | 29.49 | 27.73 |
| Coal Content, mf coal / mf feed | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Feeds | | | | | |
| Coal, maf | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Coal ash | 6.37 | 6.22 | 6.22 | 6.37 | 6.37 |
| Coal moisture | 9.88 | 11.51 | 11.51 | 9.88 | 9.88 |
| TNPS | 8.42 | 8.42 | 0.00 | 0.00 | 0.00 |
| H2S | 0.00 | 0.00 | 3.62 | 3.06 | 3.16 |
| GelCat | 6.67 | 6.75 | 6.75 | 5.78 | 6.00 |
| L-1062 (Makeup Oil) | 9.03 | 6.69 | 0.00 | 0.00 | 28.80 |
| Promoter | 0.01 | 0.01 | 0.01 | 0.02 | 0.02 |
| Water Loss on Hotplate | -13.48 | -14.71 | -14.71 | -13.00 | -13.12 |
| Light Oil Loss on Hotplate | -8.02 | -6.78 | -5.25 | -3.23 | -2.88 |
| O-6 Bottoms | 50.01 | 50.65 | 0.00 | 0.00 | 0.00 |
| VSOH | 0.00 | 0.00 | 6.71 | 6.93 | 0.00 |
| VSB | 0.00 | 0.00 | 154.21 | 137.08 | 0.00 |
| TEO | 8.34 | 6.85 | 5.09 | 3.36 | 0.00 |
| PFL | 101.14 | 104.59 | 0.00 | 0.00 | 0.00 |
| PFC | 0.00 | 0.00 | 0.00 | 7.78 | 0.00 |
| ASB (750°F+) | 0.00 | 0.00 | 0.00 | 0.00 | 42.23 |
| VSOH (750-1000°F) | 0.00 | 0.00 | 0.00 | 0.00 | 59.62 |
| Water | 35.51 | 34.49 | 36.20 | 8.69 | 88.23 |
| Hydrogen Feed | 27.90 | 32.70 | 35.18 | 0.00 | 32.95 |
| TOTAL FEED | 341.78 | 347.39 | 345.56 | 272.72 | 361.26 |
| PRODUCTS | | | | | |
| Hydrogen in Product Gases | 19.96 | 25.81 | 30.28 | 0.00 | 28.30 |
| Product Gas (H & N free) | 24.11 | 20.35 | 28.48 | 31.59 | 25.55 |
| O-6 Bottoms | 199.66 | 208.61 | 204.87 | 196.22 | 162.01 |
| HTU | 36.12 | 36.00 | 34.86 | 28.48 | 34.97 |
| SOH H2O | 50.01 | 45.84 | 48.71 | 14.00 | 99.30 |
| KO | 3.26 | 1.07 | 0.58 | 0.82 | 0.68 |
| TOTAL PRODUCTS | 333.1 | 337.7 | 347.8 | 271.1 | 350.8 |
| RECOVERY, W% | 97.5 | 97.2 | 100.6 | 99.4 | 97.1 |
| Liquid Closure, W% | 92.1 | 92.6 | 93.1 | 92.2 | 90.4 |

# TABLE 4
# PROCESS STREAMS MATERIAL BALANCE, 100 PARTS MAF COAL BASIS

| DATE | 11/01/98 | 11/04/98 | 11/06/98 | 11/09/98 | 11/16/98 |
|---|---|---|---|---|---|
| CONDITION | 1B | 2A | 2B | 3A | 4B |
| PERIOD | 5 | 8 | 10 | 13 | 20A |
| Coal Description | P-2 Sample | L-1007 | L-1007 | P-2 Sample | P-2 Sample |
| Hydrotreater (yes/no) | Yes | Yes | Yes | Yes | Yes |
| Catalyst Type | L-1010 | L-1010 | L-1010 | L-1064 | L-1064 |
| K-1 Temperature, °F | 825.5 | 824.6 | 823.5 | 824.3 | 823.6 |
| K-2 Temperature, °F | 841.5 | 842.5 | 842.6 | 842.7 | 846.4 |
| Space Velocity maf (lb/hr/ft3 stage 1) | 27.7 | 27.9 | 25.9 | 29.5 | 27.7 |
| Coal Content, mf coal / mf feed | 100 | 100 | 100 | 100 | 100 |
| FEEDS, gms | | | | | |
| Coal, maf | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Coal ash | 6.37 | 6.22 | 6.22 | 6.37 | 6.37 |
| Coal moisture | 9.88 | 11.51 | 11.51 | 9.88 | 9.88 |
| TNPS | 8.42 | 8.42 | 0.00 | 0.00 | 0.00 |
| H2S | 0.00 | 0.00 | 3.62 | 3.06 | 3.16 |
| GelCat | 6.67 | 6.75 | 6.75 | 5.78 | 6.00 |
| L-1062 (Makeup Oil) | 9.03 | 6.69 | 0.00 | 0.00 | 28.80 |
| Promoter | 0.01 | 0.01 | 0.01 | 0.02 | 0.02 |
| Water Loss on Hotplate | -13.48 | -14.71 | -14.71 | -13.00 | -13.12 |
| Light Oil Loss on Hotplate | -8.02 | -6.78 | -5.25 | -3.23 | -2.88 |
| O-6 Bottoms | 50.01 | 50.65 | 0.00 | 0.00 | 0.00 |
| VSOH | 0.00 | 0.00 | 6.71 | 6.93 | 0.00 |
| VSB | 0.00 | 0.00 | 154.21 | 137.08 | 0.00 |
| TEO | 8.34 | 6.85 | 5.09 | 3.36 | 0.00 |
| PFL | 101.14 | 104.59 | 0.00 | 0.00 | 0.00 |
| PFC | 0.00 | 0.00 | 0.00 | 7.78 | 0.00 |
| ASB (750°F+) | 0.00 | 0.00 | 0.00 | 0.00 | 42.23 |
| VSOH (750-1000°F) | 0.00 | 0.00 | 0.00 | 0.00 | 59.62 |
| Water | 35.51 | 34.49 | 36.20 | 41.95 | 88.23 |
| Hydrogen Feed | 27.90 | 32.70 | 35.18 | 30.94 | 32.95 |
| TOTAL FEED | 341.78 | 347.39 | 345.56 | 336.92 | 361.26 |
| PRODUCTS, gms | | | | | |
| Hydrogen in Product Gases | 19.96 | 25.81 | 30.28 | 25.81 | 28.30 |
| Product Gas (H & N free) | 24.11 | 20.35 | 28.48 | 31.59 | 25.55 |
| O-6 Bottoms | 50.01 | 50.65 | 0.00 | 0.00 | 0.00 |
| VSOH | 0.00 | 0.00 | 9.64 | 17.32 | 0.00 |
| VSB | 0.00 | 0.00 | 187.52 | 144.80 | 0.00 |
| TEO | 7.76 | 6.62 | 1.54 | 7.20 | 0.00 |
| TES | 12.83 | 20.04 | 6.17 | 19.12 | 0.00 |
| PFL | 129.05 | 131.30 | 0.00 | 0.00 | 0.00 |
| PFC | 0.00 | 0.00 | 0.00 | 7.78 | 0.00 |
| HTU | 36.12 | 36.00 | 34.86 | 28.48 | 34.97 |
| SOH H2O | 50.01 | 45.84 | 48.71 | 56.59 | 99.30 |
| KO | 3.26 | 1.07 | 0.58 | 0.82 | 0.68 |
| ASO (750°F-) | 0.00 | 0.00 | 0.00 | 0.00 | 14.74 |
| ASB (750°F+) | 0.00 | 0.00 | 0.00 | 0.00 | 42.23 |
| VSOH (750-1000°F) | 0.00 | 0.00 | 0.00 | 0.00 | 66.67 |
| VSB (1000°F+) | 0.00 | 0.00 | 0.00 | 0.00 | 38.37 |
| TOTAL PRODUCTS | 333.12 | 337.69 | 347.78 | 339.50 | 350.81 |
| RECOVERY, W% | 97.47 | 97.21 | 100.64 | 100.77 | 97.11 |
| Liquid Closure, W% | 92.1 | 92.6 | 93.1 | 92.2 | 90.4 |

# TABLE 5
# NORMALIZED YIELDS

| DATE | 11/01/98 | 11/04/98 | 11/06/98 | 11/09/98 | 11/16/98 |
|---|---|---|---|---|---|
| CONDITION | 1B | 2A | 2B | 3A | 4B |
| PERIOD | 5 | 8 | 10 | 13 | 20A |
| Coal Description | P-2 Sample | L-1007 | L-1007 | P-2 Sample | P-2 Sample |
| Hydrotreater (yes/no) | Yes | Yes | Yes | Yes | Yes |
| Catalyst Type | L-1010 | L-1010 | L-1010 | L-1064 | L-1064 |
| K-1 Temperature, °F | 825.5 | 824.6 | 823.5 | 824.3 | 823.6 |
| K-2 Temperature, °F | 841.5 | 842.5 | 842.6 | 842.7 | 846.4 |
| Material Recovery % | 97.5 | 97.2 | 100.6 | 100.8 | 97.1 |
| Space Velocity, maf (lb/hr/ft3 stage 1) | 27.74 | 27.90 | 25.94 | 29.49 | 27.73 |
| Recycle Ratio (maf coal/solvent, with makeup oil) | 1.69 | 1.69 | 1.66 | 1.55 | 1.31 |
| Recycle Ratio (SMT Only) | 1.61 | 1.62 | 1.59 | 1.48 | 1.25 |
| NET NORMALIZED YIELDS, W% maf fresh feed | | | | | |
| C1 gases | 5.80 | 4.28 | 5.15 | 4.53 | 4.70 |
| C2 gases | 3.89 | 3.16 | 4.15 | 3.69 | 3.51 |
| C3 gases | 4.51 | 3.89 | 5.40 | 4.84 | 4.25 |
| C4 gases | 2.70 | 2.60 | 3.49 | 3.14 | 2.62 |
| C5 gases | 1.56 | 1.76 | 5.25 | 9.18 | 3.20 |
| C6 & C7 gases | 0.62 | 1.10 | 1.41 | 1.41 | 1.35 |
| C1-C3 in Gases | 13.56 | 10.66 | 14.70 | 13.06 | 12.45 |
| C4-C7 in Gases | 4.88 | 5.46 | 10.15 | 13.73 | 7.16 |
| IBP-350 | 8.73 | 10.32 | 10.83 | 9.47 | 9.22 |
| 350-975 | 44.57 | 42.69 | 46.47 | 43.99 | 29.24 |
| 975+ | 10.49 | 16.36 | 3.76 | 5.30 | 20.47 |
| Unconverted Coal / Coke | 8.75 | 7.36 | 7.97 | 6.39 | 12.55 |
| Water | 15.12 | 12.04 | 12.22 | 14.50 | 14.02 |
| CO | 0.27 | 0.35 | 0.38 | 0.39 | 0.49 |
| CO2 | 0.87 | 0.63 | 0.69 | 0.75 | 1.17 |
| NH3 | 1.13 | 0.89 | 1.05 | 1.23 | 0.98 |
| H2S | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| PROCESS PERFORMANCE, W% maf fresh feed | | | | | |
| H2 Consumption, maf ff | 8.39 | 6.76 | 8.23 | 8.82 | 7.75 |
| Coal Conversion, maf ff | 90.9 | 92.7 | 92.3 | 93.5 | 89.7 |
| 524°C+ Conversion | 79.9 | 76.3 | 88.6 | 85.9 | 64.8 |
| C4-524°C Distillates | 58.2 | 58.5 | 67.5 | 67.2 | 45.6 |
| 524°C+ Resid Yield + Unconv Coal, maf ff | 19.2 | 23.7 | 11.7 | 11.7 | 33.0 |

# TABLE 6A
# TBP ON PERIOD 13 SOH

| Fraction Analysis | IBP-180°F | 180-360°F | 360-428°F | 428-662°F | 662+°F | Whole |
|---|---|---|---|---|---|---|
| Weight % of total | 5.13 | 29.80 | 12.43 | 44.93 | 7.53 | 100 |
| Gravity, °API | 63.6 | 48.9 | 35.1 | 27.6 | 26.8 | 36.5 |
| Specific gravity@60°F | 0.7253 | 0.7844 | 0.8493 | 0.8894 | 0.8939 | 0.8423 |
| Carbon, W% | 84.87 | 85.78 | 87.29 | 87.25 | 86.69 | 87.31 |
| Hydrogen, W% | 14.92 | 14.02 | 13.30 | 13.08 | 13.83 | 13.60 |
| Nitrogen (P&E)% | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 |
| Antek Nitrogen, ppm | <1.00 | <1.00 | <1.00 | 1.41 | 57.5 | 1.39 |
| Antek Sulfur, ppm | <1.00 | 1.28 | 7.90 | 142.8 | 282.3 | 118.6 |
| CCR, W% | | | | | 0.036 | 0.00 |
| Ash (ASTM), W% | | | | | 0.0 | 0.00 |
| Heptane Insol, W%, | | | | | 0.0 | 0.00 |
| Basic Nitrogen, ppm | | | | | | 10 |
| Pour Point, °C | | | | -59.0 | | |
| Freezing Point, °C | | | < -75.0 | -64.0 | 36.0 | < -75.0 |
| Aniline Point, °C | | | 38.0 | 57.2 | 96.7 | 52.8 |
| Viscosity,cSt@38°C (100°F) | | | 1.49 cSt | 4.17 cSt | | 1.72cSt |
| Viscosity,cSt@23.3°C (74 °F) | | | | 5.77 cSt | cSt@100°C<br>6.66 | |
| Smoke Point, mm | | | 13.4 | | | |
| Copper Corrosion, ASTM D130 | | @ 122°F 3 b | @ 212°F 3 b | @ 212 °F 1 b | | |
| Existent Gum, mg/100mL | | 0.0 | 5.75 | 32.42 | | |
| Metals, ppm | | | | | | |
| Vanadium | | | | | < 1 ppm | |
| Nickel | | | | | < 1 ppm | |
| Copper | | | | | < 1 ppm | |
| Iron, W% | | | | | 13.90 | |
| PONA Analysis, V% | | | | | | |
| Saturates | | | | | | |
| Paraffins | | 23.93 | 3.7 | 10.09 | | |
| Olefins | | 0.30 | 0.8 | 1.6 | | |
| Naphthenes | | 57.87 | 77.92 | 78.12 | | |
| Aromatics | | 14.53 | 17.01 | 10.19 | | |
| Naphthalenes | | | 0.57 | 0.4 | | |
| Losses | | 3.37 | 0 | 0 | | |
| Total | | 100 | 100 | 100 | | |
| Phenolics content, W% | | 0.55 | trace | trace | | trace |

# *TABLE 6B*
# *TBP on Period 13 VSOH*

Fraction Analysis

| | IBP-662°F | 662+ |
|---|---|---|
| Weight % of total | 51.21 | 48.03 |
| Gravity, °API | 10.5 | 0.1 |
| Specific gravity@60°F | 0.9965 | 1.0752 |
| Carbon, W% | 87.55 | 88.46 |
| Hydrogen, W% | 8.93 | 8.06 |
| Nitrogen, W% (P&E) | 0.64 | 0.46 |
| Antek Nitrogen, ppm | 3131.5 | 8584.7 |
| Antek Sulfur, ppm | 4241.6 | 1.91 |
| CCR, W% | | 2.68 |
| Ash (ASTM), W% | | 0.0 |
| Heptane Insol, W%, | | 2.51 |
| Basic Nitrogen, ppm | | |
| Pour Point, °C | -45.0 | -6.0 |
| Freezing Point, °C | -50.0 | -11.0 |
| Aniline Point, °C | 40.6 | TOO DARK |
| Viscosity,cSt@38°C °C (100°F) | 8.04 | |
| Viscosity,cSt@23.3°C (74°F) | 14.83 | cSt@100°C 3.63 |
| Smoke Point, mm | | |
| Copper Corrosion, ASTM D 130 | @ 212°F 1 a | |
| Existent Gum, mg/100mL | 624 | |
| Metals, ppm | | |
| Vanadium | | < 1 ppm |
| Nickel | | < 1 ppm |
| Copper | | 1.2 ppm |
| Iron, W% | | 294.5 |
| PONA Analysis, V% | | |
| Paraffins | 5.53 | |
| Olefins | | |
| Naphthenes | 4.50 | |
| Aromatics | 77.05 | |
| Naphthalenes | 12.92 | |
| Total | 100 | |
| Phenolics content, W% | 35.7 | |

# TABLE 7
# HYDROTREATER PERFORMANCE

HTU Balance (Based on Period 15 and HTU Bypass)
HTU Charged with 1000 cc's of C-411 Catalyst

| Space Velocity (total liquids & gases/hr/catalyst) | | |
|---|---|---|
| /gm of catalyst | 0.84 | |
| /cc of catalyst | 0.75 | |
| | Into the HTU Gms / hr | Exit from the HTU gms / hr |
| Gases | | |
| Hydrogen | 256.0 | 247.4 |
| C1 | 40.6 | 44.2 |
| C2 | 34.3 | 35.1 |
| C3 | 41.8 | 44.6 |
| C4 | 25.3 | 27.5 |
| C5 | 11.8 | 21.8 |
| C6-C7 | 10.4 | 8.9 |
| Cox | 17.9 | 12.7 |
| H2S | 25.0 | 28.6 |
| Liquids | | |
| IBP-350°F | 64.4 | 86.0 |
| 350-650°F | 167.0 | 170.4 |
| 650-850°F | 51.7 | 18.9 |
| TOTAL | 746.0 | 746.0 |
| Liquid Quality | | |
| H/C Ratio | 1.48 | 1.85 |
| API | 21.3 | 36 |
| Sulfur, wppm | 1119 | 58.6 |
| Nitrogen, wppm | 695 | <1 |

# Figure 1

## Bench Unit Simplified Flow Diagram

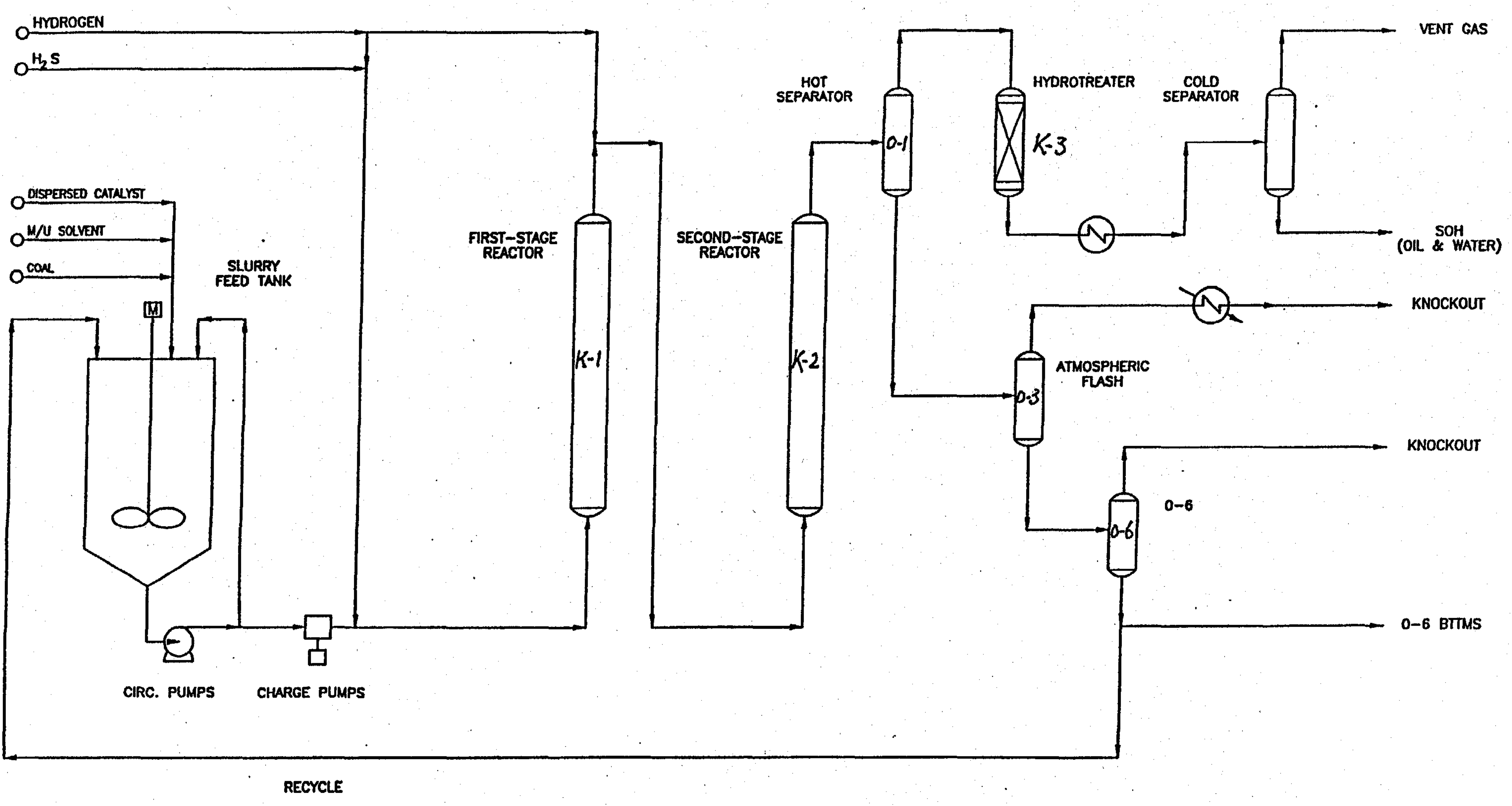


Figure 2

# Condition 1B & 2A (Periods 3B - 9A)

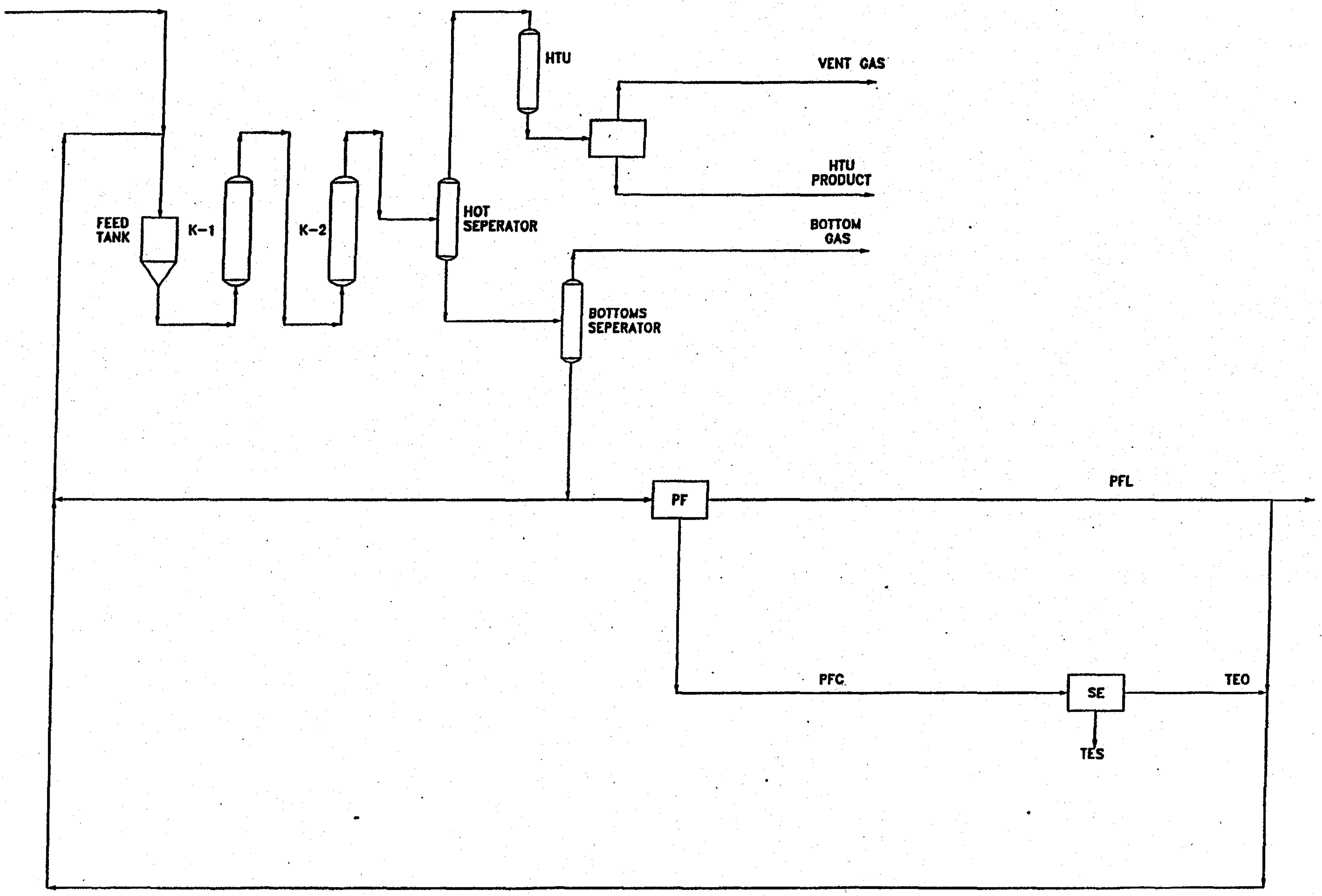


Figure 3

**Condition 2B (Periods 9B - 10B)**

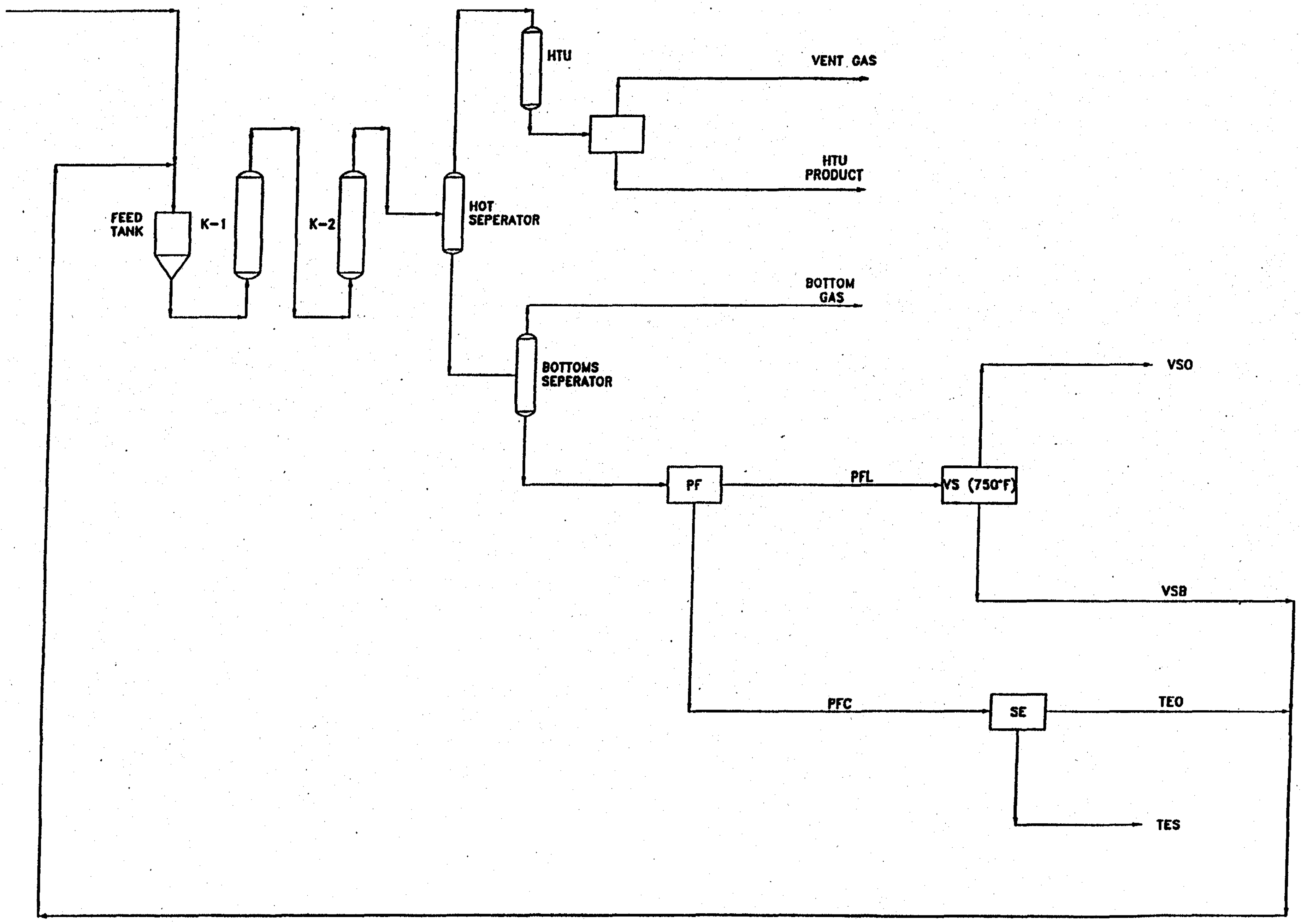


Figure 4

## Condition 3A (Periods 11A - 15B)

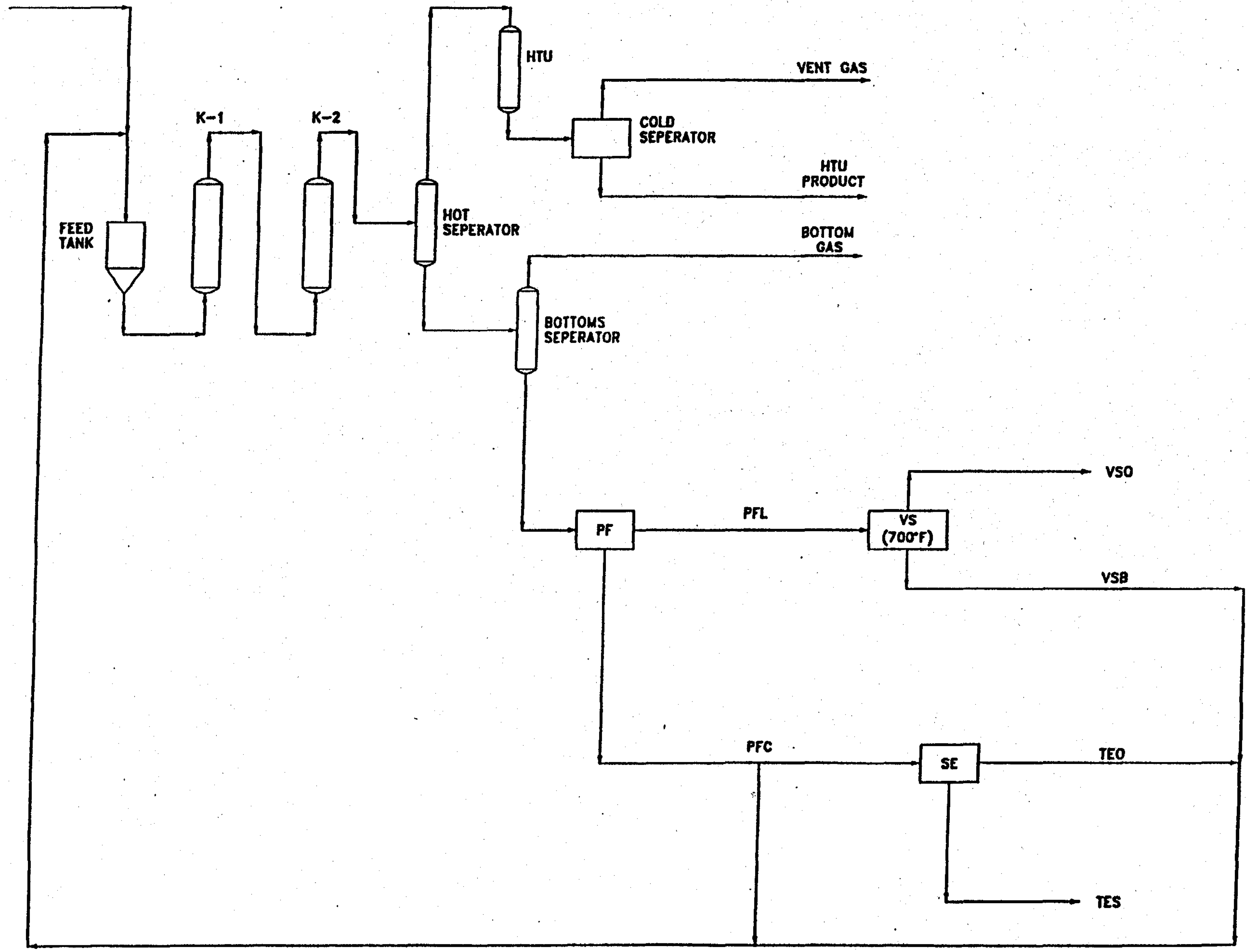


Figure 5

# Condition 4B (Periods 17B - 20A)

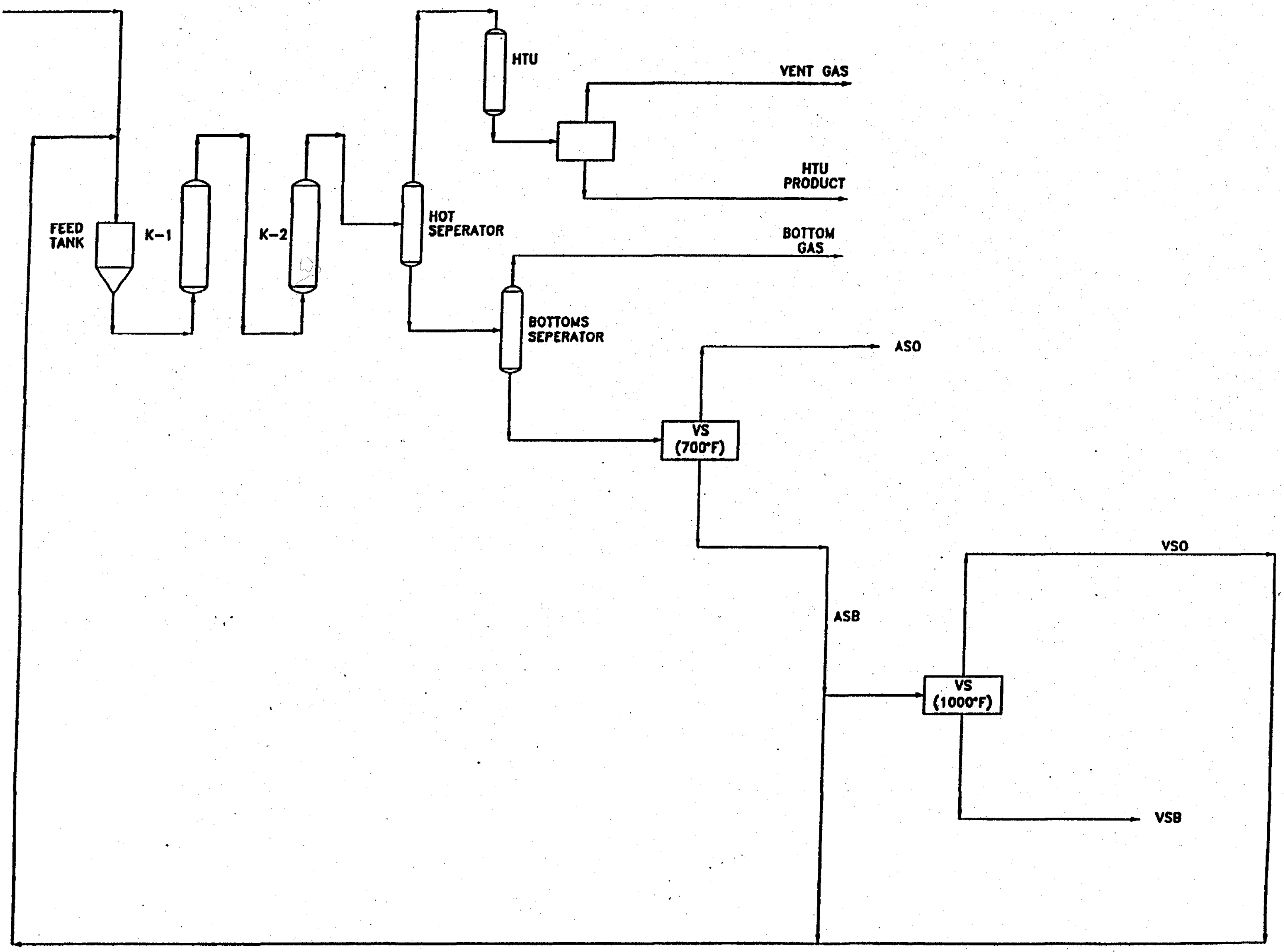
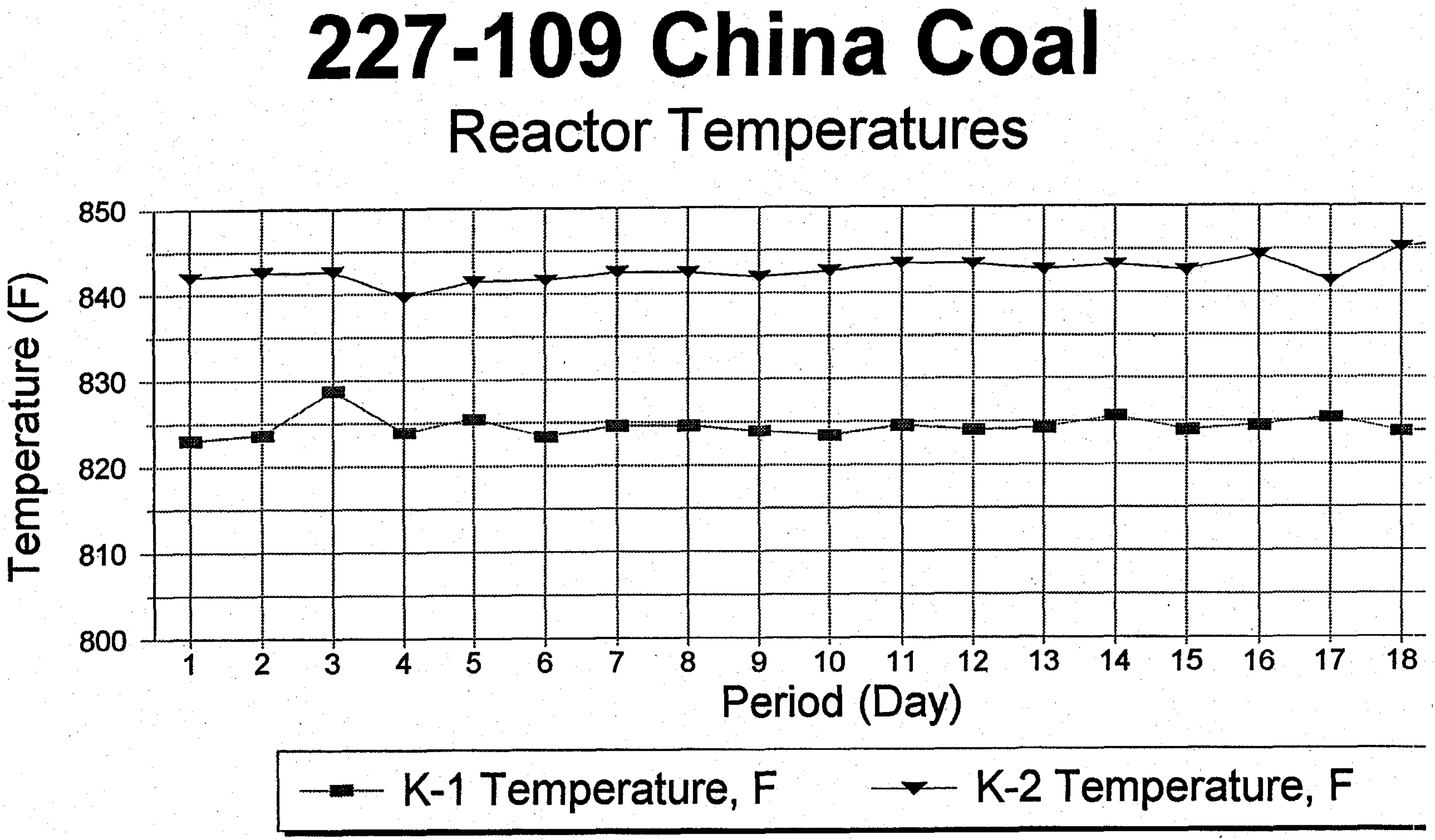
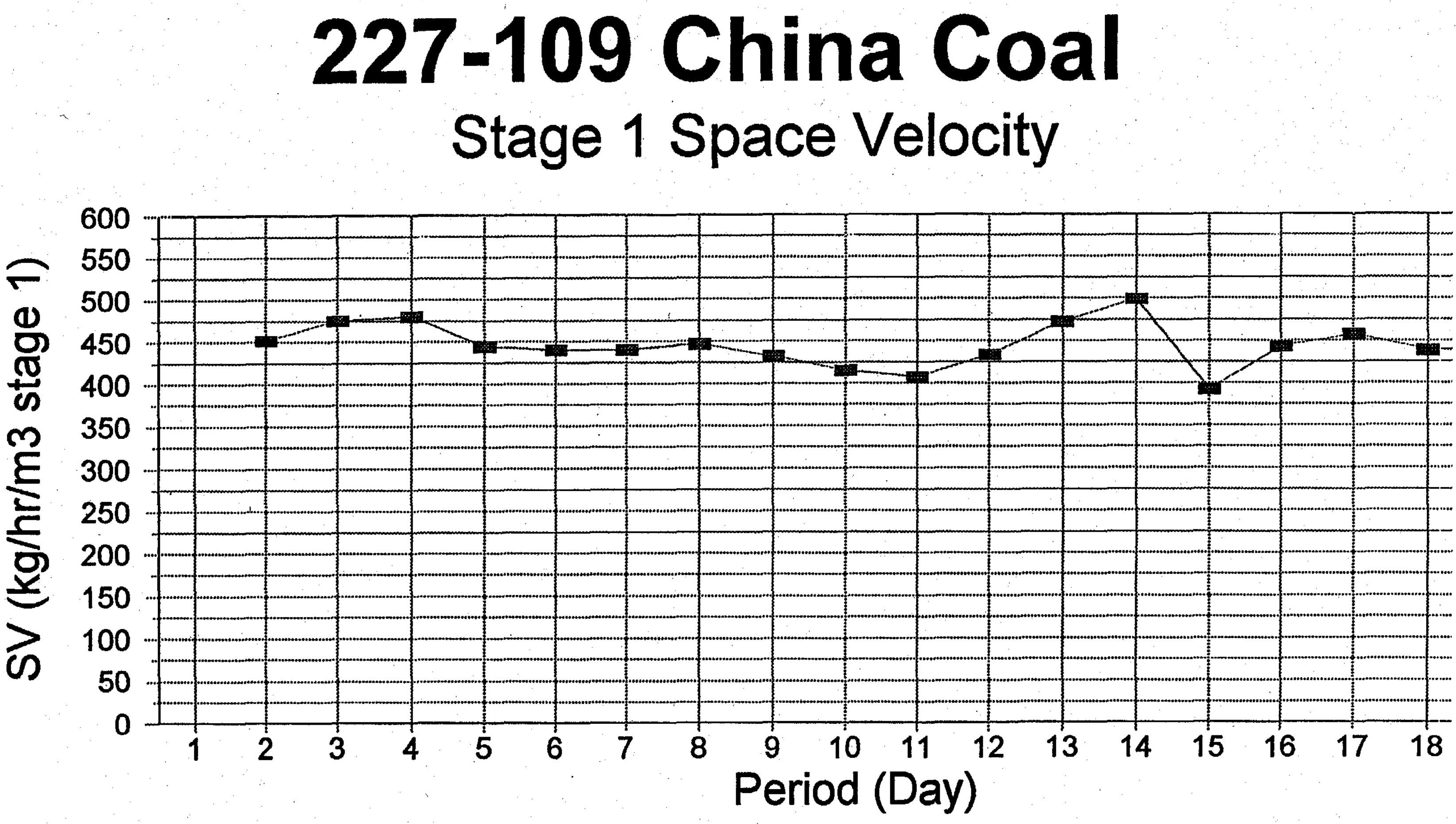


**Figure 6**

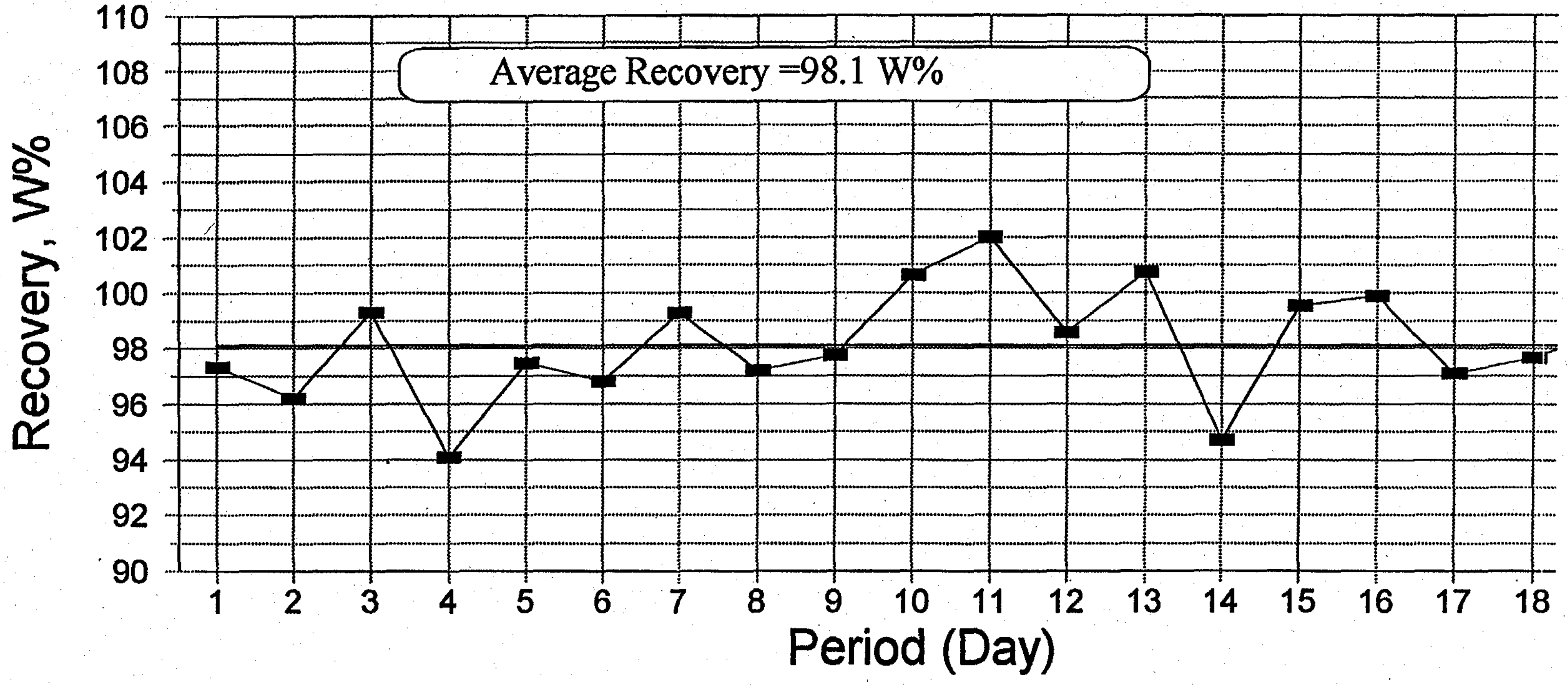

227-109 China Coal
Reactor Temperatures
Temperature (F)
850
840
830
820
810
800
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
Period (Day)
K-1 Temperature, F
K-2 Temperature, F

# Figure 7



227-109 China Coal
Stage 1 Space Velocity
SV (kg/hr/m3 stage 1)
600
550
500
450
400
350
300
250
200
150
100
50
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
Period (Day)

# Figure 8



## 227-109 China Coal

Material Recovery

# Figure 9

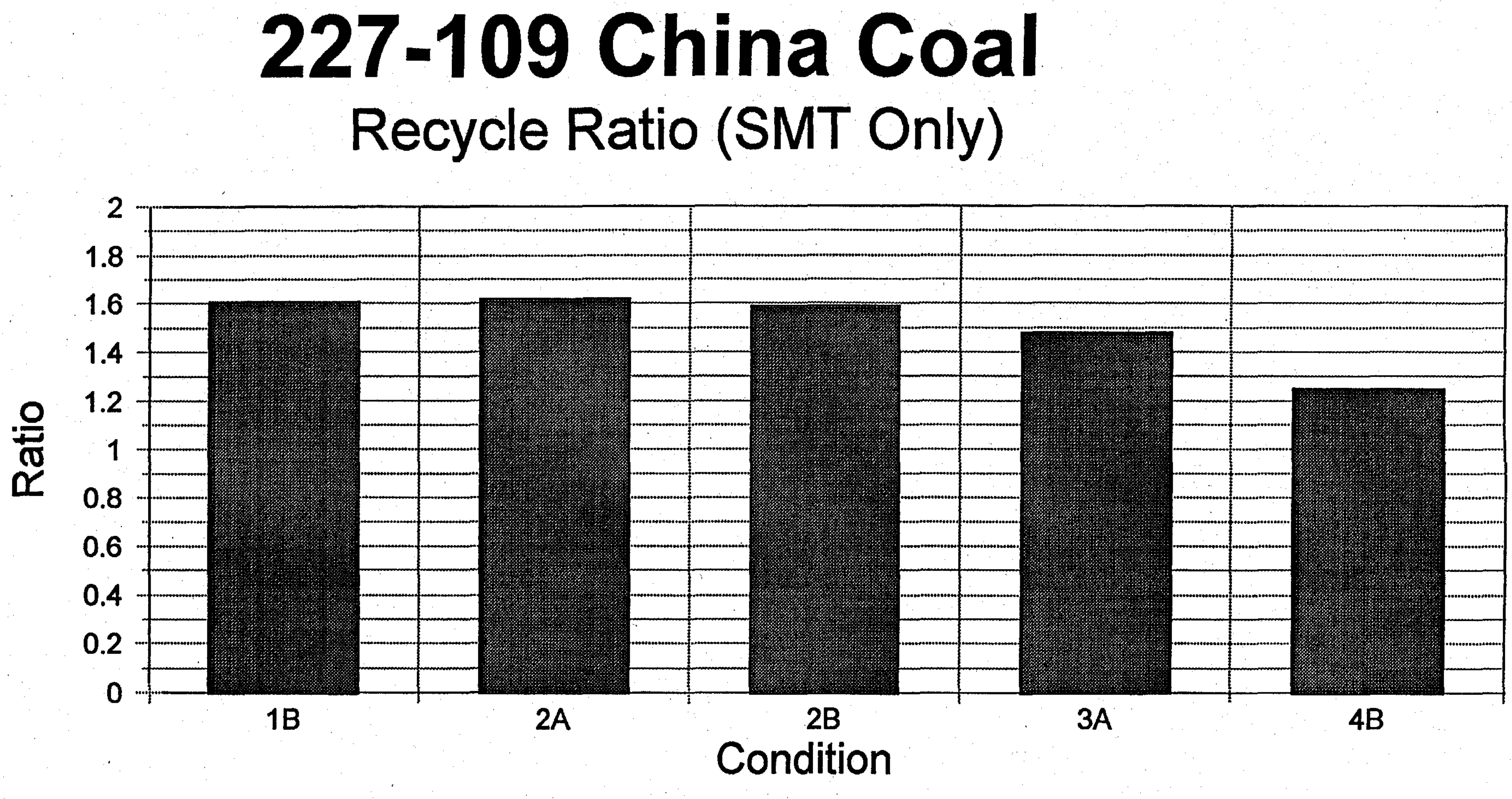

227-109 China Coal
Recycle Ratio (SMT Only)
Ratio
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
1B
2A
2B
3A
4B
Condition

**Figure 10**

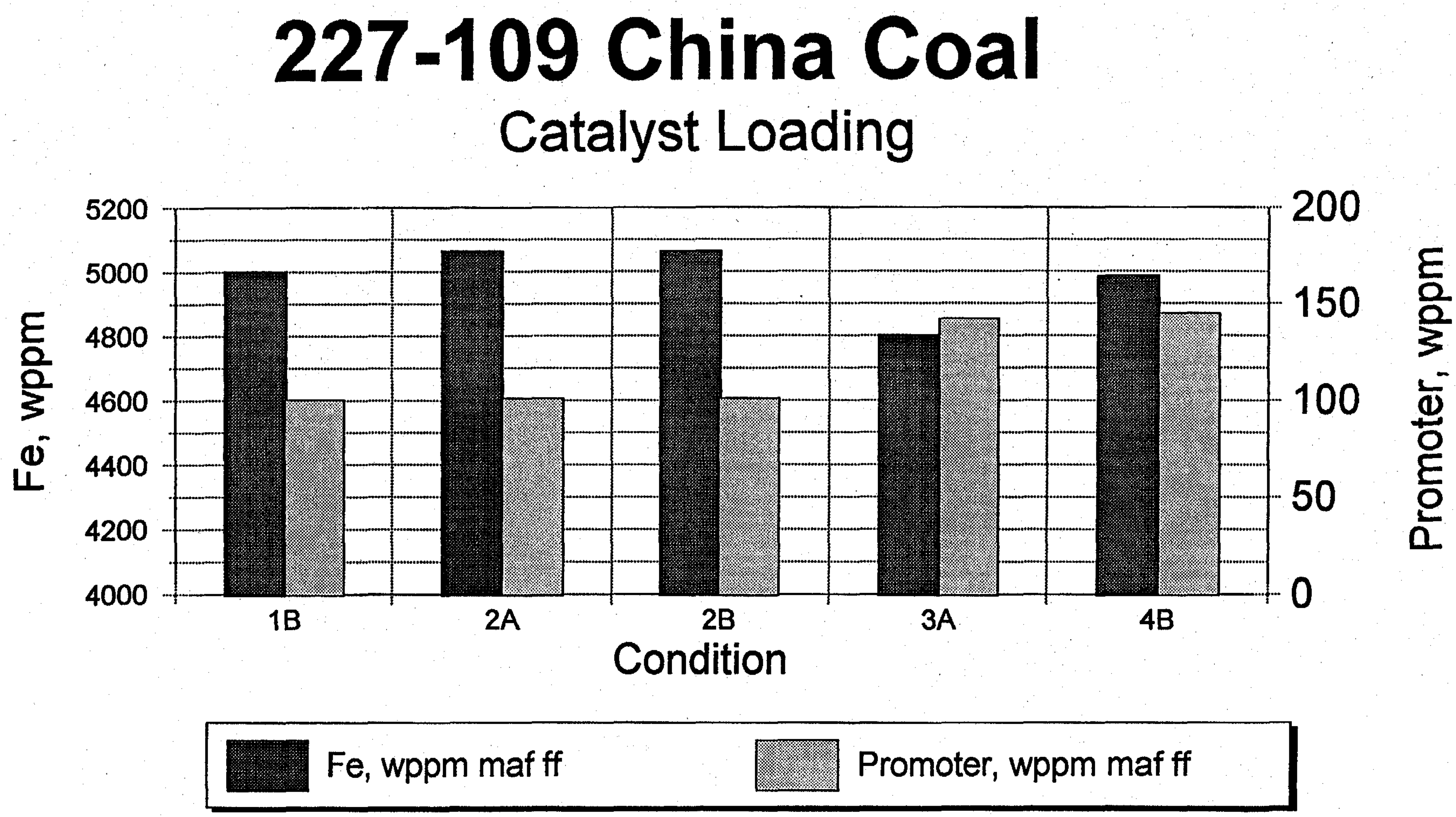
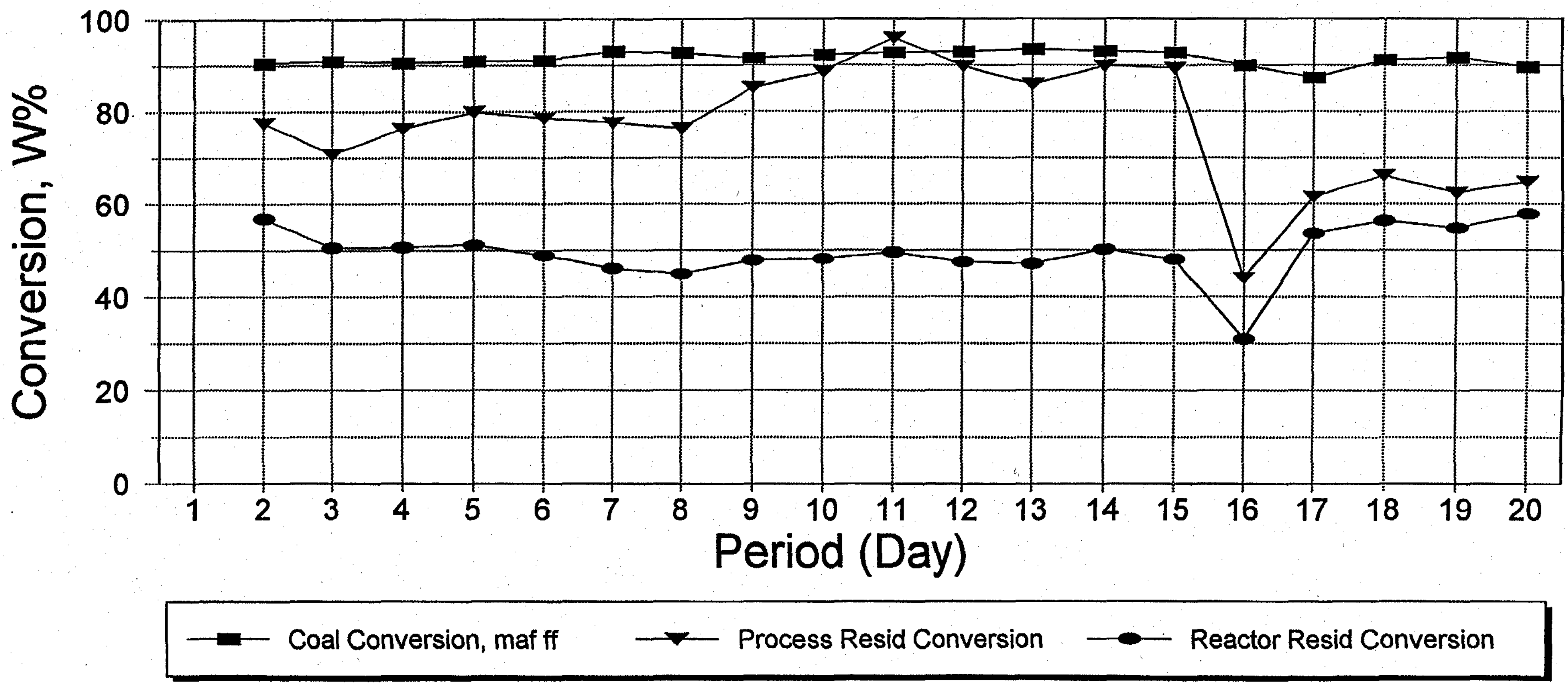

227-109 China Coal
Catalyst Loading
Fe, wppm
5200
5000
4800
4600
4400
4200
4000
Promoter, wppm
200
150
100
50
0
1B
2A
2B
3A
4B
Condition
Fe, wppm maf ff
Promoter, wppm maf ff

Figure 11



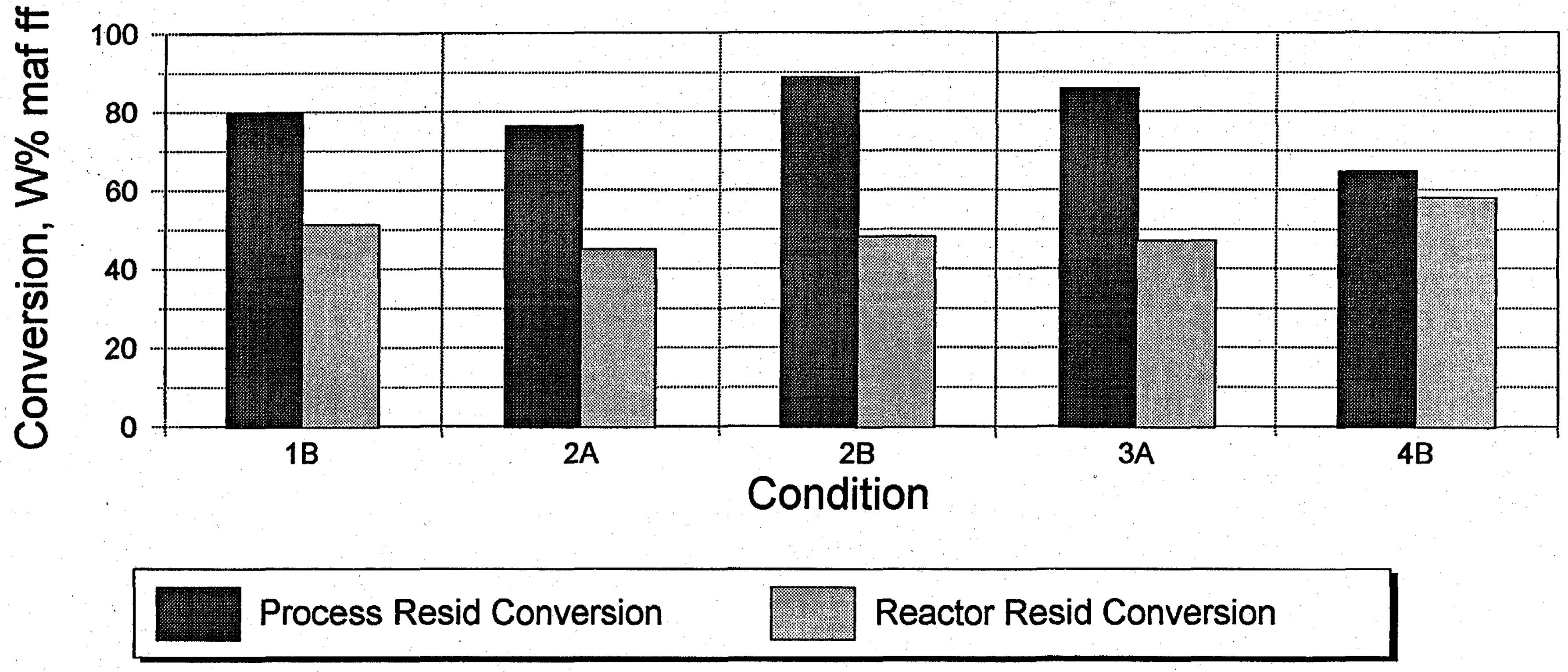
227-109 China Coal

Process Conversions

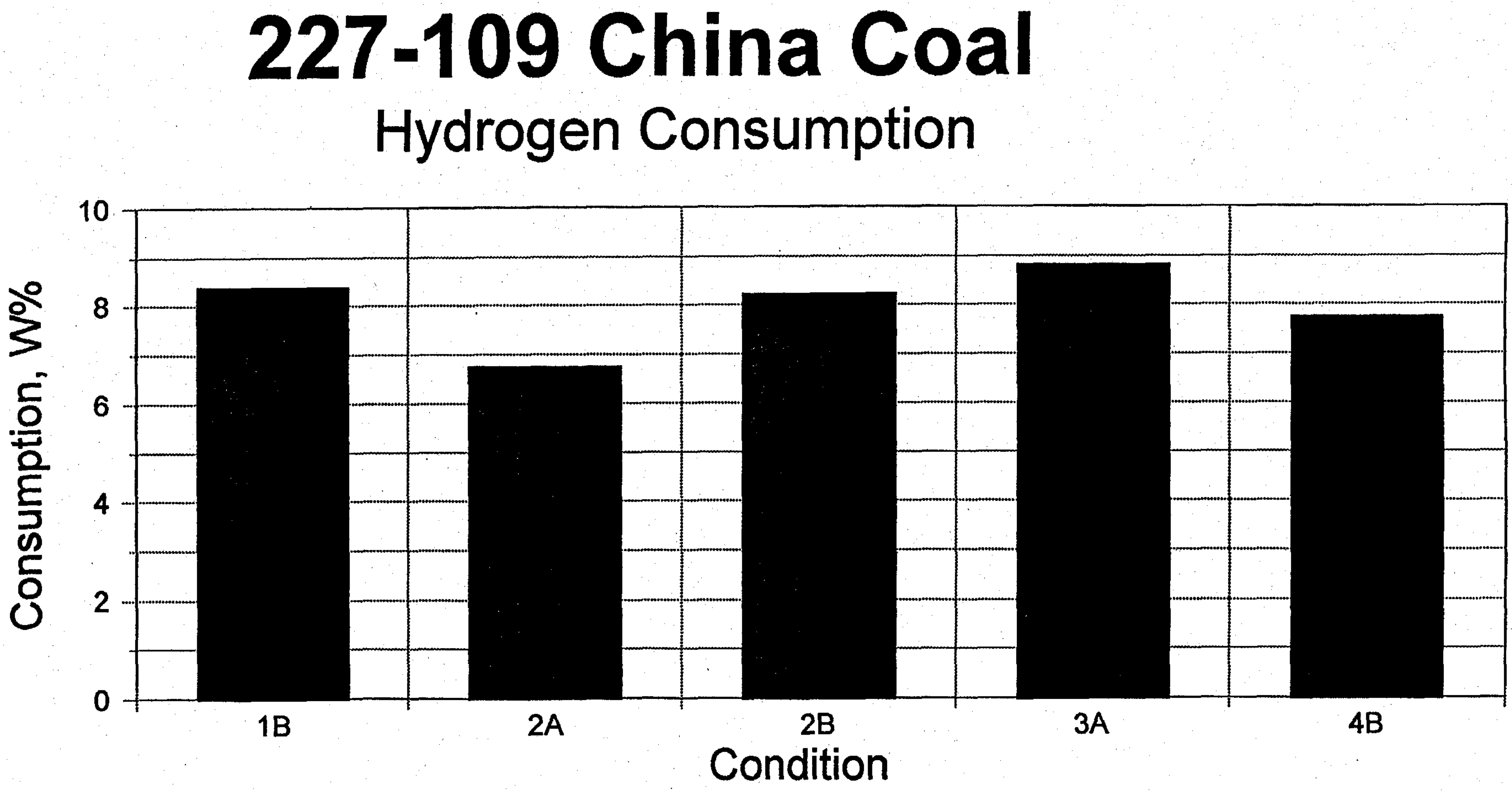
**Figure 12**

## 227-109 China Coal

Resid Conversion



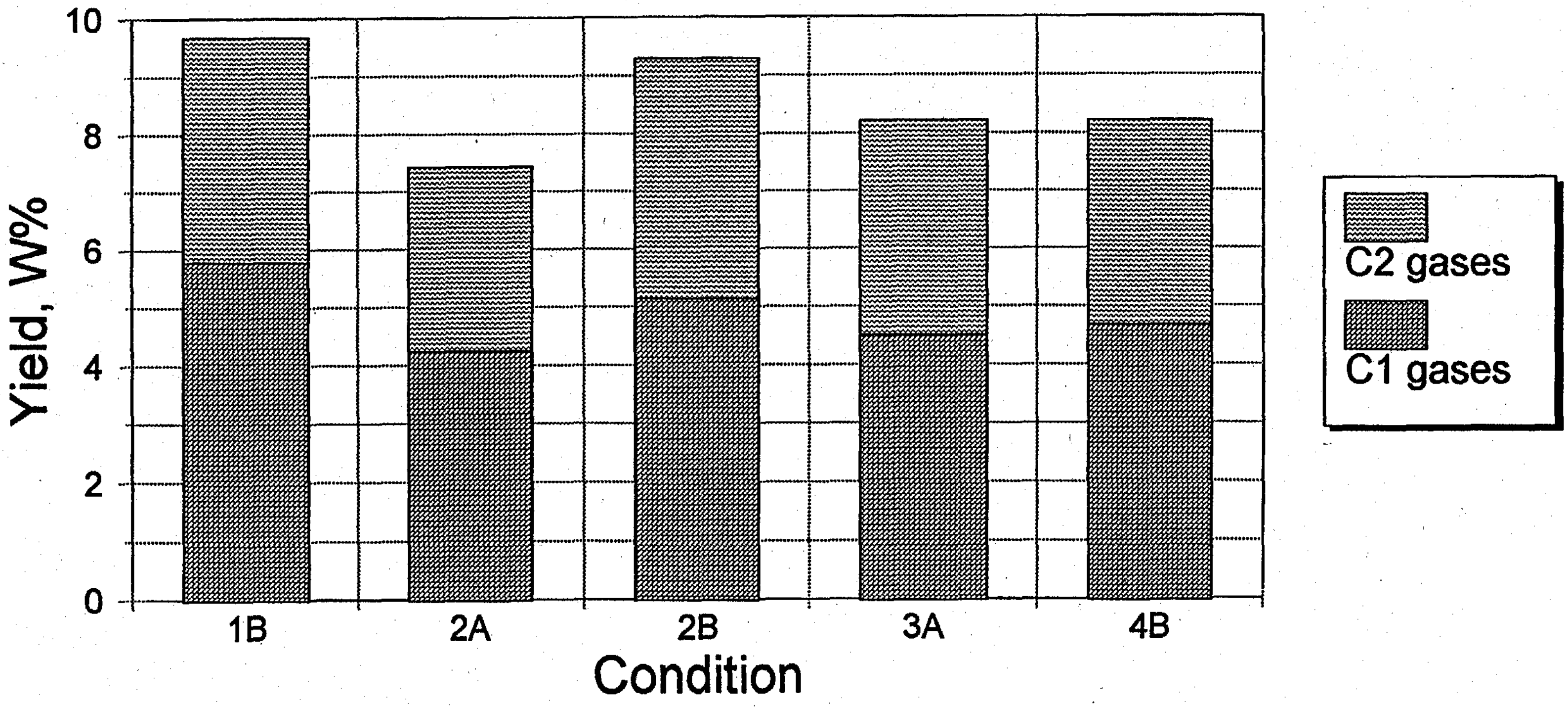
## Figure 13


227-109 China Coal
Hydrogen Consumption
Consumption, W%
10
8
6
4
2
0
1B
2A
2B
3A
4B
Condition

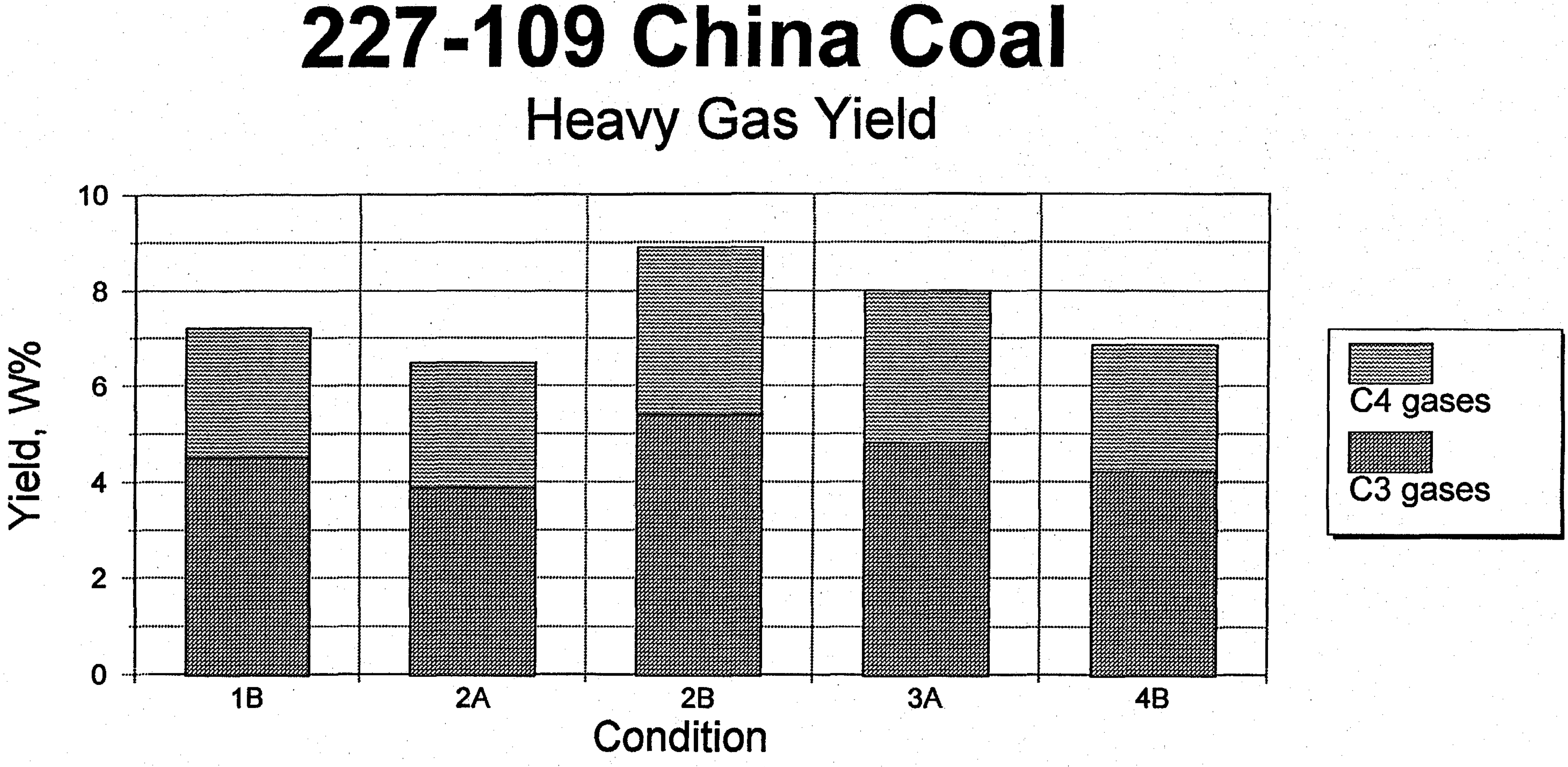
## Figure 14

# 227-109 China Coal

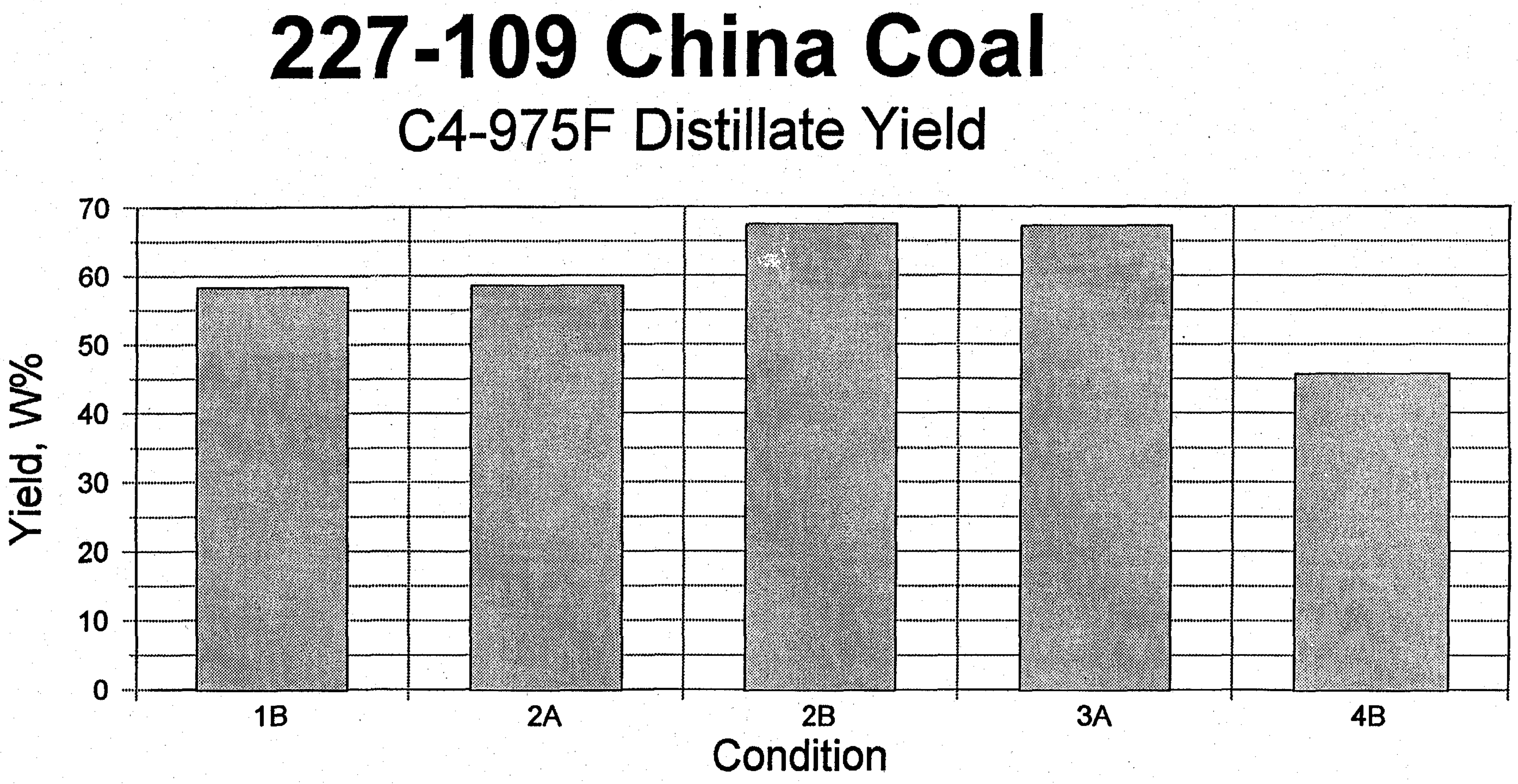
## Light Gas Yield



# Figure 15


227-109 China Coal
Heavy Gas Yield
Yield, W%
10
8
6
4
2
0
1B
2A
2B
3A
4B
Condition
C4 gases
C3 gases

## Figure 16

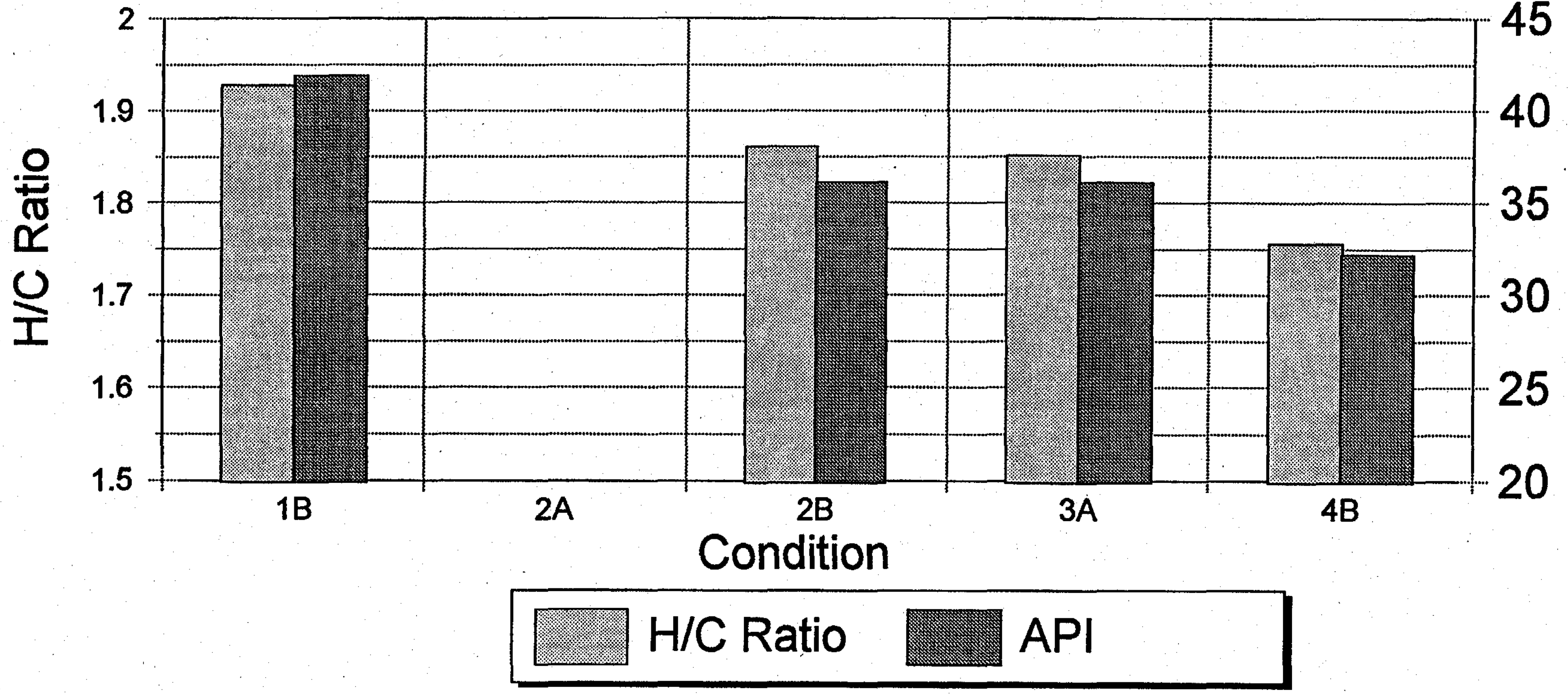


227-109 China Coal
C4-975F Distillate Yield
Yield, W%
70
60
50
40
30
20
10
0
1B
2A
2B
3A
4B
Condition

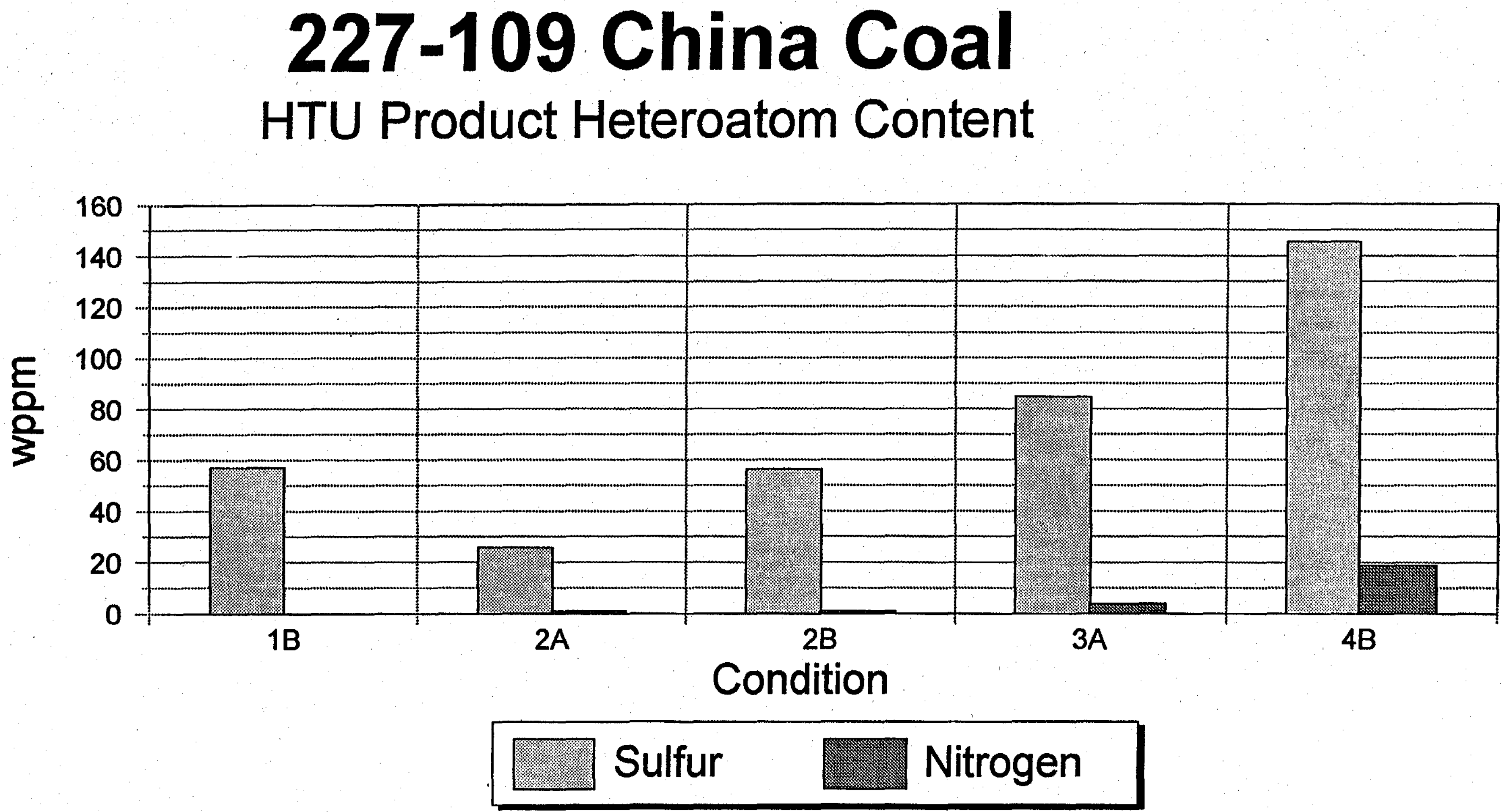
**Figure 17**

## 227-109 China Coal

### HTU Product Quality



## Figure 18



227-109 China Coal

HTU Product Heteroatom Content

**Figure 19**

# 227-109 China Coal

## O-6 Bottoms Quality