DOE/PC 93054-41

A Characterization and Evaluation of Coal Liquefaction Process Streams

Quarterly Technical Progress Report
July 1 through September 30, 1996

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August 1997

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Section 1

EXECUTIVE SUMMARY

CHARACTERIZATION OF SAMPLES FROM HTI RUN ALC-1

CONSOL completed characterization of 64 samples from five run conditions of HTI Run ALC-1 (227-94), in which raw and cleaned (oil-agglomerated at low pH) Black Thunder Mine subbituminous coal was fed and processed using only dispersed catalysts in the liquefaction reactors.

- Extraction of THF-soluble resid from the pressure-filter cakes was more complete when agglomerates were fed, leaving only 5% or less THF solubles in the extracted cakes. When raw coal was fed, the extracted cakes contained 9-34% THF solubles. HTI also observed improved filtration during the periods that agglomerates were fed. Improved operability, if verified by additional work, could be an economically significant benefit of coal cleaning by oil agglomeration at low pH. An apparently higher Mo addition rate (see fourth bullet) may have contributed to the benefits of using oil-agglomerated coal.

- Other stream sample characteristics changed when oil-agglomerated coal was fed in Conditions 2-4, relative to when uncleaned coal was fed in Conditions 1 and 5. The ash content of the O-6 bottoms samples was lower when oil-agglomerates were fed. The THF-soluble 524 $°C^+$ resid concentration in the feed slurry doubled when agglomerated coal was fed. Three factors may have influenced these characteristics. Higher coal conversion would have produced more resid. More efficient toluene-extraction of the filter cake would have recycled more resid. Removal of distillate as product to offset oil fed as part of the agglomerated coal would preferentially recycle the heaviest components.

- The analysis of process samples from Condition 5 indicates that the bench-unit dewaxing operations effectively removed paraffins from the vacuum distillate, and that the wax product is about 75% pure. The low concentration of wax in the feed minimized the impact of dewaxing on solvent quality improvement. The solvent hydrotreating operation effectively increased the hydroaromatic hydrogen content of the distillate and improved its solvent quality.

- CONSOL determined the concentrations of Mo and Fe in feed slurry and pressure filter cake ash samples from Run ALC-1. The Mo and Fe concentration results were combined with HTI material balance data to calculate Mo and Fe balances and determine the apparent addition rates of Mo and Fe catalysts. The resulting Mo and Fe material balance closures were between 80-120%. The results indicate that the Mo addition rate was inadvertently 10-169% higher than the target rate. The highest Mo addition rates were during agglomerated coal feed periods (13, 17, 20). The results show a low Fe addition rate in Period 13 (39% of the target rate), and 4% to 36% higher than design rate in the other periods.

- Although the nominal fresh Fe and Mo catalyst addition rates decreased after Condition 2, there was an improvement in performance during Condition 3. The rate of Fe catalyst addition increased, based on the calculated Fe concentration from observed Fe concentrations in two streams. The observed performance improvement likely resulted from the increase in Fe catalyst concentration.

- Although HTI observed that the in-line hydrotreater (HTU) was less effective than expected in removing nitrogen and sulfur from the second-stage separator overheads (SOH), CONSOL data for all conditions show that this stream was effectively hydrogenated. It was hydrogenated to about the same degree as SOH produced in the coal-only operations during Run CMSL-9, and more hydrogenated than those produced in the coal-only operations during Run CMSL-11. The hydrotreated SOH oils were devoid of phenolic -OH, in spite of high phenolic -OH concentration in the unhydrotreated first-stage SOH oils. These observations suggest that the hydrogenation and phenol removal effectiveness of the catalyst was minimally affected by a distillation system upset early in Run ALC-1 that seriously decreased the N and S heteroatom removal ability of the HTU catalyst.

- Feed slurry samples were found to contain less than 5% 343 $^{\circ}C^{-}$ (650 $^{\circ}F^{-}$) material, contrary to concerns that large amounts of light oil were being recycled.

- About 1% of the feed coal carbon reports to the separator overhead water product. Error resulting from omitting this stream from elemental balance calculations should be small.

RECALIBRATION OF FTIR SPECTROSCOPIC METHOD FOR PHENOLIC -OH DETERMINATION

The Fourier-transform infrared (FTIR) spectroscopic method used to determine the phenolic -OH concentration in liquefaction samples was recalibrated and implemented on a new FTIR instrument. A new calibration was obtained from spectra of standard phenol compounds. An error estimate for each determination was added to the analysis software. The method was validated by comparison of results from samples run on the old and new systems.

OIL ASSAYS OF HTI RUN PB-03 DISTILLATE PRODUCTS

CONSOL arranged to have crude oil assays conducted on the net products of HTI Run PB-03, at DOE's request. Crude oil assays were conducted on net products obtained during periods of Run PB-03 for which the on-line hydrotreater was and was not used.

RESID REACTIVITY

- Resid reactivity tests were performed at CONSOL on the fifteen-resid sample set that the University of Delaware is using in their subcontract. The tests performed at CONSOL are described fully in the Results and Discussion section of this report.

- The University of Delaware made significant progress this quarter. Conversion values for all but two of the resids were obtained. The molecular structure model for coal-derived resids was assembled and is being optimized. A full description of the results is contained in the University of Delaware Quarterly Report appended to this report.

Section 2

INTRODUCTION

This is the Technical Progress Report for the ninth quarter of activities under DOE Contract No. DE-AC22-94PC93054. It covers the period July 1 through September 30, 1996.

CONTRACT OVERVIEW

The objectives of this project are to support the DOE direct coal liquefaction process development program and to improve the useful application of chemical analyses to direct coal liquefaction process development. This project builds on work performed during DOE Contract No. DE-AC22-89PC89883. Independent analyses by well-established methods are obtained for samples produced in direct coal liquefaction processes under evaluation by DOE. New analytical instruments and techniques to examine coal-derived samples are being evaluated. The data obtained from this study are used to guide process development and to develop an improved data base on coal and coal liquids properties. A sample bank, established and maintained for use in this project, is available for use by other researchers. The reactivity of the non-distillable resids toward hydrocracking at liquefaction conditions (i.e., resid reactivity) is being examined. From the literature and experimental data, a kinetic model of resid conversion is being constructed. Such a model will provide insights to improve process performance and the economics of direct coal liquefaction.

CONTRACT ACTIVITIES THIS PERIOD

- Characterization of samples from HTI Run ALC-1 was completed. Results are described in this report.

- The phenolic -OH measurement method was recalibrated for the new FTIR system (Appendix 1).

- Sets of samples were requested from HTI Run PB-05 (Appendix 2).

- CONSOL arranged to have crude oil assays conducted on the net products of HTI Run PB-03, at DOE's request. Crude oil assays were conducted on net products obtained during periods of Run PB-03 in which the on-line hydrotreater was and was not in use. Results are provided in this report (Appendix 3).

- Resid reactivity tests were performed at CONSOL for a fifteen-resid sample set that the University of Delaware is investigating in their subcontract. The tests conducted at CONSOL are described in the Results and Discussion section of this report.

- The University of Delaware has made significant progress this quarter. Conversion values for all but two of the resids were obtained. The molecular structure model for coal-derived resids was assembled and is being optimized. A description of accomplishments is in the University of Delaware Quarterly Report appended to this report (Appendix 4).

ACTIVITIES IN PROGRESS

- Characterization work was started on samples from HTI Run PB-04. In HTI Run PB-04, Black Thunder Mine coal, Hondo resid, auto shredder residue, and other plastics were used as feedstocks.

- Plans were made to begin a literature search and experimental work on the synthesis of ethyl phenyl ethers (for high-octane oxygenate extenders for transportation fuels) from coal liquefaction phenols. Reagents were ordered.

- The University of Delaware began a parametric study of resid conversion. Variables are time and temperature. The study will initially focus on the reactivity of two resids (Wilsonville Run 259, V131B, and Wilsonville Run 260, V131B).

Section 3

RESULTS AND DISCUSSION

CHARACTERIZATION OF SAMPLES FROM HTI RUN ALC-1

INTRODUCTION

CONSOL completed characterization of 64 samples from HTI Run ALC-1 (227-94), in which raw and cleaned Black Thunder Mine subbituminous coal was fed and processed using dispersed catalysts in the liquefaction reactors. The run consisted of 25 days of operation at five run conditions. Operating conditions and yields and performance data are provided in Table 1.[1-3] The run background is described briefly below. Additional run information is presented elsewhere.[1-5]

RUN OBJECTIVES AND HISTORY

There were four main objectives for Run ALC-1: 1) to provide a baseline operating period with Black Thunder Mine subbituminous coal, 2) to demonstrate liquefaction of low ash coal produced by low pH oil agglomeration of Black Thunder Mine coal, 3) to demonstrate liquefaction of Black Thunder Mine coal with dewaxing and hydrotreatment of distillate solvent, and 4) to operate with extinction recycle of 343 $°C^+$ material, such that a hydrotreated light distillate would be the intended net product. An additional objective was to exploit the advantage of feeding low-ash coal agglomerates by decreasing the fresh catalyst make-up rate.

HTI's bench liquefaction Run ALC-1 consisted of 25 days of operation. Major accomplishments were:

- Oil agglomeration reduced the ash content of Black Thunder Mine coal by 40%, from 5.5% to 3.3% (MF, SO_3-free ash basis).

- Excellent coal conversion (98%) was obtained with oil agglomerated coal (about 3% higher than the raw Black Thunder Mine coal), which increased the potential product yield by 2-3% on an MAF coal basis.

- Agglomerates were liquefied with no handling problems. Filtration performance was improved.

- Fresh catalyst (nominal) make-up rate was decreased by 30%, with no apparent detriment to operations, when agglomerates were fed or when raw coal was fed (with solvent dewaxing and hydrotreating).

- Recycle solvent treatment by dewaxing and hydrotreating was demonstrated, but steady-state operation was not achieved.

- There was some success in achieving extinction recycle of the heaviest liquid products. This was evident in decreased resid yields and increased yields of light distillate during some run conditions.

- High yields of light distillates were obtained at several conditions during the run.

RUN PLAN AND OPERATING HISTORY FOR ALC-1

Run ALC-1 operating conditions, yields, and process performance results are shown in Table 1. The fresh catalyst addition rates for Conditions 1 and 2 matched those used in Run CMSL-9, Condition 6. The fundamental recycle strategy planned for Run ALC-1 was to recycle to extinction all materials that boil above 343 °C, except for the rejected, washed, pressure-filter cake. In practice, equipment and operability constraints did not allow this. The intent was to adjust space velocity to extinct the yield of 343 $°C^+$ material. Pressure filtration was used to reject solids; the pressure-filter cake was washed with toluene to minimize rejection of solubles. The recycle solvent (including buffers) to MF coal ratio was 1.60, and the recycle solids to MF coal ratio was 20%. Both of these are higher than typically used in recent runs at HTI. The run ALC-1 conditions were designed to provide for high catalyst and unconverted coal recycling rates, which was intended to improve coal conversion, yet maintain operability with the high solids content in the recycle stream.

In Condition 1, the continuous atmospheric still bottoms (CASB) stream was filtered, and the recycle stream consisted of pressure-filter liquid (PFL) and a portion of the toluene-washed pressure-filter cake (PFC). The initial space velocity was 670 kg MF coal/h/m^3. To shorten the solids recirculation time, it was decided near the end of Condition 1 to recycle unwashed PFC directly.

In Conditions 2, 3, and 4, oil agglomerated coal was used as the feedstock. The agglomerating oil used to prepare the ALC-1 feed was heavy recycle distillate (V1074) from the Wilsonville pilot plant Run 263 end-of-run inventory. For Conditions 2, 3, and 4, it was necessary to reduce the quantity of distillate in the recycle by an amount equal to the agglomerating oil fed with the coal (as fed to liquefaction, the agglomerates contained about 23% oil). All of the PFL was sent to the vacuum still. All of the vacuum still bottoms (VSB) and some of the vacuum still overhead (VSOH) were recycled as necessary to meet total recycle requirements. A quantity of VSOH, approximately equal to the distillate agglomerating oil added with the coal, reported as product.

Early in Condition 2, the distillation conditions and flow scheme were changed to maintain operations with the constraints imposed by the run objectives. These changes resulted in a lighter recycle and heavier product than desired. The space velocity was decreased from 670 kg MF coal/h/m^3 to 561 kg MF coal/h/m^3 early in Condition 2. In Condition 3 (feeding agglomerates), the make-up catalyst rate was cut by 30%, relative to Conditions 1 and 2. Other distillation system changes were made to minimize recycle of light material. In Condition 4, the only operating condition changed was space velocity, which was decreased to 400 kg MF coal/h/m^3 to reduce the yield of 343 °C$^+$ material.

The transition was made back to raw Black Thunder Mine coal in Condition 5. For this condition, which included dewaxing and hydrotreating, it was decided to keep the catalyst make-up rate at 70% of the Condition 1 value, and to set the space velocity at 481 kg MF coal/h/m^3 reactor. The entire PFL stream was vacuum distilled at 524 °C to produce VSOH for dewaxing/hydrotreating. This material was ketone-dewaxed to remove paraffins, and the dewaxed oil then was hydrotreated to improve the donor solvent quality prior to recycle.

GENERAL SAMPLE CHARACTERISTICS

Sample characteristics are presented in Tables 2-6 and Figures 1-7. The aromatic hydrogen content of several whole liquid sample streams or filtered solids-containing whole sample streams is shown in Figure 4. Numerous changes made to distillation conditions and configurations during the run prevent direct comparison of vacuum distillate and pressure-filter liquid (PFL) streams. The streams shown in Figure 4 increase in aromaticity in the following order:

stage 2 separator overhead (SOH) oil < stage 1 SOH oil < feed slurry = interstage slurry < O-6 bottoms = pressure-filter cake (PFC).

During the run, period-to-period variation in aromatic H content was generally small (Figure 4). However, for the feed slurry and interstage slurry streams, the Period 5-6 samples contained about 5% to 8% more aromatic H than the Period 13-21 samples, which in turn contained about 5% more aromatic H than did the Period 25 samples. The aromatic H differences are probably associated with use of raw (Periods 1-6 and 21-25) vs. oil-agglomerated (Periods 7-20) coal, use of different space velocities in several run periods (space velocity was changed three times during the run), and use of recycle solvent dewaxing and hydrogenation in Periods 21-25. Analogous, but inverted, trends were observed for the corresponding paraffinic H content of these same samples (Figure 5). The same pattern of high aromatic H content in the Period 5-6 samples, intermediate aromatic H content in the Period 13-20 samples, and lower aromatic H content in the Period 25 samples also holds for other sample types (Table 3), such as the IBP-454 °C (IBP-850 °F) distillate portion of the O-6 bottoms samples, and the 454-524 °C (650-975 °F) distillate portion of the feed slurry samples. The corresponding IBP-454 °C (IBP-850 °F) portion of the O-6 bottoms samples, and the 454-524 °C (650-975 °F) resid portion of the feed slurry samples show higher aromatic H in period 6, but nearly equivalent aromatic H for periods 13-25 (Table 4).

The phenolic -OH concentration of process streams (Table 5, Figure 7) decreased according to the trend:

first stage SOH > feed slurry = O-6 bottoms = PFC > product SOH (second stage SOH)

No phenolic -OH was detected in the second-stage SOH samples. The phenolic -OH concentration in the feed slurry, O-6 bottoms, and PFC streams decreased slightly as the run progressed. The phenolic -OH concentration in the first stage SOH increased after period 6, and then decreased for the remainder of the run. It is not clear why this particular trend is observed. The large increase after period 6 may be related to the increase in resid concentration in the feed slurry while agglomerates were fed (see later section). It was observed that the first stage SOH phenolic -OH concentration was inversely proportional to H_2 consumption on a MAF basis (% H_2

consumption = -2.40*phenolic -OH concentration + 11.2, standard error of Y_{est} = 0.13, R^2 = 0.981).

SOH OIL CHARACTERISTICS

As expected for normal hydrotreater operation, the second-stage SOH oils (the in-line hydrotreater product oils) are lower in aromatic H content and higher in paraffinic H content than the first-stage SOH oils. HTI indicated that the HTU was not operating at optimum performance during Run ALC-1, apparently because a distillation system upset early in the run caused some higher-boiling material to enter the HTU, decreasing the activity of the catalyst. Although HTI observed that the in-line hydrotreater was less effective than expected in removing nitrogen and sulfur from the second-stage SOH, CONSOL data (Table 3) show that the aromatic, hydroaromatic, and paraffinic hydrogen contents of these samples were generally similar to those produced in the coal-only operations during Run CMSL-9.[6] The Run ALC-1 product (second-stage) SOHs also were less aromatic (more hydrogenated) than those produced in the coal-only operations during Run CMSL-11.[7] These observations suggest that the hydrogenation effectiveness of the catalyst was minimally affected by the event early in Run ALC-1 that seriously decreased the N and S heteroatom removal ability of the HTU catalyst.

The hydrotreated product (second stage SOH) oils from HTI Run ALC-1 were devoid of phenolic -OH, in spite of high phenolic -OH concentration in the unhydrotreated first-stage SOH oils and of higher-than-normal concentrations of nitrogen and sulfur in the hydrotreated oil. The phenol removal activity, like the hydrogenation activity, evidently was not significantly impaired by the event that impaired the nitrogen and sulfur removal activity of the catalyst. The high phenolic -OH concentration of the unhydrotreated first stage SOH oil indicates that it contains a high concentration (perhaps 25%) of phenolic compounds, and is a potential source of phenolic chemicals.

ELEMENTAL ANALYSIS OF THE SOH WATER

Separator overhead (SOH) water samples from Stage 1 and Stage 2 were analyzed for pH and weight percent C, N, and S (Table 7) for HTI's elemental balances. Relative to the Stage 1 samples, the Stage 2 samples had higher pH and S and N concentrations, and lower C concentration. At the SOH water product flow rates in Run ALC-1 (about 14 kg/day), the carbon content in the water is about 210 g/day (depending on the production split between the two stages). At the coal feed rate of Run ALC-1 (about 26 kg/day), the coal carbon feed rate is about 16.6 kg/day. Based on this analysis, about 1 wt % of the coal carbon and somewhat greater proportions of the coal sulfur and nitrogen reported to the SOH water. It appears that any error resulting from omitting this stream from elemental balance calculations should be small.

The phenolic compounds in one SOH water sample (Period 13B, first stage) were qualitatively characterized by GC/MS analyses. The procedure used was acidification of the sample with HCl from a pH of about 9 to a pH of about 1 and extraction of the tar acids with methylene chloride. The appearance of the liquid changed from clear, bright yellow to colorless and slightly turbid upon acidification. Odors detected during acidification (in chronological order) included H_2S, SO_2, and phenols. Only phenol and the cresols were identified by GC/MS analysis of the tar acids.

FEED SLURRY CHARACTERISTICS

There had been concern that significant amounts of light oils were recycled during Run ALC-1, due to inefficient distillation and other operating conditions. To address this concern, feed slurry samples from each condition of Run ALC-1 were distilled to 343 °C (650 °F) and 524 °C (975 °F). The resid was extracted with tetrahydrofuran, and ashed to determine other components as shown in Table 8. An important result from this characterization is that only a small quantity of 343 $°C^-$ (650 $°F^-$) material was recycled in Run ALC-1 ($\leq$5% of feed slurry). This presumes that no light material was lost before the samples were shipped to CONSOL.

CONSOL data (Figure 1, Tables 2 and 8) indicate that the feed slurry during Conditions 2-4 (Periods 13, 17 and 20) contained about twice as much THF-soluble $524°C^+$ (975 $°F^+$) resid as the feed slurry from Conditions 1 and 5 (Periods 6 and 25). The resid content of the Period 5 feed slurry was very high, for no apparent reason. Perhaps the higher concentration of higher-boiling material in the feed slurry contributed to the lower distillate yields obtained when oil-agglomerated coal was fed. Three factors may relate to the higher resid concentration in the feed slurry when oil-agglomerated coal was fed in Conditions 2-4. First, the higher coal conversion obtained when agglomerates were fed could result in a higher concentration of resid in the recycle streams. Second, removal of an amount of distillate roughly equivalent to that fed with the agglomerated coal may have left only the higher-boiling material for recycle. In fact, the recycle strategy for the entire run was to take the lowest-boiling material as product; perhaps this degree of selectivity only became available with operating-condition changes that came into effect when oil-agglomerated coal was fed. The third possible factor is more efficient extraction of soluble resid from the PFC when oil agglomerates were fed (discussed below). This would increase the availability of soluble resid for recycle, although the quantitative effect on feed slurry composition was not considered. The recycle stream composition was controlled by the total recycle rate and solids recycle rate; soluble-resid recycle rate was not directly controlled by the plant operators.

CONCENTRATIONS OF Mo AND Fe IN FEED AND PRODUCT STREAMS

Mo and Fe concentrations in the second-stage pressure-filter cake (PFC) samples from Run ALC-1 were determined. They are presented in Table 9 on three bases: (1) metal oxide as weight percent of the SO_3-containing ash; (2) element as a weight percent of the SO_3-free ash; and (3) element as a weight percent of the SO_3-containing ash. The CONSOL Mo concentrations agree with those reported by HTI for the second-stage pressure-filter cake. However, many of CONSOL's Fe concentrations are 20% (or more) higher than those reported by HTI.

Mo and Fe concentrations were determined in the 524 $°C^+$ (975 $°F^+$) resids from all of the Run ALC-1 feed slurry samples (Table 10). The data provide a direct measurement of the total Mo and Fe concentrations in the reactor feed. The concentrations of the metals in the ash agree well with the pressure filter cake (PFC) results reported in Table 9. Elemental Mo concentrations for the entire set (PFC and feed slurry) ranged from 0.19 to 0.47 wt % of the SO_3-free ash, and approximately doubled when agglomerated coal was fed (partly in response to the reduced ash content of the agglomerated coal). Fe concentrations for the entire set ranged from 17.9 to 28.1 wt % of the SO_3-free ash, and decreased from Condition 2 through the end of the run.

The Mo and Fe concentrations were combined with HTI material balance data to calculate Mo and Fe balances and to determine the apparent Mo and Fe catalyst addition rates. Intermediate and final calculated results are shown in Table 11. The following assumptions were used in the calculations: raw coal SO_3-free ash content of 5.50% MF; agglomerated coal SO_3-free ash content of 3.30% MF; added Fe_2O_3 was 1.4% or 0.98% of the MF coal rate (for calculating total ash fed to system); Mo in the raw or agglomerated coal was ignored (the MF raw or agglomerated coal contains ca. 2 ppm Mo); the Fe_2O_3 content of the raw coal SO_3-free ash was 6.66%; and the Fe_2O_3 content of the agglomerated coal SO_3-free ash was 9.57%. The data on which the calculations are based were obtained from this report and other reports.[1,4-5]

The Mo and Fe balances were calculated for each test period shown as the ratio of total grams of Mo or Fe out to the total grams of Mo or Fe in. Total Mo or Fe in was obtained by multiplying the weight percent Mo or Fe in the feed slurry SO_3-free ash by the grams of ash fed in that period in the fresh coal and fresh iron catalyst, and in the pressure-filter cake (PFC) recycle. The amount of ash fed in the fresh coal was obtained from the amount of MF coal fed (HTI material balance data) and the SO_3-free ash content of the raw or agglomerated coal. The ash fed in the added Fe catalyst was obtained as 1.4 wt % of the amount of MF coal fed (for the 10,000 mg/kg Fe target addition rate), or as 0.98 wt % of the amount of MF coal fed (for the 7,000 mg/kg Fe target addition rate). The ash fed in the recycle PFC was obtained from HTI's recycle ratio of insoluble organic matter (IOM) plus SO_3-free ash to MF coal, and HTI's measurements of IOM and SO_3-free ash contents of the PFC. The total amount of Mo or Fe out was obtained by multiplying the wt % Mo or Fe in the PFC by the grams of total PFC produced for the period (HTI material balance data).

Apparent catalyst addition rates were obtained by subtracting the amount of Mo or Fe in the recycled PFC from the total amount of Mo or Fe fed in the feed slurry. The rates were calculated on an MF coal basis. In the Fe case, the coal Fe contribution was backed out, so that the results represent only added Fe. The ca. 2 mg/kg Mo in the raw coal was not backed out.

The resulting Mo balance closures were 80-119%, and the Fe balance closures were 98-120%. The balances were generally good; that is, there was not a consistent or gross imbalance between feed and product rates. These balances generally verify the validity and consistency of the analytical measurements. The calculated apparent Mo catalyst addition rates (MF coal basis) were 110-269 mg/kg Mo when the target Mo concentration was 100 mg/kg, and 94-161 mg/kg when the target rate was 70 mg/kg Mo. The calculated apparent Fe catalyst addition rates (MF coal basis) were 3,928-13,642 mg/kg Fe when the target Fe concentration was 10,000 mg/kg, and 7,309-8,571 mg/kg when the target rate was 7,000 mg/kg Mo.

These results indicate that the Mo addition rate was 10-169% in excess of the target rate. The excess amount seemed to be largest during periods when agglomerated coal was fed. The generally good Mo balance suggests that the Mo addition rate was inadvertently high, rather than being caused by introduction of Mo from a spurious source. Any spurious source of Mo would have to introduce a high Mo concentration in the feed slurry to be consistent with these results.

The results show that the Fe added in Period 13 (Condition 2) was only 39% of the target rate, but in the other periods was 4-36% in excess of the target rate.

EFFECT ON PROCESS STREAM PROPERTIES OF FEEDING COAL CLEANED BY OIL AGGLOMERATION AT LOW pH

The ash content of the O-6 bottoms stream was lower when agglomerates were fed in Conditions 2-4, Periods 13, 17, and 20 (9.3-9.8%, vs. 11.3-12.7% in Conditions 1 and 5). A similar but less pronounced pattern was observed in the feed slurry stream (6.9-7.8% vs. 8.2-8.9% in Conditions 1 and 5). The lower ash concentrations in the samples seem to be related to the lower ash content of the agglomerated feed coal, but the relationship is not completely straightforward, especially for the O-6 bottoms samples. Although the feed coal ash content was lower in the oil-agglomerates, the addition rate of Fe-based catalyst was changed, which contributes significantly to the ash content of the total feed. There is also a significant rate of ash recycle, which is larger than the feed rate of ash from the fresh coal. The ash recycle rate was held roughly constant, although changes that result from feeding agglomerates (such as an increase in coal conversion) would be expected to affect the recycle ash rate.

Extraction of THF-soluble 524°C^+ (975 °F^+) resid from the PFCs was more complete when oil-agglomerated coal was fed in Conditions 2-4 (Periods 13, 17, and 20, Figure 3). When raw coal was fed (Periods 6 and 25), 9-34% solubles remained in the toluene-extracted PFCs. When oil-agglomerated coal was fed, only 5% or less THF-solubles remained in the toluene-extracted PFCs. HTI's data for hot-quinoline solubles confirm this effect.[1] Perhaps the improvement in extraction efficiency was related to the improved filtration observed when agglomerates were fed.[8] If it can be demonstrated that oil agglomeration at low pH improves liquefaction plant operability, low pH agglomeration would have a significant economic impact. The apparently higher Mo addition rate during these periods may have contributed to the benefits of feeding oil-agglomerated coal.

EFFECT OF THE DECREASE IN NOMINAL FRESH CATALYST ADDITION RATE

Decreasing the nominal fresh catalyst addition rate after Condition 2 had no detrimental effect on process sample characteristics. In fact, performance improved (as manifested by increased light distillate yield and decreased resid yield). As noted in a prior section, the concentrations of Mo and Fe in feed slurry and PFC samples suggest that the actual catalyst feed rates differed considerably from the nominal rates in some cases. Those results (Table 11) indicate that the Fe addition rate approximately doubled from Condition 2 to Condition 3, whereas the Mo addition rate decreased by 60% (but remained much higher than the target rate of 70 mg/kg). Perhaps the increased Fe rate contributed to improved performance during Condition 3. No operating condition obviously changed sufficiently to account for the improved performance, although changes in distillation configuration and distillation conditions could have some effect on performance that is difficult to account for.

EFFECT OF RECYCLE SOLVENT DEWAXING AND HYDROGENATION

Vacuum distillates from Run ALC-1 were dewaxed in the laboratory and the dewaxing feeds and fractions were analyzed. Microautoclave solvent quality tests and ^{1}H-NMR spectroscopy were used) to determine the effectiveness of the Run ALC-1 dewaxing and hydrotreating operations. Either vacuum still overhead (VSOH) samples or the laboratory-generated 454 $°C^-$ (850 $°F^-$) distillate of O-6 bottoms samples were tested.

Table 12 shows that the O-6 bottoms distillate contained about 5-6% wax before dewaxing operations were started (Period 20). During dewaxing operations (Period 25), the wax content of the second-stage heavy distillate (VSOH) product was reduced to 1-2%, presumably mostly freshly produced wax. The heavy distillate (VSOH, dewaxed), also taken from period 25 but downstream of dewaxing, was virtually devoid of wax.

The ^{1}H-NMR proton distributions and microautoclave solvent quality test results for heavy distillates relevant to the dewaxing operations are shown in Tables 3 and 6. The data are summarized in Table 13; averages and standard deviations are shown where possible. The data in Table 13 show that the paraffinic nature of the heavy distillate was reduced by dewaxing and there was a minor improvement in solvent quality, as measured by two types of microautoclave liquefaction tests. Hydrotreating was effective in producing hydroaromatics and in improving solvent quality.

For reasons not clear, the VSOH and O-6 bottoms distillates from periods 13, 17, and 20 were more phenolic than the corresponding untreated period 25 samples (Table 13, 0.98 meq/g vs. 0.54 meq/g). Dewaxing had no impact on phenolic -OH concentration, but hydrotreating virtually eliminated the phenols (0.07 meq/g found in hydrotreated samples).

In summary, the results indicate that the bench-unit dewaxing operations were effective. However, the low concentration of wax in the feed limited the impact of the dewaxing on improving solvent quality. The solvent hydrotreating operation increased the hydroaromatic hydrogen content of the distillate and improved its solvent quality.

WAX YIELDS AND CHARACTERISTICS

The wax produced during Run ALC-1 period 25 was characterized by elemental, ^{1}H-NMR, and GC/MS analyses (Table 14). The wax was black and had a strong odor. The elemental analyses indicate that it contained over 13% hydrogen; however, four replicate elemental analyses gave poor repeatability (about 5% relative). It appears that the reported carbon content (74 wt %) is incorrectly low, because the ^{1}H-NMR and GC/MS analyses did not indicate major concentrations of any components except alkanes. The wax was de-oiled via CONSOL's standard dewaxing procedure at -5 °C in acetone to indicate wax purity. This yielded 75% de-oiled wax, but also 2% THF insolubles (Table 14). The de-oiled wax has less than 1% aromatic protons (Table 14).

RECALIBRATION OF FTIR SPECTROSCOPIC METHOD FOR PHENOLIC -OH DETERMINATION

The Fourier-transform infrared spectroscopic (FTIR) method used to determine phenolic -OH concentration in liquefaction samples was recalibrated and implemented on a new FTIR instrument. A calibration was obtained from spectra of six standard phenol compounds. An error estimate for each determination was added to CONSOL's analysis procedure software. The method was validated by comparison of results from samples run on both the old and new systems. A detailed discussion is presented with the results in Appendix 1.

CRUDE OIL ASSAYS OF NET PRODUCTS OF HTI RUN PB-03

CONSOL arranged to have crude oil assays conducted on the net products of HTI Run PB-03, at DOE's request. Crude oil assays were conducted on net products obtained during periods of Run PB-03 with and without use of the on-line hydrotreater.

HTI provided CONSOL with fifteen individual separator overhead (SOH) samples from HTI Run PB-03. The samples were contained in 1 qt (0.95 L) plastic bottles. CONSOL prepared and blended these materials to produce two large samples for crude oil assay testing. The blended samples represent periods with (Periods 6, 7, and 8) and without (Periods 9, 10, and 11) the on-line hydrotreater in operation. Most of the eight individual samples of hydrotreated oil contained a water layer and a sediment (or emulsion) at the oil/water interface. The blends consisted of only the sediment-free oil phase, which was obtained by decanting the easily separated portion of the oil phase, then passing the remainder of the decant through a filter to retain any water or sediment. The water, sediment, and a small remaining oil layer were discarded. Although there was no visual evidence of water or sediment in the seven individual unhydrotreated samples, their dark color made it difficult to ascertain this; therefore, the same procedure was used for blending them. The blended materials weighed 19.96 kg and 17.64 kg, respectively. Each of the two blended materials was placed in two steel 5 gal (3.79 L) cans and shipped to Inchcape Testing Services Caleb Brett Laboratory in Houston, TX. Caleb Brett's reports on the crude oil assays appear as Appendix 3.

Caleb Brett returned all remaining samples and fractions of the two crudes to CONSOL after completion of the crude oil assays. One of the crudes and several of the fractions could not be returned to CONSOL because they were consumed during the crude oil assays. CONSOL determined the proton distributions by ^{1}H-NMR and the phenolic -OH concentrations by FTIR on the returned materials. Those results appear in Table 15.

RESID REACTIVITY STUDIES

Knowledge of the chemistry of resids and understanding of resid reactivity is key to improving direct liquefaction process design and process economics. One approach to acquiring this knowledge is to correlate the chemical characteristics of the resids with the process conditions under which the resids were produced. This was examined in CONSOL's Contract DE-AC22-89PC89883.[12] Another approach is to correlate the relative reactivities of the resids with their chemical characteristics. In order to acquire reactivity data on coal liquefaction resids, a standard empirical test for resid reactivity was developed by the University of Delaware (UOD) under subcontract to CONSOL. Distillation resids chosen by CONSOL for their representation of many different process conditions are being tested (Appendix 4).

To further study the reactivity of resids and their correlation with chemical characteristics, two models are being developed by the University of Delaware: a structural representation model and a reactivity model. Both models can be used for predictive purposes. Analytical data acquired by CONSOL and UOD on the resids being examined by UOD will be incorporated into the structural model. Conversion data and reaction product characteristics will be used to train the reactivity model. However, the University of Delaware resid reactivity tests are being carried out in a short time batch reactor (STBR) with very rapid heating and cooling times. All products of reaction are consumed in the resid conversion determinations. Therefore, there are insufficient reaction products to obtain chemical characterization of them for development of the reactivity model. Additionally, the product gases from the STBR are not analyzed. In order too produce sufficient sample for analysis of products (including gases), a series of resid reactivity tests were made in 45 mL microautoclaves.

These resid reactivity tests were carried out at CONSOL on the set of fifteen resids that currently are under study at the University of Delaware. Temperature and residence time were the same as those used in the STBR: 420 °C for 30 min residence time. The reactor charge in all tests was 4.00 g resid, 8.00 g tetralin, and 10.3 MPa (1500 psi) (cold) H_2. Each resid was reacted with and without molybdenum naphthenate catalyst precursor. In tests with molybdenum naphthenate, a charge of methyldisulfide (also known as dimethyldisulfide, DMDS) was added to sulfide the catalyst precursor. The molybdenum naphthenate charge, when used, was 2.00 g (3 wt % Mo on resid); each catalytic test used 0.5 g of DMDS. A full description of experimental methods is given in the Experimental section (Section 4).

Product analyses included gas chromatography of the collected gases, distillation of the condensed products to an atmospheric equivalent temperature of 454 °C (850 °F), and elemental analysis of the 454 $°C^+$ (850 $°F^+$) product fraction. Product distributions are presented in Table 16. The table gives the feed, product distribution, and resid conversion on an SO_3-free ash-free basis. The analyses of the 454 $°C^+$ (850 $°F^+$) fraction of the products also are provided on that basis. Gas chromatography data were obtained on a volume percent basis. From the vent gas mass and the volume percent of each component, a gram quantity is determined; the gram quantities are provided in Table 16. The nitrogen and oxygen concentrations are presumed to be due to air introduced during charging, sampling collection, and sample analysis; they are normalized out of the analyses. A blank space in Table 16 under Analysis of Product Gases indicates that GC analysis was not performed for that sample.

There was <<1 vol % methane and H_2S detected in the non-catalytic tests. In catalyzed tests, hydrogen reacts with the DMDS to produce methane and H_2S.

Many of the tests were repeated; these replicates are represented on Table 16 by a postscript letter (e.g., 22a means this was the second test at the same conditions of test 22, or 14c means that this is the fourth test at the same conditions as test 14). All analyses were sent to UOD for inclusion in the structural model.

A comparison to the resid conversion values obtained at Delaware (Table 17) shows that the microreactor gave lower conversions than the STBR. The slow heat-up of the microreactor or other scaling factors may be responsible for the difference. Because of the differences in conversion values obtained with the two reaction systems, the microautoclave data were not included in UOD's modeling of resid conversion. The model could conceivably be reworked to account for different reactor systems and accept these data; however, there is no current plan to do this.

Section 4

EXPERIMENTAL

RESID REACTIVITY TESTS

The sequence for resid reactivity tests was: The total charge for all tests was 4.00 g resid, 8.00 g tetralin, and 10.3 MPa (1500 psi) (cold) H_2. In tests with molybdenum naphthenate, a charge of methyldisulfide (also known as dimethyldisulfide, DMDS) was added to sulfide the catalyst precursor. The molybdenum naphthenate charge, when used, was 2.00 g (3 wt % Mo on resid); each catalytic test used 0.5 g of DMDS. After sealing, the vessel was connected to the shaker and shaking was started. While shaking, the microautoclave was lowered into a preheated sand bath. Reaction temperatures were achieved in two to four min. Reaction temperature for all tests was 420 °C. At the end of the 30 min residence time, the microautoclave was removed from the sand bath and immersed in ambient-temperature water while still shaking. The contents were quenched to ca. 100 °C in less than 90 sec. The 30 min reaction time includes the time from immersion of the microautoclave in the sand bath until immersion in the water bath.

A post-run weight was obtained to check for leaks. The microautoclave was attached to a pressure gauge at the fitting above the fill valve. It then was submersed in a dry ice/acetone bath until the internal thermocouple read -70 °C. The valve was opened and a cold temperature gas pressure reading was obtained. The microautoclave was removed from the dry ice/acetone bath and allowed to slowly warm to room temperature; pressure was continually monitored as the vessel warmed. When the internal thermocouple registered room temperature (20-22 °C), a final pressure reading was recorded. The gas was vented into a one-liter evacuated chamber; samples were taken from this chamber for gas chromatography analyses. Gas chromatography was performed using a four-column Carle Model III analytical gas chromatograph. A calibrated standard gas mixture (Table 18) was used to calculate gas composition. The remaining gas was vented. The microautoclave and contents were reweighed. Gas make was determined by the difference in the weight before the tests and after venting.

Prior to distillation of the sample to 454 °C (850 °F), the microautoclave head was removed. A distillation head was attached. The microautoclave body was encased in a custom-designed heating mantle. The microautoclave was heated to a temperature of 265 °C (510 °F) at 5 torr (454 °C (850 °F) atmospheric equivalent temperature). The distillate was retained for further analysis. The 454 °C$^+$ (850 °F$^+$) contents of the microautoclave were submitted for elemental analysis.

Resid conversion was determined on an SO_3-free, ash-free basis as follows:

$$\text{resid conversion} = 100 - \frac{\text{filter cake} - SO_3\text{-free resid ash}}{\text{MAF resid}} \times 100$$

OTHER TESTS

The experimental procedures used to produce results presented in this report have been described previously.[9-11]

Section 5

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TABLE 1

HTI OPERATING CONDITIONS AND PROCESS PERFORMANCE RESULTS FROM RUN ALC-1 (227-94)

Date, 1996	4/24	4/30	5/1	5/5	5/8	5/13
Period	6	12	13	17	20	25
Coal Type	Raw	Agglom	Agglom	Agglom	Agglom	Raw
Condition	1	2	2	3	4	5
Net Normalized Yields, wt % MAF Coal						
C_1 in Gases	3.26	2.98	4.22	3.41	3.32	2.90(a)
C_2 in Gases	2.79	3.28	3.39	3.09	3.49	2.90(a)
C_3 in Gases	3.37	5.01	4.31	4.34	5.59	3.60(a)
C_1-C_3 in Gases	9.42	11.27	11.92	10.84	12.40	9.40(a)
C_4-C_7 in Gases	4.95	5.52	5.19	5.06	6.48	4.17(a)
IBP-177 °C (IBP-350 °F)	13.65	13.40	13.86	14.55	16.53	12.66
177-260 °C (350-500 °F)	11.29	7.71	7.33	7.75	9.70	13.43
260-343 °C (500-650 °F)	23.34	20.13	19.76	23.60	23.88	31.11
343-454 °C (650-850 °F)	14.60	13.77	8.86	12.87	13.24	5.46
454-524 °C (850-975 °F)	1.21	-0.34	1.50	-2.52	-3.75	-1.01
524 °C$^+$ (975 °F$^+$)	4.33	12.34	15.42	10.38	4.56	7.38
Unconverted Coal	5.04	2.16	2.31	2.42	2.51	5.28
Water Yield by Material Balance	13.83	15.23	17.03	16.19	16.20	13.74
COx	5.92	4.28	2.79	4.44	4.27	6.59
NH_3	0.73	0.55	0.53	0.62	0.90	1.04
H_2S	-0.77	-0.25	-0.27	0.03	-0.09	-1.13
Wax Product	-	-	-	-	-	4.79
Process Performance, wt % MAF Coal						
Hydrogen Consumption	7.5	5.8	6.2	6.2	6.8	8.1
Coal Conversion (SO_3 free)	95.0	97.8	97.7	97.6	97.5	94.7
524 °C$^+$ Conversion	90.6	85.5	82.3	87.2	92.9	87.5
C_4-524 °C Distillate	69.0	60.2	56.5	61.3	66.1	70.6(b)
524 °C$^+$ Resid Yield, MAF	4.3	12.3	15.4	10.4	4.6	7.4
C_4-343 °C Distillate	53.2	46.8	46.1	51.0	56.6	66.2(b)
343 °C$^+$ Yield	20.1	25.8	25.8	20.7	14.1	11.8
Process Conditions						
Space Velocity (each reactor) kg MF coal/h/m^3	675	527	558	550	418	466
Reactor Temperatures , °C						
K-1	442	443	442	443	443	443
K-2	453	453	453	452	452	454
Fresh Catalyst Concentration, metal mg/kg MF coal						
Mo in Molyvan A	100	101	100	71	68	70
Fe in Fe-based	10000	10000	9870	7120	6810	6980
Recycle Solvent	PFL/PFC	PFC/VSB/ VSOH	PFC/VSB/ VSOH	PFC/VSB/ VSOH	PFC/VSB/ VSOH	PFC/VSB/ DWHT-VSOH (c)

Other Conditions:

1.60 Recycle to MF coal ratio (lowered slightly by wax yield in Condition 5).
0.20 Recycle solids to MF coal ratio.
15 MPa system pressure

Notes:

(a) Not including small quantities produced in DW-VSOH hydrotreating.
(b) Includes 4.79% wax product in 343-524 °C range.
(c) VSOH dewaxed and then hydrotreated (DWHT) prior to recycle.

TABLE 2

COMPONENT DISTRIBUTION OF WHOLE SAMPLES
HTI Run ALC-1

Sample Type	Period	Component wt % of Sample				
		454 °C⁻ (850 °F⁻) Distillate	THF-Soluble Resid	IOM	Ash	Total
O-6 Bottoms	6B	46.4	36.0	5.0	12.7	100.1
O-6 Bottoms	13B	53.2	32.1	4.9	9.7	99.9
O-6 Bottoms	17B	50.2	34.0	5.6	9.8	99.6
O-6 Bottoms	20B	46.8	34.7	5.3	9.3	96.1
O-6 Bottoms	25B	46.6	33.4	4.6	11.3	95.7
PFC (Toluene Extracted)	6B	-	9.3(a)	23.1	67.6	100.0
PFC (Toluene Extracted)	13B	-	3.0(a)	31.1	65.7	100.0
PFC	13B	-	34.1(a)	20.8	45.1	100.0
PFC	13A	-	39.2(a)	19.2	41.6	100.0
PFC (Toluene Extracted)	17B	-	1.9(a)	20.8	77.3	100.0
PFC	17B	-	35.0(a)	19.6	45.4	100.0
PFC	17A	-	33.6(a)	21.8	44.6	100.0
PFC	20B	-	24.9(a)	32.6	42.5	100.0
PFC (Toluene Extracted)	20B	-	5.0(a)	31.1	63.9	100.0
PFC	25B	-	42.5(a)	16.7	40.8	100.0
PFC (Toluene Extracted)	25B	-	33.7(a)	20.7	45.6	100.0

<u>Note</u>:
Feed slurry component concentrations are provided in Table 8.

(a) Obtained by difference.

TABLE 3

PROTON DISTRIBUTION OF WHOLE SOLIDS-FREE SAMPLES AND DISTILLATE SAMPLES
HTI Run ALC-1

Sample Type	Period	Proton Distribution, %						
		Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
L-814 Start-Up Oil	6A	14.0	6.6	13.3	12.5	13.5	24.0	16.2
SOH First Stage	6	5.2	7.9	9.0	10.4	17.3	25.6	24.7
SOH First Stage	13	4.9	7.2	9.5	10.3	18.6	27.2	22.2
SOH Second Stage	13	1.9	3.1	6.3	5.7	20.6	33.6	28.8
SOH First Stage	17	4.4	7.8	7.8	9.8	17.3	27.4	25.4
SOH Second Stage	17A	2.7	3.7	6.6	5.3	19.3	34.2	28.2
SOH Second Stage	17B	2.6	3.4	6.4	5.6	19.7	33.2	29.2
SOH First Stage	20B	4.5	8.9	8.3	10.3	16.7	28.0	23.4
SOH Second Stage	20B	2.6	3.5	6.6	6.2	20.8	34.7	25.5
SOH First Stage	25B	3.7	7.7	7.7	9.5	19.5	27.0	25.0
SOH Second Stage	25B	3.0	3.7	6.7	6.2	20.1	31.2	29.0
PFL	6	19.5	10.5	14.4	11.4	11.2	20.2	12.9
VSOH	13	12.2	10.5	15.5	12.5	14.1	23.9	11.1
VSOH	17	13.1	10.3	14.9	11.5	13.1	24.9	12.3
VSOH (IBP x 343 °C)	20	9.9	12.4	15.5	13.4	14.3	23.4	11.1
VSOH (343 °C x 399 °C)	20	15.3	8.6	16.3	10.8	12.9	25.9	10.2
VSOH dewaxed, hydrotreated, 343 °C$^+$	21A	9.0	6.6	15.3	10.5	19.8	23.4	15.3
VSOH dewaxed, hydrotreated, 343 °C$^-$	21A	5.0	6.7	12.8	9.6	23.6	25.1	17.1
VSOH (IBP x 524 °C)	25	17.8	6.2	17.8	11.0	15.7	20.8	10.6
VSOH dewaxed, hydrotreated, 343 °C$^+$	25	10.8	6.3	18.4	10.6	19.7	21.3	13.0
VSOH, dewaxed	25	17.8	9.8	17.1	11.3	14.5	18.0	11.5
VSOH, dewaxed, hydrotreated	25	8.2	6.1	15.8	9.9	21.4	23.1	15.5
Wax	25	2.5	1.4	4.6	3.2	8.1	68.7	11.5
VSB from PFL	25	29.9	11.5	18.9	10.1	11.1	10.8	7.9
Feed Slurry (343 °C$^-$)	6	10.4	9.7	15.1	14.0	15.7	23.0	12.0
Feed Slurry (343 x 524°C$^-$)	6	23.1	6.5	17.1	12.1	12.6	20.1	8.5
Feed Slurry (343 x 524°C$^-$)	13	16.3	5.8	16.1	10.0	13.7	28.0	10.2
Feed Slurry (343 x 524°C$^-$)	17	15.8	6.5	16.4	9.1	13.0	28.3	10.9
Feed Slurry (343 °C$^-$)	17	8.9	8.6	14.0	11.3	16.1	27.3	13.7
Feed Slurry (343 x 524°C$^-$)	20	15.5	7.3	15.2	9.4	13.2	27.2	12.1
Feed Slurry (343 x 524°C$^-$)	25	8.4	7.2	15.3	10.2	19.8	23.5	15.6
Feed Slurry (343 °C$^-$)	25	3.7	6.9	11.6	9.9	23.3	26.3	18.4
O-6 Bottoms (454 °C$^-$)	6	20.6	8.5	18.2	12.5	12.4	19.9	7.9
O-6 Bottoms (454 °C$^-$)	13	15.8	9.9	15.8	11.1	12.6	23.8	10.8
O-6 Bottoms (454 °C$^-$)	17	15.4	10.1	15.6	10.7	12.5	24.5	11.2
O-6 Bottoms (454 °C$^-$)	20	16.0	10.0	16.2	11.2	12.8	23.3	10.5
O-6 Bottoms (454 °C$^-$)	25	14.3	9.2	14.8	11.3	15.4	21.2	13.8

TABLE 4

PROTON DISTRIBUTION OF FILTERED WHOLE SOLIDS-CONTAINING SAMPLES AND RESID SAMPLES
HTI Run ALC-1

		Proton Distribution, %						
Sample Type	Period	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
Feed Slurry	5	21.2	11.3	14.3	12.4	11.0	18.1	11.8
Feed Slurry	6	23.5	8.8	17.4	11.5	11.4	17.8	9.7
Feed Slurry	13	19.6	5.9	18.7	9.7	13.0	23.1	10.1
Feed Slurry	17	20.1	6.4	18.2	9.6	13.3	23.4	9.1
Feed Slurry	20	18.5	8.9	14.7	9.2	12.1	23.6	13.0
Feed Slurry	25	11.0	8.5	12.8	9.5	16.9	24.1	17.2
First-Stage Slurry	14	16.8	8.5	16.8	11.4	13.6	22.6	10.4
First-Stage Slurry	17	19.2	7.1	18.2	10.9	13.7	21.6	9.3
First-Stage Slurry	21	21.0	6.7	17.8	10.5	13.3	21.5	9.2
First-Stage Slurry	25	16.4	7.0	18.7	10.8	16.0	20.5	10.8
O-6 Bottoms	6	21.6	12.5	16.3	11.7	11.3	16.6	10.1
O-6 Bottoms	13	18.1	11.9	16.0	11.2	12.5	19.4	10.9
O-6 Bottoms	17	20.7	8.6	18.7	10.4	12.9	19.4	9.2
O-6 Bottoms	20	21.3	8.6	18.8	10.7	12.7	20.3	7.6
O-6 Bottoms	25	20.0	7.9	18.5	11.2	13.9	18.2	10.2
PFC (toluene extracted)	6A	27.2	5.5	15.5	10.0	12.0	16.6	13.2
PFC	13A	25.0	5.7	18.4	10.8	12.8	19.0	8.5
PFC	13B	23.7	5.5	18.9	10.2	13.3	18.8	9.5
PFC	17A	23.3	5.1	20.3	11.6	13.3	17.1	9.2
PFC	17B	25.5	4.5	19.8	10.0	13.4	18.6	8.1
PFC	20B	22.3	7.3	16.6	10.1	12.8	19.9	11.0
PFC	25B	21.6	7.1	15.6	10.4	13.7	18.3	13.2
Feed Slurry (524 °C$^+$)	6	34.1	7.0	15.8	9.6	10.4	14.8	8.2
Feed Slurry (524 °C$^+$)	13	27.6	5.5	17.7	9.2	12.5	17.9	9.7
Feed Slurry (524 °C$^+$)	17	26.6	6.4	16.9	8.7	13.6	16.4	11.8
Feed Slurry (524 °C$^+$)	20	26.5	7.2	14.8	8.2	12.0	18.5	12.7
Feed Slurry (524 °C$^+$)	25	27.8	4.7	16.8	7.4	12.1	18.3	12.8
O-6 Bottoms (454 °C$^+$)	6	40.6	3.7	21.2	9.9	10.5	9.5	4.5
O-6 Bottoms (454 °C$^+$)	13	31.1	8.6	18.7	10.1	11.7	11.8	7.9
O-6 Bottoms (454 °C$^+$)	17	31.1	7.5	18.7	9.9	11.6	12.2	8.9
O-6 Bottoms (454 °C$^+$)	20	33.1	5.7	20.0	9.5	12.0	12.8	6.9
O-6 Bottoms (454 °C$^+$)	25	33.9	6.2	19.9	9.3	11.9	12.2	6.6

TABLE 5

PHENOLIC -OH CONCENTRATION IN SAMPLES FROM HTI RUN ALC-1

		Phenolic -OH	
	Period	Conc., meq/g	Peak Pos., cm-1
Whole Samples			
SOH First Stage Oil	6B	1.48	3304
SOH First Stage Oil	13B	2.12	3307
SOH First Stage Oil	17B	2.04	3307
SOH First Stage Oil	20B	1.79	3307
SOH First Stage Oil	25B	1.33	3308
SOH Second Stage Oil	13B	ND	
SOH Second Stage Oil	17A	ND	
SOH Second Stage Oil	17B	ND	
SOH Second Stage Oil	20A	ND	
SOH Second Stage Oil	20B	ND	
SOH Second Stage Oil	25B	ND	
VSOH	13B	1.17	3311
VSOH	17B	0.91	3308
VSOH (IBPx343 C)	20B	1.48	3311
VSOH (343x399 C)	20B	0.74	3306
VSOH, Dewaxed, Hydrotreated (343 C+)	21A	0.06	3314
VSOH, Dewaxed, Hydrotreated, (343 C-)	21A	ND	
VSOH (IBPx524 C)	25B	0.50	3304
VSOH, Dewaxed, Hydrotreated	25B	0.06	3317
VSOH, Dewaxed, Hydrotreated (343 C+)	25B	0.07	3311
VSOH, Dewaxed	25B	0.54	3303
Wax	25B	0.23	3310
L-814 Start-Up Oil	6A	0.12	3307
VSB	13B	0.87	3297
VSB	17B	0.92	3296
VSB	20B	0.81	3297
Distillates			
O-6 Btms (454 C-)	6B	1.08	3304
O-6 Btms (454 C-)	13B	1.04	3304
O-6 Btms (454 C-)	17B	0.97	3305
O-6 Btms (454 C-)	20B	0.82	3304
O-6 Btms (454 C-)	25B	0.58	3307
Resid THF Sols			
O-6 Btms (454 C+)	6B	0.94	3291
O-6 Btms (454 C+)	13B	1.00	3298
O-6 Btms (454 C+)	17B	0.88	3292
O-6 Btms (454 C+)	20B	0.81	3292
O-6 Btms (454 C+)	25B	0.77	3294
Feed Slurry (524 C+)	5A	0.79	3289
Feed Slurry (524 C+)	6A	0.86	3287
Feed Slurry (524 C+)	5-5-96	0.78	3291
Feed Slurry (524 C+)	13B	0.78	3289
Feed Slurry (524 C+)	17B	0.90	3289
Feed Slurry (524 C+)	20A	0.75	3289
Feed Slurry (524 C+)	25A	0.63	3288
THF-Sols From Whole Samples			
PFC	13A	0.87	3298
PFC	13B	0.86	3299
PFC	17A	0.82	3298
PFC	25B	0.67	3298

ND = Not Detected

TABLE 6

MICROAUTOCLAVE CONVERSIONS
HTI Run ALC-1

Sample Type	Period	Conversion, % (a)	
		Whole Sample	454 °C$^-$ Distillate
O-6 Bottoms	6	71.1	88.7
O-6 Bottoms	13	76.3	88.1
O-6 Bottoms	17	77.1	87.6
O-6 Bottoms	20	74.3	86.7
O-6 Bottoms	25	79.6	88.4
VSOH, Dewaxed Hydrotreated, 343 °C$^+$	21A	92.7	-
VSOH, Dewaxed	25	89.7	-
VSOH, IBP x 524 °C	25	89.5	-
VSOH, Dewaxed Hydrotreated, 343 °C$^+$	25	94.4	-
VSOH, Hydrotreated, Dewaxed	25	92.6	-
VSOH, Dewaxed (acetone free) Can #2	(b)	86.8	-
L-814 Start-Up Oil	6	86.8	-
Feed Slurry	13	-	81.4(c)
Feed Slurry	20	-	84.4(c)

(a) Microautoclave coal conversion, wt % MAF, in modified equilibrium test, Old Ben Mine coal
(b) Pre-run make-up
(c) Distillate was 343 x 524 °C

TABLE 7

COMPOSITION OF SOH WATER PRODUCTS FROM HTI RUN ALC-1

SOH Water Sample Source			Elemental Composition, wt % of Sample		
Period	Stage	pH	S (a)	N (b)	C (b)
6B	First	9.22	0.79	1.52	1.72
13B	First	9.07	1.24	1.77	1.84
13B	Second	9.74	2.69	2.14	0.54
17B	First	9.19	1.07	1.43	1.69
17B	Second	9.21	2.50	2.00	0.27
20B	First	9.02	1.05	1.46	1.70
20B	Second	9.29	2.52	1.85	0.12
25B	First	8.94	0.66	1.25	1.71
25B	Second	9.09	2.22	1.63	0.15

(a) Total S analysis by LECO SC-32 Sulfur Analyzer
(b) C and N analysis by LECO CHN-1000 Analyzer

TABLE 8

CONCENTRATION OF COMPONENTS OF THE FEED SLURRY SAMPLES FROM HTI RUN ALC-1

Date, 1996	4/23	4/24	5/1	5/5	5/8	5/13	5/5(a)
Period	**5A**	**6A**	**13A**	**17A**	**20A**	**25A**	**13A (a)**
Component, wt %							
-343 °C	4.3(b)	5.2(b)	trace	0.9(b)	0.0	4.2(b)	trace
343 °C x 524 °C	6.7	28.7(c)	15.7	20.7	19.1	28.5	16.3
THF-Soluble 524 °C$^+$	52.5	16.4	31.9	24.9	27.8	16.0	39.7
IOM	28.8	39.0	41.1	45.3	43.6	41.6	36.1
Ash	6.8	8.9	6.9	7.8	7.8	8.2	5.0
Total	99.1	98.2	95.6	99.6	98.3	98.5	97.1

(a) Date/period in question
(b) May have contained an unknown amount of water
(c) End point was ca. 529 °C (985 °F), instead of 524 °C (975 °F)

Actual distillation conditions were 5 torr/348 °F pot/378 °F head (176 °C pot/192 °C head) and 5 torr/608 °F pot/638 °F head (320 °C pot/337 °C head) to provide effective cut points of 343 °C (650 °F) and 524 °C (975 °F).

TABLE 9

ASH ELEMENTAL COMPOSITION OF PRESSURE-FILTER CAKES FROM HTI RUN ALC-1

	Period 6 (Toluene-extracted)	Period 13 (Unextracted)	Period 17 (Unextracted)	Period 20 (Unextracted)	Period 25 (Unextracted)
Moisture, wt % As-Determined	0.30	0.23	0.31	0.43	0.41
Ash, wt % MF, Including SO_3	70.45	47.49	45.24	45.61	45.20
Ash, wt % MF, SO_3-Free	51.58	42.60	40.58	41.19	31.64
Major Ash Elements, Oxide wt % of Ash					
MoO_2	0.21	0.31	0.49	0.56	0.21
Na_2O	1.17	0.37	0.37	0.32	1.17
K_2O	0.29	0.33	0.41	0.37	0.27
CaO	14.76	6.47	6.86	6.52	17.45
MgO	3.29	1.14	1.14	1.02	3.58
Fe_2O_3	22.27	36.00	30.45	26.93	17.94
TiO_2	0.77	1.42	1.58	1.61	1.02
P_2O_5	0.81	1.18	1.33	1.33	1.00
SiO_2	19.03	27.76	30.60	32.05	15.24
Al_2O_3	10.34	15.11	16.76	17.97	11.55
SO_3	26.78	10.29	10.31	9.68	29.99
Total	99.72	100.38	100.30	98.36	99.42
Mo wt % of SO3-free ash	0.22	0.26	0.41	0.47	0.23
Fe wt % of SO3-free ash	21.27	28.07	23.75	20.85	17.92
Mo wt % of SO3-containing ash	0.16	0.23	0.37	0.42	0.16
Fe wt % of SO3-containing ash	15.58	25.18	21.30	18.84	12.55
HTI Analyses, wt % of ASTM Ash (a)					
Mo - Second Stage Sample	0.195	0.232	0.400	0.403	0.190
Fe - Second Stage Sample	9.226	21.098	18.766	17.372	13.206
Mo - First Stage Sample	0.198	0.188	0.218	0.232	0.162
Fe - First Stage Sample	13.316	17.468	12.721	12.202	10.708

(a) Source: Table 16 (page 40) of the HTI draft Run ALC-1 report of June 1996 (early draft of Reference 1).

TABLE 10

ASH ELEMENTAL COMPOSITION OF FEED SLURRY 524 °C$^+$ RESIDS FROM HTI RUN ALC-1

	Period 5	Period 6	Period 13	Period 17	Period 20	Period 25 (a)
Moisture, wt % As-Determined	0.26	2.14	1.48	1.25	0.96	2.16
Ash, wt % MF, Including SO_3	8.12	15.26	10.29	10.18	10.02	13.20
Ash, wt % MF, SO_3-Free	6.06	11.42	9.27	9.26	9.16	9.90
Major Ash Elements, Oxide wt % of Ash						
MoO_2	0.20	0.20	0.43	0.48	0.56	0.20
Na_2O	1.15	1.17	0.43	0.38	0.31	1.18
K_2O	0.29	0.29	0.33	0.39	0.36	0.28
CaO	14.53	14.41	6.88	6.57	6.14	16.44
MgO	3.16	3.18	1.25	1.09	0.98	3.43
Fe_2O_3	23.13	23.64	33.63	30.29	29.37	19.17
TiO_2	0.79	0.78	1.41	1.59	1.57	1.02
P_2O_5	0.80	0.82	1.16	1.31	1.27	0.99
SiO_2	19.76	19.08	28.28	30.92	31.64	19.63
Al_2O_3	10.73	11.04	15.94	17.38	17.74	12.37
SO_3	25.41	25.16	9.90	9.03	8.60	25.01
Total	99.95	99.77	99.64	99.43	98.54	99.72
Mo wt % of SO_3-free ash	0.20	0.20	0.36	0.40	0.46	0.20
Fe wt % of SO_3-free ash	21.69	22.09	26.11	23.29	22.47	17.88
Mo wt % of SO_3-containing ash	0.15	0.15	0.32	0.36	0.42	0.14
Fe wt % of SO_3-containing ash	16.18	16.53	23.52	21.19	20.54	13.41

(a) Single determination, others are averages of duplicate determinations.

TABLE 11

Mo AND Fe BALANCE AND APPARENT ADDITION RATES FOR HTI RUN ALC-1

TOTAL Mo & Fe in Feed Slurry, (Based on coal ash and recycle IOM+ash rates):

Period	MF Coal In, g	Fe_2O_3, g	Coal Ash In, g	Coal + Cat Ash In, g	Recycle Ash In, g	Total Ash In, g	Total Mo In, g	Total Fe In, g
6	32,405	454	1,782	2,236	4,439	6,675	13.35	1,475
13	26,786	375	884	1,259	3,536	4,795	17.26	1,252
17	26,365	258	870	1,128	3,691	4,820	19.28	1,122
20	20,078	197	663	859	2,570	3,429	15.77	771
25	22,362	219	1,230	1,449	2,303	3,752	7.50	671

Mo & Fe in TOTAL PFC OUT:

Period	Tot. PFC Out, g	PFC Ash Out, g	PFC Mo Out, g	PFC Fe Out, g	Mo Out/In, Ratio	Fe Out/In, Ratio
6	13,215	6,816	15.00	1,450	1.12	0.98
13	12,537	5,341	13.89	1,499	0.80	1.20
17	11,775	4,778	19.59	1,135	1.02	1.01
20	8,919	3,674	17.27	766	1.09	0.99
25	12,307	3,894	8.96	698	1.19	1.04

Mo & Fe from RECYCLE PFC:

Period	PFC Recycle, g	PFC Ash Recycle, g	PFC Mo Recycle, g	PFC Fe Recycle, g	Mo Rec/In, Ratio	Fe Rec/In, Ratio
6	8,638	4,455	9.80	948	0.73	0.64
13	9,084	3,870	10.06	1,086	0.58	0.87
17	9,030	3,664	15.02	870	0.78	0.78
20	6,618	2,726	12.81	568	0.81	0.74
25	7,420	2,348	5.40	421	0.72	0.63

Mo & Fe NET in Fresh Feed:

Period	Total Mo In, g	Total Fe In, g	PFC Mo Recycle, g	PFC Fe Recycle, g	Calc. Fresh Mo In, g	Calc. Fresh Fe In, g	Calc. Fresh Mo Rate, ppm	Target Mo Rate, ppm
6	13.35	1,475	9.80	948	3.55	527	110	100
13	17.26	1,252	10.06	1,086	7.20	166	269	100
17	19.28	1,122	15.02	870	4.25	252	161	70
20	15.77	771	12.81	568	2.96	202	148	70
25	7.50	671	5.40	421	2.11	250	94	70

Period	Calc Fe Total Rate, ppm	Fe from Coal, ppm	Calc. Fresh Added Fe Rate, ppm	Target Added Fe Rate, ppm
6	16,261	2,618	13,642	10,000
13	6,184	2,256	3,928	10,000
17	9,565	2,256	7,309	7,000
20	10,071	2,256	7,815	7,000
25	11,189	2,618	8,571	7,000

All ppm values are on a ppm (mg/kg) MF coal basis.

TABLE 12

WAX CONTENT IN VSOH
HTI RUN ALC-1

Stream	Period	Wax Yield at Temperature (a), %		
		-5 °C	-20 °C	-35 °C
O-6 Bottoms Distillate	20	5.2	5.9	6.1
VSOH	25	1.2	1.5	2.6
VSOH, dewaxed	25	0.1	0.1	0.2

(a) Yield of wax obtained in laboratory dewaxing test with acetone at the specified temperature.

TABLE 13

SUMMARY OF ^{1}H-NMR ANALYSES AND MICROAUTOCLAVE TESTS RELEVANT TO RUN ALC-1 DEWAXING OPERATIONS

Period	Stream	^{1}H-NMR Proton Types, %			Microautoclave Coal Conversion, % MAF		Phenolic -OH Concentration, meq/g
		Aromatic	Cyclic	Paraffinic	Test A (a)	Test B (b)	
13,17, 20	**VSOH/O-6 Bottoms Distillate**	24.5 ±1.4	28.7 ±0.6	35.4 ±1.2	87.4 ±0.7	78.3 (c)	0.98 ±0.13
25	**VSOH/O-6 Bottoms Distillate**	23.7 ±0.3	31.6 ±1.7	33.4 ±1.4	89.0 ±0.8	80.9/79.4	0.54 ±0.06
25	**VSOH, dewaxed**	27.6	31.6	29.5	89.7	82.3	0.54
25	**VSOH, dewaxed and hydrotreated**	14.3	37.2	38.6	92.6	89.9	0.06
25	**VSOH, dewaxed and hydrotreated, 343 °C$^+$ (650 °F$^+$)**	17.1	38.1	34.3	94.4	90.5	0.07

(a) Modified equilibrium tests with Old Ben Mine coal

(b) Black Thunder Mine coal (4.55 g), solvent (5.45 g), 30 min, 440 °C (824 °F), 10.3 MPa (1500 psig) H_2 (cold)

(c) Data shown from period 20 O-6 bottoms sample

TABLE 14

ANALYSIS AND DE-OILING YIELDS OF WAX FROM RUN ALC-1, PERIOD 25

	wt % (a)
<u>Elemental Analysis of Original Wax</u>	
C	73.82 (b)
H	13.70
N	0.20
S	0.06

(a) Average of four determinations. Repeatability was ca. ±5% relative.

(b) Suspect low, cf. text.

Yields from De-Oiling Original Wax	
Fraction	wt %
De-Oiled Wax	74.7
Oil	19.4
Insolubles	1.7
Losses	4.2

Proton Distributions of De-Oiling Fractions							
	Proton Distribution, %						
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
Original Wax	2.5	1.4	4.6	3.2	8.1	68.7	11.5
De-Oiled Wax	0.5	0.2	0.6	0.7	3.3	80.4	14.4
Oil	8.9	3.3	8.5	5.5	10.6	49.9	13.3

TABLE 15

CONSOL ANALYSES OF PB-03 CRUDE OIL ASSAY FRACTIONS

	Proton Distribution, %							Phenolic -OH	
	Cond Arom	Uncond Aom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma	Conc., meq/g	Peak, cm^{-1}
PB-03-6,7,8									
70 x 180 °F	1.1	0.4	2.8	2.7	30.2	37.6	25.2	BD	-
180 x 350 °F	1.0	2.1	3.7	4.4	27.3	32.0	29.4	BD	-
400 x 550 °F	1.1	5.9	8.6	8.2	22.2	31.1	22.8	BD	-
550 x 650 °F	1.8	4.4	8.9	16.1	20.2	29.6	19.0	BD	-
650 °F$^+$	2.6	2.6	7.3	5.4	16.8	45.5	19.8	BD	-
PB-03-9,10,11									
Crude, as received	5.1	10.4	9.0	11.1	16.9	28.3	19.1	1.69	3309
70 x 180 °F	1.0	0.3	1.5	3.5	28.1	37.8	27.8	BD	-
180 x 350 °F	1.9	6.7	4.0	5.9	22.0	31.2	28.2	1.15	3300
400 x 550 °F	4.7	14.0	12.2	16.0	13.9	23.9	15.2	2.44	3314
550 x 650 °F	9.4	8.5	14.0	11.6	15.7	26.3	14.6	0.92	3314
650 °F$^+$	14.3	8.3	13.8	10.9	12.7	26.9	13.2	0.75	3304

BD = Below Detection Limit

Temperature Equivalents:
70 °F = 21 °C
180 °F = 82 °C
350 °F = 177 °C
400 °F = 204 °C
550 °F = 288 °C
650 °F = 343 °C

TABLE 16

RESID REACTIVITY TESTS

Run No.	RR-8 8/19/96	RR-9 8/21/96	RR-10 8/22/96	RR-11 8/23/96	RR-12 8/26/96	RR-13 8/27/96
Feed						
Resid Source	W259 V1067	W259 V1067	W261 R1235	W261 R1235	W259 V131B	W260 V131B
Resid, g	4.00	4.00	4.00	4.00	4.00	4.00
Mo Naphthenate, g	-	2.00	2.00	-	2.00	2.00
DMDS, g	-	0.50	0.50	-	0.50	0.50
Tetralin, g	8.00	8.00	8.00	8.00	8.00	8.00
H_2, g	0.33	0.28	0.37	0.24	0.32	0.28
Product						
Gas, g	0.06	0.39	0.43	0.13	0.51	0.39
Distillate						
Cold Trap 1, g	1.22	0.33	0.95	0.87	0.52	0.15
Cold Trap 2, g	-	1.36	1.43	0.30	0.20	0.66
Receiver 1, g	6.28	0.14	7.42	6.71	7.94	7.03
Receiver 2, g	-	5.65	-	-	0.55	0.55
Dist. Overhead, g	0.01	0.10	0.05	0.01	0.01	0.04
Dist. Column, g	0.06	0.61	0.45	0.05	0.30	0.29
Reactor Head, g	0.29	0.87	0.30	0.15	1.45	0.32
Reactor Bottom, g	3.78	3.71	3.32	3.66	3.25	3.54
Total						
Recovery, g	11.70	13.16	14.35	11.88	14.73	12.97
Recovery (% of feed)	94.89	89.04	96.50	97.06	99.39	87.75
Resid Ash Content	10.07	10.07	12.76	12.76	8.42	11.69
Conversion on ash-free basis	5.83	10.71	22.60	9.61	23.23	16.92
Analysis of 454 °C$^+$ (850 °F$^+$) Product, wt % dry						
C	80.52	77.71	72.61	77.18	78.29	73.08
H	5.89	6.32	6.05	6.09	5.86	5.60
N	0.95	0.65	0.64	1.03	0.68	0.67
S	1.02	3.11	3.43	1.23	2.99	3.61
O (by diff)	1.24	-1.22	-1.37	0.65	-1.03	-0.06
Ash (SO_3-free)	10.38	13.42	18.65	13.82	13.47	17.10
Comments:						ice used for trap - not CO_2
Analysis of Product Gases, g						
methane		0.1502	0.1656	0.0006	0.2127	0.0515
ethane		0.0090	0.0116	0.0016	0.0108	0.0035
propane		0.0044	0.0065	0.0009	0.0057	0.0015
propylene		0.0005	-	-	-	-
n-butane		0.0007	0.0015	0.0004	-	-
CO		0.0056	0.0060	-	-	-
CO_2		0.0165	0.0152	0.0073	0.0209	0.2568
H_2S		0.0166	0.0486	0.0007	0.0403	0.0052
H_2		0.1864	0.1750	0.1185	0.2195	0.0714
Total		0.39	0.43	0.13	0.51	0.39

TABLE 16 (Continued)

RESID REACTIVITY TESTS

Run No.	RR-14 8/29/97	RR-15 9/3/96	RR-16 9/4/96	RR-17 9/5/96	RR-18 9/6/96	RR-19 9/9/96
Feed						
Resid Source	W260 R1235	W262 V1067	W261 V1067	W260 V1067	W261 V131B	W259 R1235
Resid, g	4.00	4.00	4.00	4.00	4.00	4.00
Mo Naphthenate, g	2.00	2.00	2.00	2.00	2.00	2.00
DMDS, g	0.50	0.50	0.50	0.50	0.50	0.50
Tetralin, g	8.00	8.00	8.00	8.00	8.00	8.00
H_2, g	0.26	0.28	0.34	0.30	0.27	0.28
Product						
Gas, g	0.43	0.76	0.32	0.36	0.45	0.46
Distillate						
Cold Trap 1, g	3.19	2.07	0.95	0.90	0.56	1.80
Cold Trap 2, g	3.03	2.01	0.26	0.06	1.12	0.04
Receiver 1, g	7.67	5.87	7.84	8.12	8.01	7.35
Receiver 2, g	1.02	0.45	0.64	0.60	0.39	0.82
Dist. Overhead, g	0.07	0.02	0.03	0.03	0.03	0.03
Dist. Column, g	0.28	0.20	0.57	0.27	0.26	0.22
Reactor Head, g	0.30	0.78	0.77	0.34	0.61	0.75
Reactor Bottom, g	3.71	3.21	3.34	3.62	3.45	3.18
Total						
Recovery, g	19.70	15.37	14.72	14.30	14.88	14.65
Recovery (% of feed)	133.47	103.99	99.19	96.62	100.74	99.12
Resid Ash Content	13.13	13.29	14.74	14.25	9.32	8.61
Conversion on ash-free basis	15.04	25.65	21.55	15.19	18.42	24.61
Analysis of 454 °C$^+$ (850 °F$^+$) Product, wt % dry						
C	71.94	70.5	71.88	70.99	77.28	78.83
H	5.48	6.16	6.06	5.50	6.94	5.70
N	0.70	0.54	0.57	0.64	0.71	1.00
S	3.81	4.08	3.45	3.89	2.76	3.01
O (by diff)	-2.36	-0.94	-1.86	-0.66	-1.92	-1.27
Ash (SO_3-free)	20.43	19.66	19.90	19.64	14.23	13.33
Comments:		material lost during venting				
Analysis of Product Gases, g						
methane	0.1147		0.0019	0.1501	0.1870	0.1789
ethane	0.0087		0.0090	0.0077	0.0109	0.0102
propane	0.0037		0.0053	0.0037	0.006	0.0044
propylene	-		-	-	-	-
n-butane	-		-	-	0.0018	-
CO	-		-	-	-	0.0123
CO_2	0.1094		0.0236	0.0107	0.0121	0.0387
H_2S	0.0172		0.0193	0.0190	0.0227	0.0155
H_2	0.1769		0.2575	0.1688	0.2095	0.1999
Total	0.43		0.32	0.36	0.45	0.46

TABLE 16 (Continued)

RESID REACTIVITY TESTS

Run No.	**RR-20** **9/10/96**	**RR-21** **9/11/96**	**RR-22** **9/12/96**	**RR-23** **9/13/96**	**RR-24** **9/16/96**	**RR-9a** **9/17/96**
Feed						
Resid Source	W262 R1235	W262 V131B	W258 V131B	HRI O-43 POC-1 57B	HRI O-43 POC-02	W259 V1067
Resid, g	4.00	4.00	4.00	4.00	4.00	4.00
Mo Naphthenate, g	2.00	2.00	2.00	2.00	2.00	2.00
DMDS, g	0.50	0.50	0.50	0.50	0.50	0.50
Tetralin, g	8.00	8.00	8.00	8.00	8.00	8.00
H_2, g	0.26	0.26	0.25	0.37	0.33	0.26
Product						
Gas, g	1.28	0.36	0.57	0.43	0.48	0.43
Distillate						
Cold Trap 1, g	0.32	0.91	0.58	0.52	0.71	0.49
Cold Trap 2, g	0.06	0.10	0.13	0.37	0.36	0.13
Receiver 1, g	8.05	8.22	7.88	8.02	8.17	8.38
Receiver 2, g	0.62	0.68	0.67	0.96	0.69	0.72
Dist. Overhead, g	0.02	0.03	0.02	0.09	0.04	0.03
Dist. Column, g	0.21	0.23	0.25	0.22	0.22	0.25
Reactor Head, g	0.90	0.40	0.37	0.18	0.38	0.31
Reactor Bottom, g	3.17	3.63	3.74	3.46	3.59	3.72
Total						
Recovery, g	14.63	14.56	14.21	14.25	14.64	14.46
Recovery (% of feed)	99.12	98.64	96.34	95.83	98.72	97.97
Resid Ash Content	11.94	12.23	13.71	0.4	4.09	10.07
Conversion on ash-free basis	26.97	15.04	11.92	17.49	14.82	11.63
Analysis of 454 °C$^+$ (850 °F$^+$) Product, wt % dry						
C	70.4	72.13	72.01	85.3	82.45	76.92
H	6.49	6.45	4.88	9.34	8.32	5.78
N	0.57	0.57	0.82	0.42	0.38	0.07
S	4.20	3.87	3.59	2.10	2.19	3.07
O (by diff)	-0.51	-0.85	0.00	-2.16	-2.31	-1.02
Ash (SO_3-free)	18.85	17.83	18.71	5	8.97	14.55
Comments:	material lost during venting					
Analysis of Product Gases, g						
methane		0.0275	0.1491	0.0511	0.0739	
ethane		0.0008	0.0093	0.0030	0.0045	
propane		0.0012	0.0037	0.0014	0.0221	
propylene		-	-	-	-	
n-butane		0.0006	0.0246	0.0068	0.0146	
CO		0.0817	0.0317	0.101	0.0732	
CO_2		0.2025	0.0758	0.1851	0.1267	
H_2S		0.0039	0.0241	0.009	0.0160	
H_2		0.0418	0.2516	0.0726	0.1490	
Total		0.36	0.57	0.43	0.48	

TABLE 16 (Continued)

RESID REACTIVITY TESTS

Run No.	RR-13a 9/18/96	RR-14a 9/19/96	RR-17a 9/20/96	RR-22a 9/23/96	RR-25 9/24/96	RR-26 9/25/96
Feed						
Resid Source	W260 V131B	W260 R1235	W260 V1067	W258 V131B	W259 R1235	W260 R1235
Resid, g	4.00	4.00	4.00	4.00	4.00	4.00
Mo Naphthenate, g	2.00	2.00	2.00	2.00	-	-
DMDS, g	0.50	0.50	0.50	0.50	-	-
Tetralin, g	8.00	8.00	8.00	8.00	8.00	8.00
H_2, g	0.23	0.31	0.26	0.26	0.27	0.27
Product						
Gas, g	0.28	0.39	0.33	0.35	0.23	0.29
Distillate						
Cold Trap 1, g	0.45	1.14	0.69	0.95	0.58	0.9
Cold Trap 2, g	0.31	0.29	0.34	0.35	0.25	0.14
Receiver 1, g	7.99	7.62	8.06	7.74	7.2	7.07
Receiver 2, g	0.79	0.85	0.76	0.8	0.04	0.01
Dist. Overhead, g	0.04	0.04	0.04	0.04	0.03	0.03
Dist. Column, g	0.2	0.28	0.2	0.18	0.09	0.08
Reactor Head, g	0.3	0.58	0.34	0.39	0.24	0.16
Reactor Bottom, g	3.65	3.3	3.66	3.76	3.73	3.67
Total						
Recovery, g	14.01	14.49	14.42	14.56	12.39	12.35
Recovery (% of feed)	95.11	97.84	97.70	98.64	100.98	100.65
Resid Ash Content	11.69	13.13	14.25	13.71	8.61	13.13
Conversion on ash-free basis	14.57	22.21	14.42	11.65	7.78	12.26
Analysis of 454 °C$^+$(850 °F$^+$) Product, wt % dry						
C	73.16	70.13	70.92	72.46	81.63	75.68
H	5.92	5.38	5.46	5.15	5.90	5.78
N	0.67	0.65	0.68	0.77	1.02	0.99
S	3.66	4.53	4.11	3.72	1.42	1.73
O (by diff)	-0.73	1.22	-0.97	-0.99	0.41	-1.11
Ash (SO_3-free)	17.32	18.09	19.8	18.9	9.62	16.93
Comments:						
Analysis of Product Gases, g						
methane					0	0.0054
ethane					0.0078	0.0038
propane					0.0063	0.0018
propylene					-	-
n-butane					0.0041	-
CO					-	0.0062
CO_2					0.0084	0.0184
H_2S					0	0
H_2					0.2034	0.2545
Total					0.23	0.29

TABLE 16 (Continued)

RESID REACTIVITY TESTS

Run No.	RR-27 9/26/96	RR-28 9/27/96	RR-29 9/28/96	RR-30 10/1/96	RR-31 10/2/96	RR-32 10/3/96
Feed						
Resid Source	W261 R1235	W262 R1235	HRI O-43 POC-1 57B	W59 V131B	W260 V131B	W260 V131B
Resid, g	4.00	4.00	4.00	4.00	4.00	4.00
Mo Naphthenate, g	-	-	-	-	-	-
DMDS, g	-	-	-	-	-	-
Tetralin, g	8.00	8.00	8.00	8.00	8.00	8.00
H_2, g	0.29	0.27	0.25	0.26	0.25	0.27
Product						
Gas, g	0.16	0.18	0.21	0.18	0.21	0.29
Distillate						
Cold Trap 1, g	0.28	0.33	0.77	0.03	0.02	0.46
Cold Trap 2, g	0.3	0.23	0.19	0.03	0.06	0.08
Receiver 1, g	7.05	7.38	7.25	7.93	7.85	7.3
Receiver 2, g	0.02	0.02	0.01	0.03	0.04	0.01
Dist. Overhead, g	0.01	0.01	0.01	0	0	0
Dist. Column, g	0.03	0.1	0.06	0	0.1	0.06
Reactor Head, g	0.38	0.23	0.12	0.2	0.1	0.09
Reactor Bottom, g	3.66	3.64	3.72	3.78	3.66	3.79
Total						
Recovery, g	11.89	12.12	12.34	12.18	12.04	12.08
Recovery (% of feed)	96.75	98.78	100.73	99.35	98.29	98.45
Resid Ash Content	12.76	11.94	0.4	8.42	11.69	9.32
Conversion on ash-free basis	9.07	10.10	7.00	5.71	9.02	5.64
Analysis of 454 °C$^+$ (850 °F$^+$) Product, wt % dry						
C	77.76	76.28	86.73	83.47	77.39	80.69
H	6.36	6.60	9.79	5.84	5.58	6.44
N	1.02	0.90	0.80	1.17	0.90	0.81
S	1.28	1.72	0.29	1.15	1.53	0.92
O (by diff)	0.28	14.5	1.99	-0.25	2.41	1.45
Ash (SO_3-free)	13.30	13.00	0.40	8.62	12.19	9.69
Comments:						
Analysis of Product Gases, g						
methane	0.0014	0.0011	0.0004	0.0008	0.0020	0.0035
ethane	0.0023	0.0027	0.0018	0.0018	0.0031	0.0035
propane	0.0010	0.0010	0.0011	0.0010	0.0015	0.0022
propylene	-	-	-	-	-	-
n-butane	-	-	-	-	-	-
CO	0.0015	-	-	-	0.0025	0.0028
CO_2	0.0077	0.0055	0.0011	0.0021	0.0055	0.0074
H_2S	0.0004	0	0	0	0	0
H_2	0.1458	0.1697	0.2056	0.1744	0.1954	0.2705
Total	0.16	0.18	0.21	0.18	0.21	0.29

TABLE 16 (Continued)

RESID REACTIVITY TESTS

Run No.	RR-33 10/4/96	RR-34 10/7/96	RR-35 10/8/96	RR-36	RR-37 10/22/96	RR-38 10/23/96
Feed						
Resid Source	W262 V131B	HRI O-43 POC-2	W259 V1067	W260 V1067	W261 V1067	W262 V1067
Resid, g	4.00	4.00	4.00	4.00	4.00	4.00
Mo Naphthenate, g	-	-	-	-	-	-
DMDS, g	-	-	-	-	-	-
Tetralin, g	8.00	8.00	8.00	8.00	8.00	8.00
H_2, g	0.27	0.28	0.28	0.26	0.25	0.27
Product						
Gas, g	0.17	0.25	0.27	0.18	0.25	0.21
Distillate						
Cold Trap 1, g	0.5	0.21	0.46	0.62	0.64	0.65
Cold Trap 2, g	0.05	0.1	0.08	0.12	0.15	0.12
Receiver 1, g	7.53	7.69	7.53	7.44	7.27	7.43
Receiver 2, g	0.01	0.14	0.02	0.02	0.01	0.03
Dist. Overhead, g	0.01	0	0.01	0	0.01	0
Dist. Column, g	0.01	0.07	0.05	0.15	0.02	0.03
Reactor Head, g	0.16	0.11	0.11	0.05	0.07	0.1
Reactor Bottom, g	3.71	3.8	3.76	3.64	3.75	3.71
Total						
Recovery, g	12.15	12.37	12.29	12.22	12.17	12.28
Recovery (% of feed)	99.02	100.73	100.08	99.67	99.35	100.08
Resid Ash Content	12.23	4.09	10.07	14.25	14.74	13.29
Conversion on ash-free basis	7.86	5.16	6.27	9.46	6.78	8.81
Analysis of 454 °C$^+$ (850 °F$^+$) Product, wt % dry						
C	76.39	81.73	81.18	74.98	75.63	74.57
H	5.95	11.65	5.40	5.15	6.05	6.04
N	0.91	0.43	0.91	0.93	0.94	0.85
S	1.59	0.20	1.32	1.74	1.25	1.72
O (by diff)	2.34	1.74	0.86	2.52	0.91	2.07
Ash (SO_3-free)	12.81	4.25	10.33	14.68	15.22	14.75
Comments:						
Analysis of Product Gases, g						
methane	0.0026	0		0.0019		
ethane	0.0029	0.0024		0.0028		
propane	0.0014	-		0.0016		
propylene	-	-		-		
n-butane	-	-		-		
CO	0.0045	-		0.0023		
CO_2	0.0078	0.0036		0.0049		
H_2S	0	0		0		
H_2	0.2508	0.2440		0.1663		
Total	0.27	0.25		0.18		

TABLE 16 (Continued)

RESID REACTIVITY TESTS

Run No.	RR-39 10/10/96	RR-13B 10/24/96	RR-14B 10/25/96	RR-22B 10/30/96	RR-14C 10/29/96	RR-26A 10/31/96
Feed						
Resid Source	W258 V131B	W260 V131B	W260 V1235	W258 V131B	W260 R1235	W260 R1235
Resid, g	4.00	4.00	4.00	4.00	4.00	4.00
Mo Naphthenate, g	-	2.00	2.00	2.00	2.00	-
DMDS, g	-	0.50	0.50	0.50	0.50	-
Tetralin, g	8.00	8.00	8.00	8.00	8.00	8.00
H_2, g	0.27	0.27	0.26	0.26	0.28	0.27
Product						
Gas, g	0.25	0.38	0.35	0.49	0.37	0.31
Distillate						
Cold Trap 1, g	0.65	0.69	0.87	0.88	0.47	0.17
Cold Trap 2, g	0.15	0.14	0.25	0.26	0.16	0.15
Receiver 1, g	7.19	8.27	7.83	7.69	8.67	7.86
Receiver 2, g	0.01	0.75	0.88	0.8	0.86	0.02
Dist. Overhead, g	0.01	0.01	0	0.01	0.01	0
Dist. Column, g	0.04	0.31	0.26	0.26	0.23	0.02
Reactor Head, g	0.11	0.1	0.12	0.12	0.13	0.06
Reactor Bottom, g	3.8	3.67	3.67	3.76	3.68	3.71
Total						
Recovery, g	12.21	14.32	14.23	14.27	14.58	12.30
Recovery (% of feed)	99.51	96.95	96.41	96.68	98.65	100.24
Resid Ash Content	13.71	11.69	13.13	13.71	13.13	13.13
Conversion on ash-free basis	5.58	13.86	15.17	11.19	14.04	7.74
Analysis of 454 °C$^+$ (850 °F$^+$) Product, wt % dry						
C	76.49	73.43	71.32	72.51	71.91	75.35
H	4.91	5.7	5.34	5.15	5.45	5.42
N	0.98	0.65	0.66	0.74	0.73	0.95
S	1.57	3.67	3.97	3.73	3.83	1.45
O (by diff)	1.81	-0.54	-0.96	-0.59	-0.75	3.24
Ash (SO_3-free)	14.24	17.09	19.68	18.47	18.83	13.59
Comments:						
Analysis of Product Gases, g						
methane	0.0002	0.1424	0.1092	0.1571	0.1270	-
ethane	0.0024	0.0086	0.0083	0.0171	0.0081	-
propane	0.0014	0.0036	0.0038	0.0053	0.0040	-
propylene	-	-	-	-	-	-
n-butane	-	-	-	-	-	-
CO	0.0013	-	0.0012	-	0.0050	-
CO_2	0.0042	0.0108	0.0103	0.0105	0.0119	0.0111
H_2S	0	0.0196	0.0145	0.0204	0.0123	-
H_2	0.2404	0.1950	0.2026	0.2795	0.2017	0.2989
Total	0.25	0.38	0.35	0.49	0.37	0.31

TABLE 17

COMPARISON OF RESID CONVERSION DATA OBTAINED IN 45 mL MICROAUTOCLAVES (CONSOL R&D) AND SHORT TIME BATCH REACTOR (UNIVERSITY OF DELAWARE)

Resid	UOD Conversion		CONSOL Conversion	
	Thermal	Catalytic	Thermal	Catalytic
W258, V131B	16.1	35.3	5.6	11.9
W259, V1067	14.4	40.8	5.8	10.7
W259, R1235	15.3	36.9	7.8	24.6
W259, V131B	15.8	31.7	5.7	23.2
W260, V1067	18.1		9.5	15.2
W260, R1235	18.4	33.5	12.3	15.0
W260, V131B	21.8	44.9	9.0	16.9
W261, V1067	21.1	43.0	6.8	21.6
W261, R1235	15.4		9.6	22.6
W261, V131B	16.3	34.4	5.6	18.4
W262, V1067	15.8	36.2	8.8	25.7
W262, R1235	17.8	30.2	10.1	27.0
W262, V131B	18.7	34.1	7.9	15.0
HRI POC1 O-43			7.0	17.5
HRI POC2 O-43			5.2	14.8

TABLE 18

GAS CHROMATOGRAPHY CALIBRATION GASES

Component	Volume, %
methane	8.0
ethane	3.0
ethylene	0.5
propane	2.0
propylene	0.5
n-butane	1.0
I-butane	0.5
1-butene	0.5
trans-2-butene	0.5
cis-2-butene	0.5
n-pentane	0.5
I-pentane	0.5
carbon monoxide	1.0
carbon dioxide	1.0
nitrogen	0.5
argon	1.0
hydrogen	78.5

HTI Run ALC-1

Component Distribution Of Feed Slurry Samples

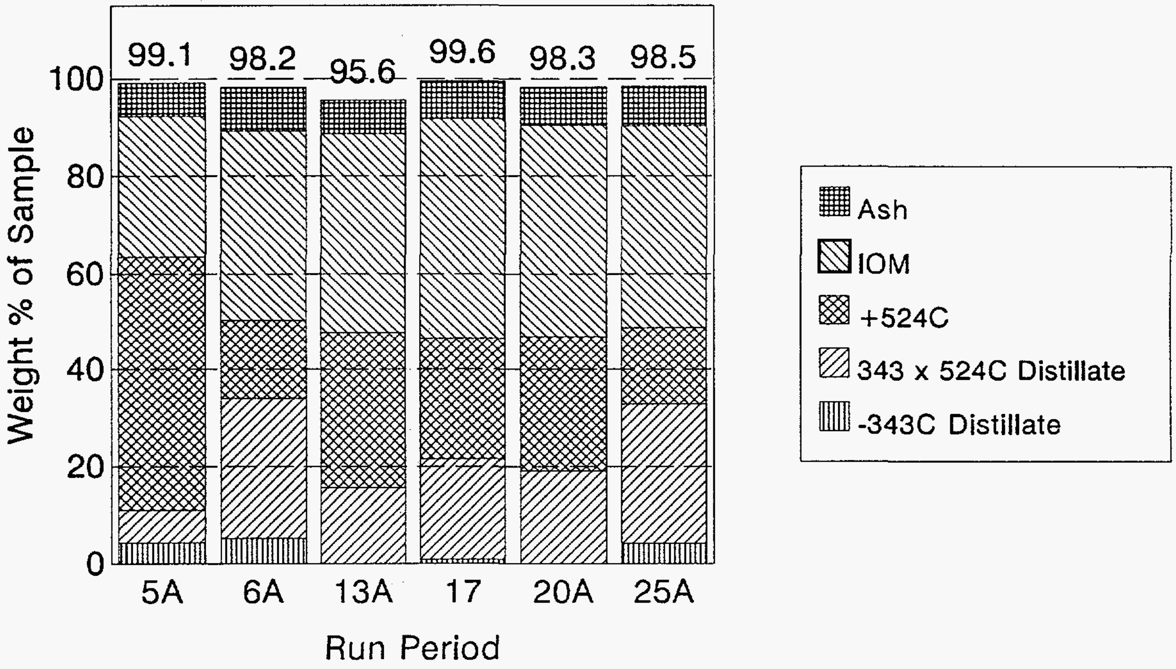


Figure 1. Component Distribution of Feed Slurry Samples from HTI Run ALC-1.

HTI Run ALC-1

Component Distribution Of O-6 Bottom Samples

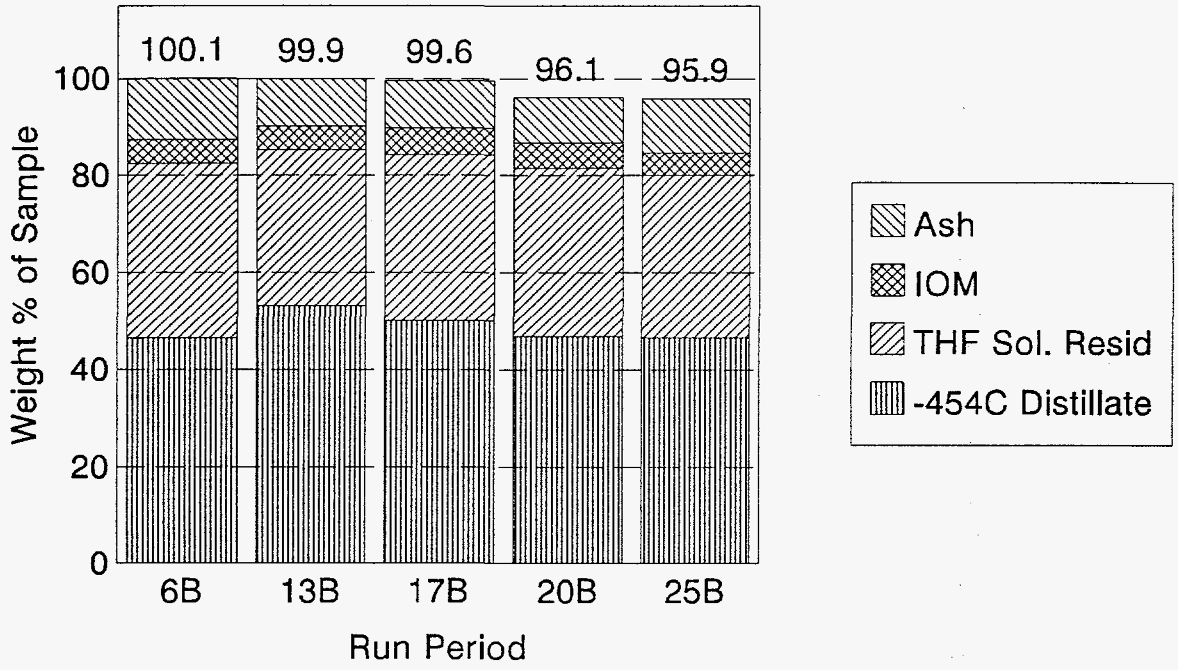
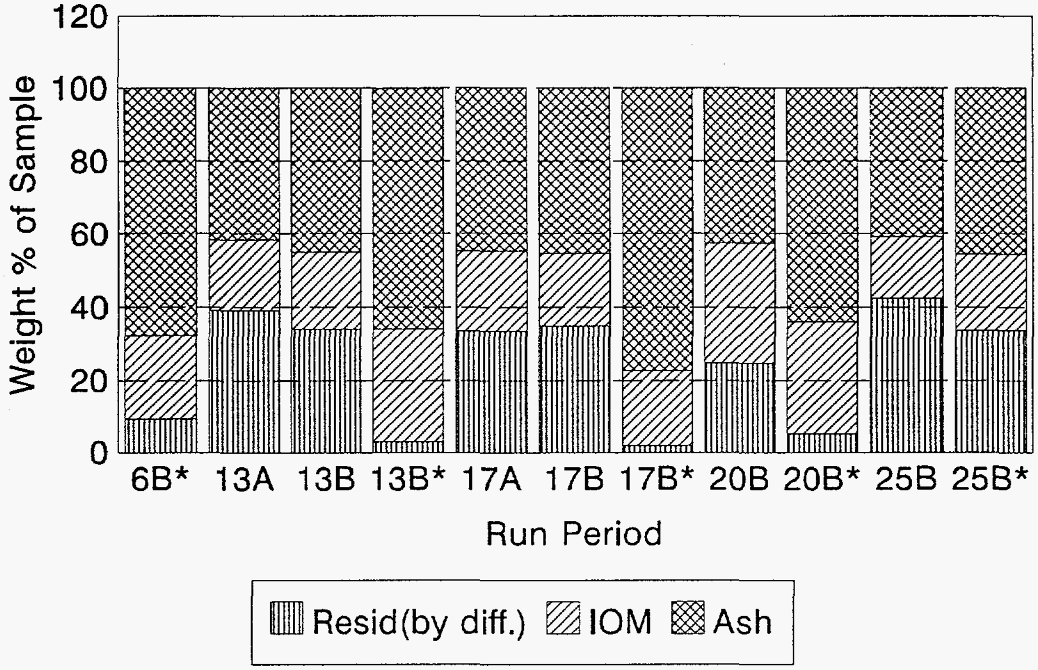


Figure 2. Component Distribution of O-6 Bottoms Samples from HTI Run ALC-1.

HTI Run ALC-1

Component Distribution Of PFC Samples



* Toluene Extracted

Figure 3. Component Distribution of Pressure-Filter Cake (PFC) Samples from HTI Run ALC-1.

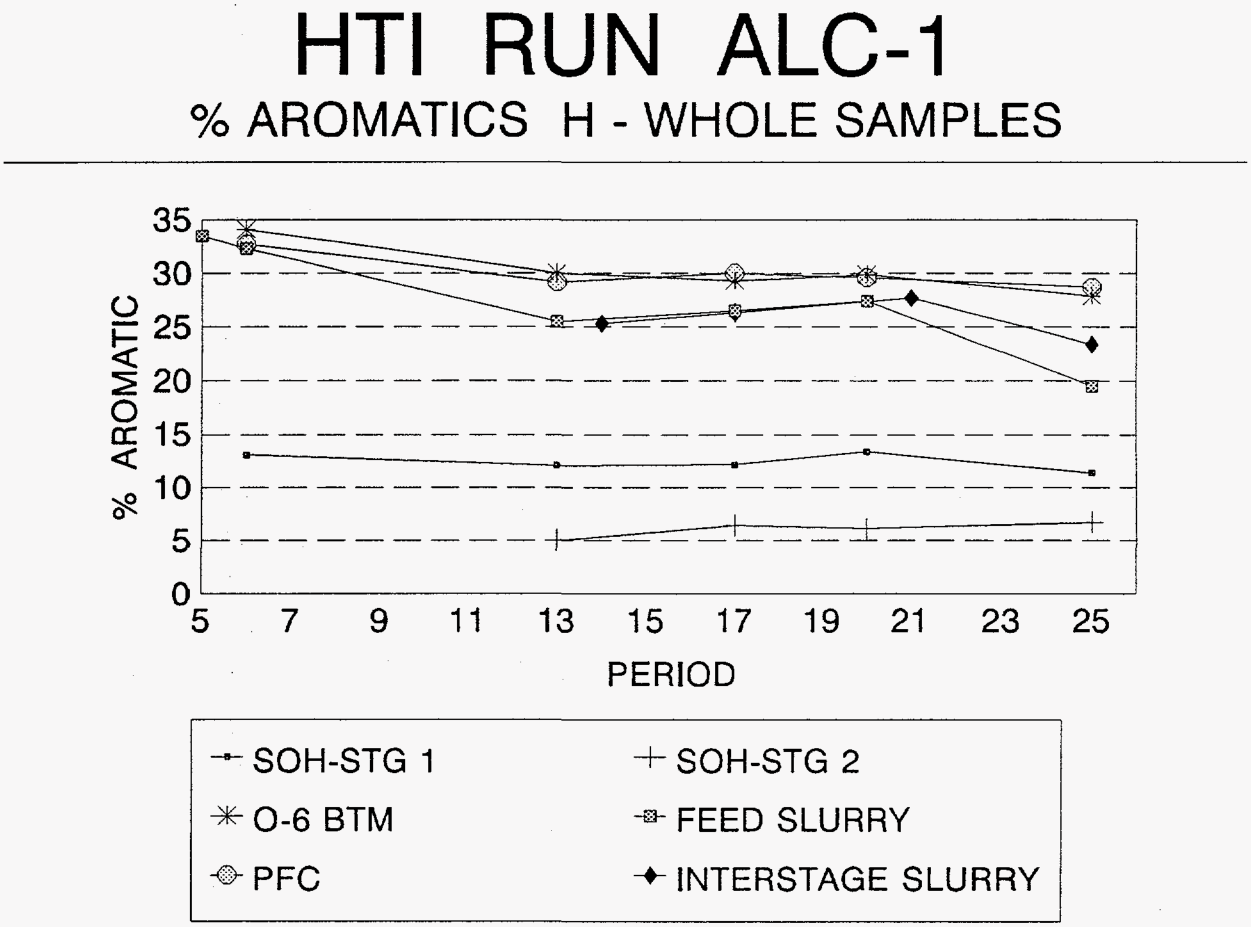


Figure 4. Proton Aromaticity of Whole Process Stream Samples from HTI Run ALC-1.

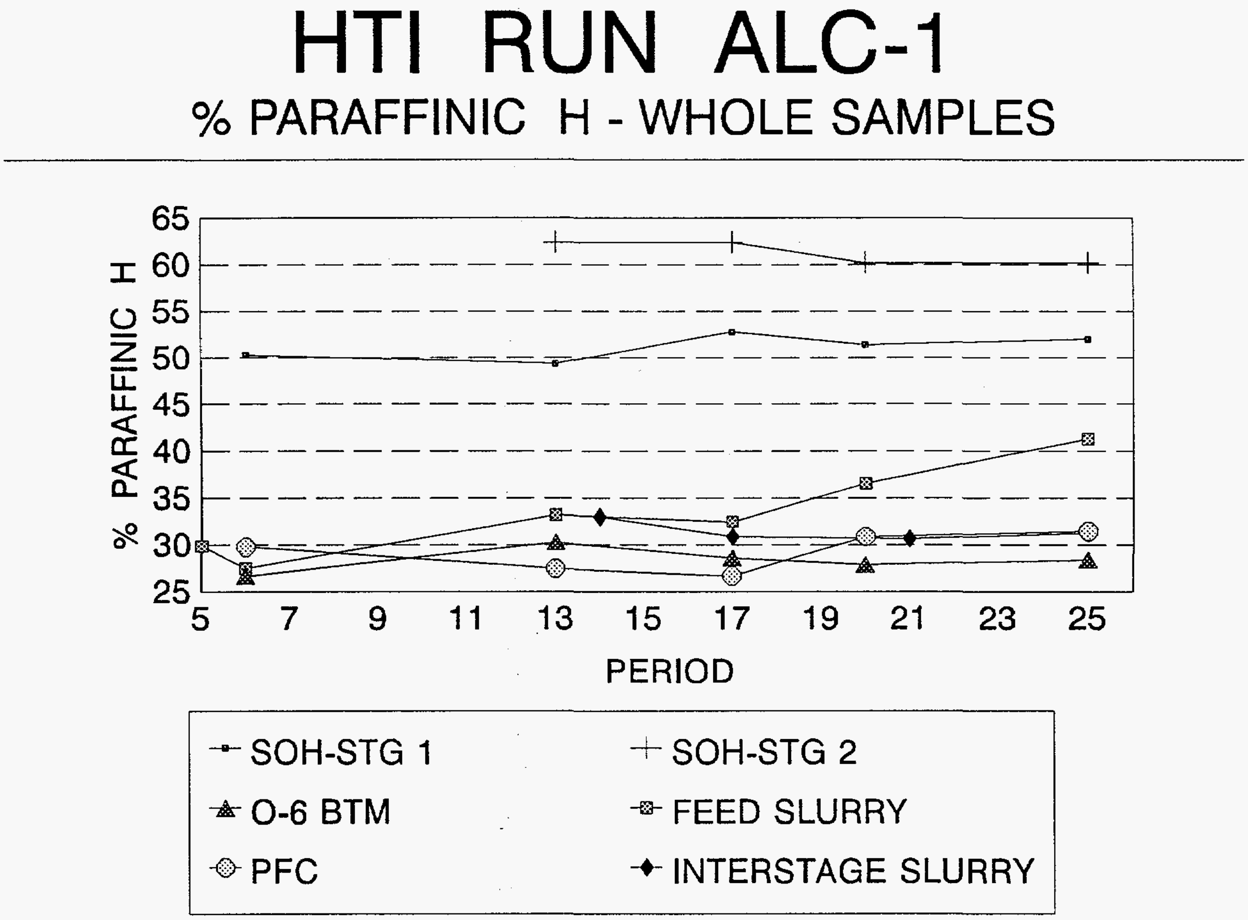


Figure 5. Paraffinic Proton Content of Whole Process Stream Samples from HTI Run ALC-1.

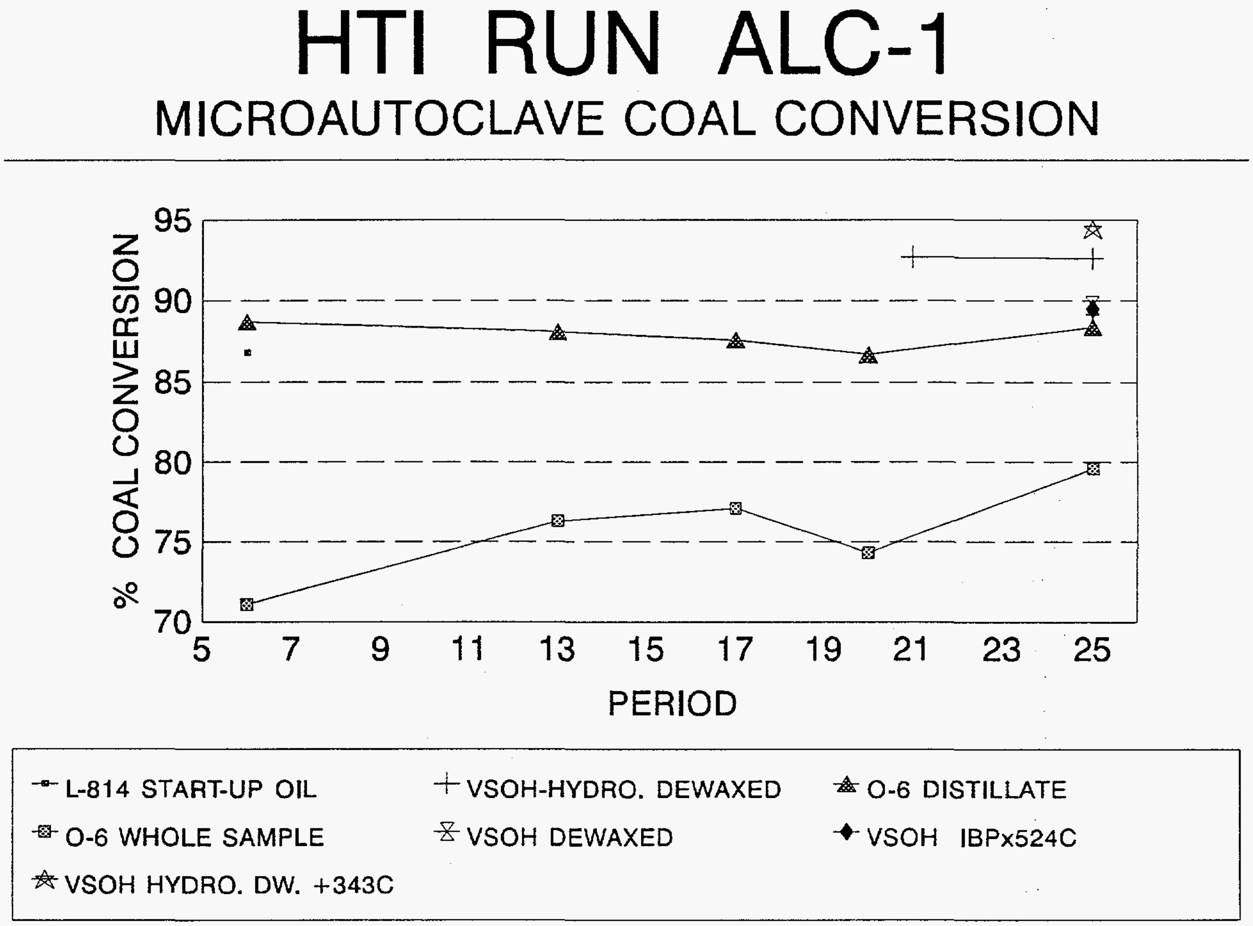
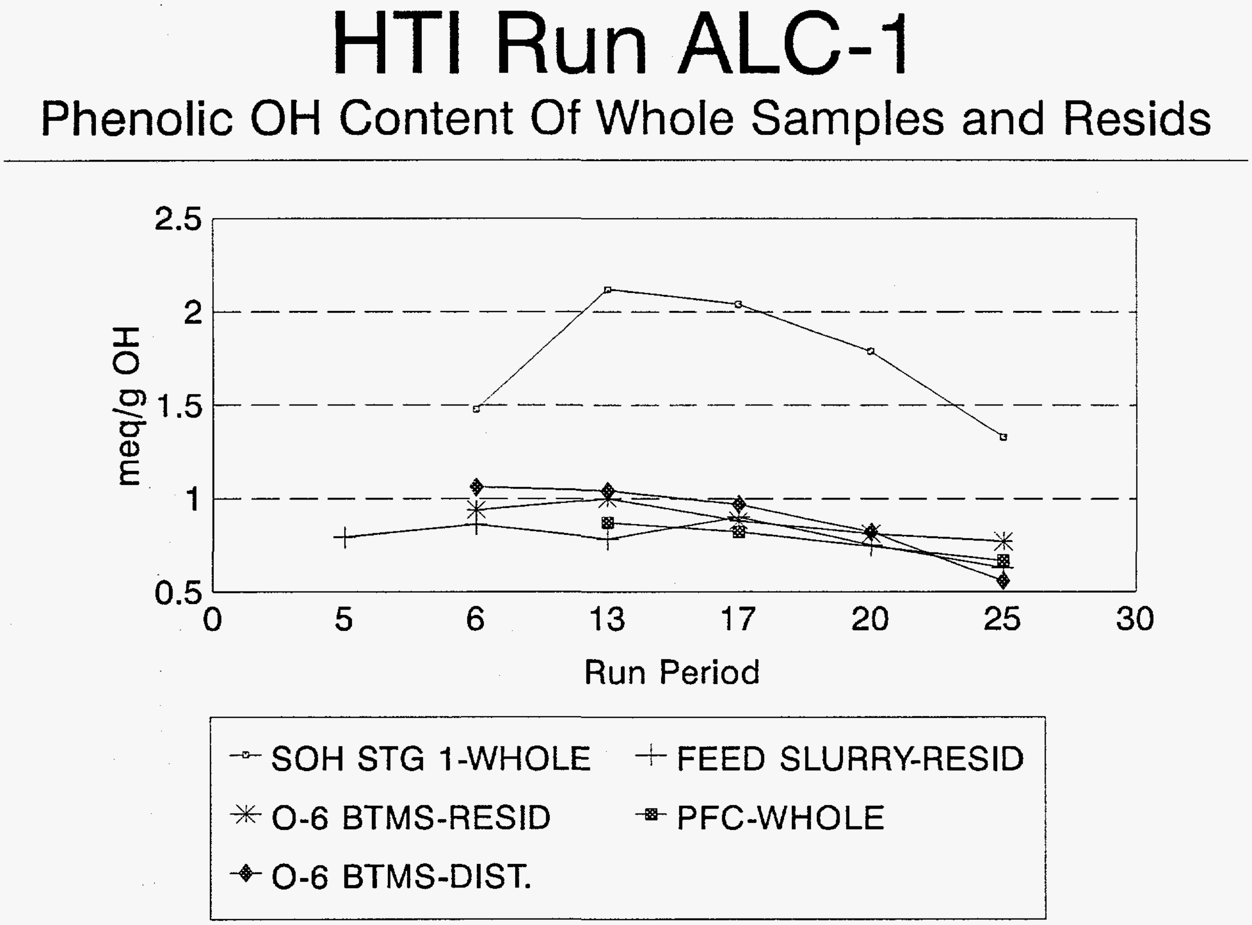


Figure 6. Donor Solvent Quality of Selected Whole and Distillate Samples from HTI Run ALC-1.



HTI Run ALC-1

Phenolic OH Content Of Whole Samples and Resids

Figure 7. Phenolic -OH Concentration in Process Stream Samples from HTI Run ALC-1.

APPENDIX 1

RECALIBRATION OF FTIR SPECTROSCOPIC PHENOLIC -OH METHOD

RECALIBRATION OF FTIR SPECTROSCOPIC METHOD FOR PHENOLIC -OH DETERMINATION

INTRODUCTION

Replacement of CONSOL's original Fourier-transform infrared (FTIR) spectroscopic system (Nicolet 170SX) with a new model (Nicolet Magna 550) required that the FTIR method used for determination of phenolic -OH concentration in liquefaction samples be recalibrated for the new system. Implementation of the method on the new system also required that the method be reprogrammed to facilitate analysis and calculations performed on the new system. To accomplish these tasks, several steps were performed. Solutions were prepared of six phenol standards at four concentrations each. The spectrum of each standard solution was run on the new FTIR instrument system, and the spectra were visually inspected for quality. The net absorbance of the phenolic -OH peak was measured using a re-implemented version of the software. The absorbance-concentration relation of each standard was checked statistically for consistency with the other concentrations of each standard. A regression was used to obtain the new calibration relationship, and the calbration was tested by analyzing authentic coal liquids that were run on both the old and new systems.

METHOD DETAILS AND VALIDATION

The six model compounds that were previously used as standards[1-3] were selected as standards for recalibration, except that 2,3,5-trimethyl phenol was substituted for the 2,4,5-trimethyl phenol used in the original calibration. Four solutions of each phenol standard were prepared and run on the new FTIR instrument. The spectra were visually inspected for quality, and repeats were run as needed. The solution concentrations were randomly designed to span a range of 8.5-50.0 mm-meq/L in concentration times pathlength (PL) to cover the range of the original calibration. The solution concentration of phenolic -OH was calculated from the spectrum of each standard solution using the original calibration equation

*conc*PL (in mm-meq/L) = 53.61*net absorbance - 0.1892*
(± 1.8 as the standard error of the Y estimate),

where net absorbance is the height of the phenol O-H stretch peak (found in the 3200-3400 cm^{-1} region) minus the baseline absorbance (integration over the 3650-3750 cm^{-1} region). From this, the sample concentration is obtained by

*conc (in meq/g) = (conc*PL)*(solution Vol in L)/((PL in mm)*(sample wt in g))*

Because the sample (weight) concentration of phenolic -OH in each phenol standard should be independent of solution concentration, this value (meq OH/g sample) provides a measure of agreement within the set of four solutions of each standard. These values were subjected to the Q-test for statistical outliers (at the 90% and higher confidence level). Questionable solutions of each standard were remade and rerun to obtain a new result to replace each outlying result. After a good set of data was obtained, regressions of net absorbance on conc*PL and vice versa were calculated using the four data points for each of the six standard phenols (i.e., six sets of regressions were calculated). Regressions of absorbance on conc*PL give the extinction coefficient or absorptivity of each of the phenols as the slope, whereas regressions of conc*PL on absorbance give the slope and intercept in a form useful as a calibration line for analysis. The six individual regressions of four points each gave regression coefficients (R^2) of 0.993 to 0.999, whereas the overall regression of 24 points gave a regression coefficient (R^2) of 0.955 (see Table 1). The individual regressions assured that the solution standards were of high quality. Figure 1 shows all the standard data, with identification of the subsets of different phenol standards. The tabulated data are given in Table 2.

The new calibration is given as

*conc*PL (in mm-meq/L) = 50.20(±2.32) *net absorbance + 1.19*
(±2.63 as the standard error of the Y estimate), $R^2 = 0.955$

from which the sample concentration is obtained as described above. The calibration error (standard error of the Y estimate) is higher than in the original calibration; this is discussed later.

In order to validate the calibration, a set of 10 samples was analyzed both on the old instrument with the old calibration, and on the new instrument with the new calibration. The sample set included several representative coal liquids, and a few model compounds (standards). For three of the samples, runs were made in duplicate on the new instrument and weighted averages were used to determine bias (i.e., each replicate contributed one-half as much to the total error as each single determination). The validation results are shown in Table 3. Figure 2 shows a parity plot of new versus reference (original) concentrations. In general, the peak position reported by the new calibration was biased 1.8 cm^{-1} lower than the original data (range 1-3 cm^{-1} lower).

For the entire set (3 standards, 2 high concentration samples, 5 normal concentration samples), the concentration bias was 0.00 meq/g, and the regression equation was

*new conc. = old conc. * 1.01(±0.02) - 0.04 (±0.19 as the standard error of the Y estimate),* $R^2 = 0.997$

For the set without the standards (2 high concentration samples, 5 normal concentration samples), the concentration bias was -0.08 meq/g,and the regression equation was

*new conc. = old conc. * 0.92(±0.01) + 0.02 (±0.08 as the standard error of the Y estimate),* $R^2 = 0.998$

For the set of normal concentration samples (5 normal concentration samples), the concentration bias was 0.02 meq/g, and the regression equation was

*new conc. = old conc. * 1.04(±0.01) + 0.003 (±0.01 as the standard error of the Y estimate),* $R^2 = 0.999$

The results indicate a trivial bias of less than 0.02 meq/g in magnitude for the samples. The phenolic extract sample with a low new result created a larger magnitude bias when the standards were omitted from the validation. The discrepancy between old and new results was larger for samples with higher phenolic -OH concentrations (i.e. >1.0 meq/g, which is outside the most common range for coal liquids). The largest concentration differences observed between the old instrument and new instrument amounted to a relative error of ca. 7%. The calibration regression showed a larger error than the original calibration, but the new one involved more standards and was much more rigorously set up from a statistical standpoint. It is likely that the earlier calibration underestimated the actual error. It appears that the calibration regression also represents a relative error of ca. 5%, but this error analysis may not be rigorous. A calculation of the calibration error for each sample was implemented in the new version of the method, according to the following formula:

conc error due to calibration (in meq/g) = 2.63(solution Vol in L)/((PL in mm)*(sample wt in g))*

This approach essentially disregards the error in measurement of solution volume, sample weight, and cell pathlength, and assumes that the error arising from differences in the model compounds used as standards is the largest error contributor. A printout of the computer program used to process each spectrum and perform the concentration calculations and a printout of the report output are shown in Listings 1 and 2. Because the old FTIR command set was not fully implemented on the new FTIR system, software implementation of the method on the new instrument was not straightforward. This required an advanced macro language (Visual Basic 3.0) implementation, rather than the simpler standard macro language provided by Nicolet.

REFERENCES

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3. Robbins, G. A.; Winschel, R. A.; Burke, F. P. "Phenolic -OH as a Process-Performance Indicator in Two-Stage Liquefaction", Am. Chem. Soc. Div. Fuel Chem. Prepr. 1985, 30(4), 155-163.

TABLE A1-1

REGRESSION RESULTS FOR FTIR SPECTROSCOPIC PHENOLIC -OH METHOD RECALIBRATION

OverallRegression Output: X=Conc., Y=Abs.			Overall Regression Output: X=Abs.,Y=Conc.		
Constant		0.0027	Constant		1.19
Std Err of Y Est		0.0512	Std Err of Y Est		2.63
R Squared		0.9552	R Squared		0.96
No. of Observations		24	No. of Observations		24
Degrees of Freedom		22	Degrees of Freedom		22
X Coefficient(s)	0.019		X Coefficient(s)	50.2	
Std Err of Coef.	0.0009		Std Err of Coef.	2.319	
Individual Regression Output: X=Conc., Y=Abs. No. of Observations=4, Degrees of Freedom =2			Individual Regression Output: X=Abs.,Y=Conc. No. of Observations=4, Degrees of Freedom =2		
2,3,5-trimethylphenol					
Constant		0.0192	Constant		-1.05
Std Err of Y Est		0.0111	Std Err of Y Est		0.62
R Squared		0.9991	R Squared		0.9991
X Coefficient(s)	0.0177		X Coefficient(s)	56.42	
Std Err of Coef.	0.0004		Std Err of Coef.	1.21	
2,5-ditertbutylphenol					
Constant		-0.027	Constant		1.58
Std Err of Y Est		0.0241	Std Err of Y Est		1.20
R Squared		0.9929	R Squared		0.9929
X Coefficient(s)	0.02		X Coefficient(s)	49.58	
Std Err of Coef.	0.0012		Std Err of Coef.	2.96	
2-naphthol					
Constant		-0.022	Constant		1.02
Std Err of Y Est		0.0133	Std Err of Y Est		0.58
R Squared		0.9981	R Squared		0.9981
X Coefficient(s)	0.0228		X Coefficient(s)	43.84	
Std Err of Coef.	0.0007		Std Err of Coef.	1.35	
9-phenanthrol					
Constant		-0.002	Constant		0.19
Std Err of Y Est		0.0163	Std Err of Y Est		0.96
R Squared		0.9969	R Squared		0.9969
X Coefficient(s)	0.017		X Coefficient(s)	58.81	
Std Err of Coef.	0.0007		Std Err of Coef.	2.32	
hydroquinone					
Constant		-0.03	Constant		1.51
Std Err of Y Est		0.0097	Std Err of Y Est		0.48
R Squared		0.9993	R Squared		0.9993
X Coefficient(s)	0.0201		X Coefficient(s)	49.77	
Std Err of Coef.	0.0004		Std Err of Coef.	0.94	
p-cresol					
Constant		-0.005	Constant		0.39
Std Err of Y Est		0.0198	Std Err of Y Est		1.00
R Squared		0.9957	R Squared		0.9957
X Coefficient(s)	0.0196		X Coefficient(s)	50.75	
Std Err of Coef.	0.0009		Std Err of Coef.	2.36	

TABLE A1-2

STANDARDS DATA FOR FTIR SPECTROSCOPIC PHENOLIC -OH METHOD RECALIBRATION

Std. No.	Phenol Standard	Mol weight	Eq/ Mol	g used per 25 mL sol'n	Actual Conc., meq/L (PL=1 mm)	Net Abs.	Pk. Pos., cm^{-1}
7	2,3,5-trimethylphenol	136.19	1	0.0299	8.78	0.170	3322
21	2,3,5-trimethylphenol	136.19	1	0.0856	25.14	0.477	3318
8	2,3,5-trimethylphenol	136.19	1	0.1202	35.30	0.637	3320
9	2,3,5-trimethylphenol	136.19	1	0.1654	48.58	0.879	3320
12	2,5-ditertbutylphenol	206.33	1	0.0932	18.07	0.317	3316
11	2,5-ditertbutylphenol	206.33	1	0.1502	29.12	0.564	3314
22	2,5-ditertbutylphenol	206.33	1	0.1654	32.07	0.639	3313
10	2,5-ditertbutylphenol	206.33	1	0.2389	46.31	0.885	3314
23	2-naphthol	144.17	1	0.0436	12.10	0.248	3279
15	2-naphthol	144.17	1	0.1000	27.75	0.624	3280
13	2-naphthol	144.17	1	0.1207	33.49	0.742	3280
14	2-naphthol	144.17	1	0.1328	36.85	0.807	3280
24	9-phenanthrol	194.23	1	0.0758	15.61	0.248	3245
18	9-phenanthrol	194.23	1	0.1060	21.83	0.384	3252
17	9-phenanthrol	194.23	1	0.1523	31.36	0.536	3250
16	9-phenanthrol	194.23	1	0.2320	47.78	0.803	3248
2	hydroquinone	110.11	2	0.0241	17.51	0.312	3331
3	hydroquinone	110.11	2	0.0259	18.82	0.351	3330
1	hydroquinone	110.11	2	0.0354	25.72	0.495	3330
19	hydroquinone	110.11	2	0.0680	49.41	0.960	3332
5	p-cresol	108.14	1	0.0404	14.94	0.278	3310
6	p-cresol	108.14	1	0.0881	32.59	0.657	3309
4	p-cresol	108.14	1	0.0920	34.03	0.664	3309
20	p-cresol	108.14	1	0.1223	45.24	0.869	3310

TABLE A1-3

VALIDATION DATA FOR FTIR SPECTROSCOPIC PHENOLIC -OH METHOD RECALIBRATION

		NEW		REFERENCE		DIFFERENCE	
SAMPLE	DESCRIPTION	CONC., meq/g	PK. POS., cm^{-1}	CONC., meq/g	PK. POS., cm^{-1}	CONC., meq/g	PK. POS., cm^{-1}
p-cresol	Std., MW=108.1	9.36	3309	9.25	NA	0.11	NA
3,4,5-trimethylphenol	Std., MW=136.2	7.46	3323	7.34	NA	0.12	NA
2-naphthol	Std., MW=144.2	7.27	3279	6.94	NA	0.33	NA
#3351	High	1.02	3294	1.25	3295	-0.22	-1
#3351 (Repeat)	High	1.07	3294	1.25	3295	-0.18	-1
#3290	High	5.80	3309	6.25	3311	-0.44	-2
#1981	Normal	0.86	3296	0.83	3298	0.03	-2
#1981 (Repeat)	Normal	0.88	3296	0.83	3298	0.05	-2
#3239	Normal	0.08	3315	0.09	3318	0.00	-3
#3239 (Repeat)	Normal	0.09	3315	0.09	3318	0.00	-3
#3231	Normal	0.09	3313	0.09	3314	0.00	-1
#3352	Normal	0.89	3296	0.85	3297	0.04	-1
#2051	Normal	0.00*	3357*	0.00*	3357*	0.00	NA
Bias as Weighted Avg. - Stds, High & Normal						0.00	NA
Bias as Weighted Avg. - High & Normal Conc.						-0.08	-1.7
Bias as Weighted Avg. - Normal Conc.						0.02	-1.8

*Peak found represents a non-phenolic component. The concentrations are reported here as 0.00 meq/g for the purpose of determining bias; a more accurate representation of the concentration is “none detected”.

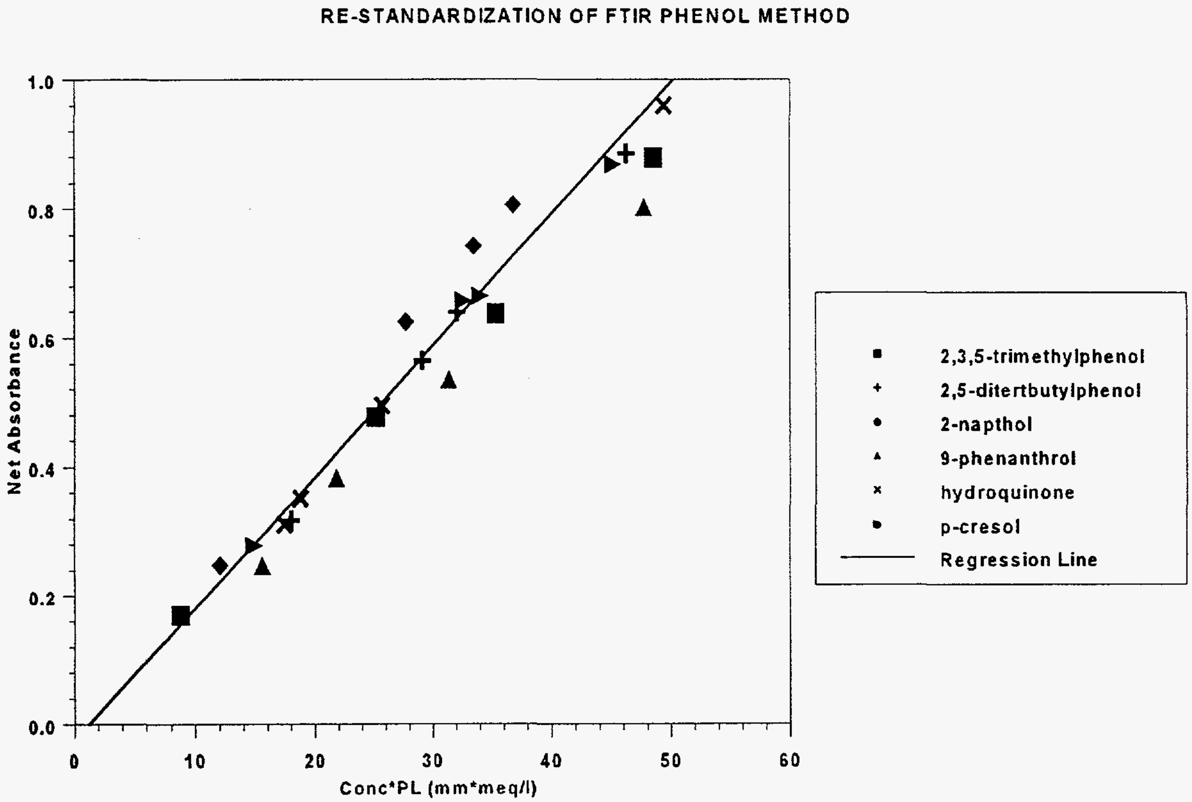


Figure A1-1. Calibration Data Showing Subsets of Each Standard and Overall Regression Line.

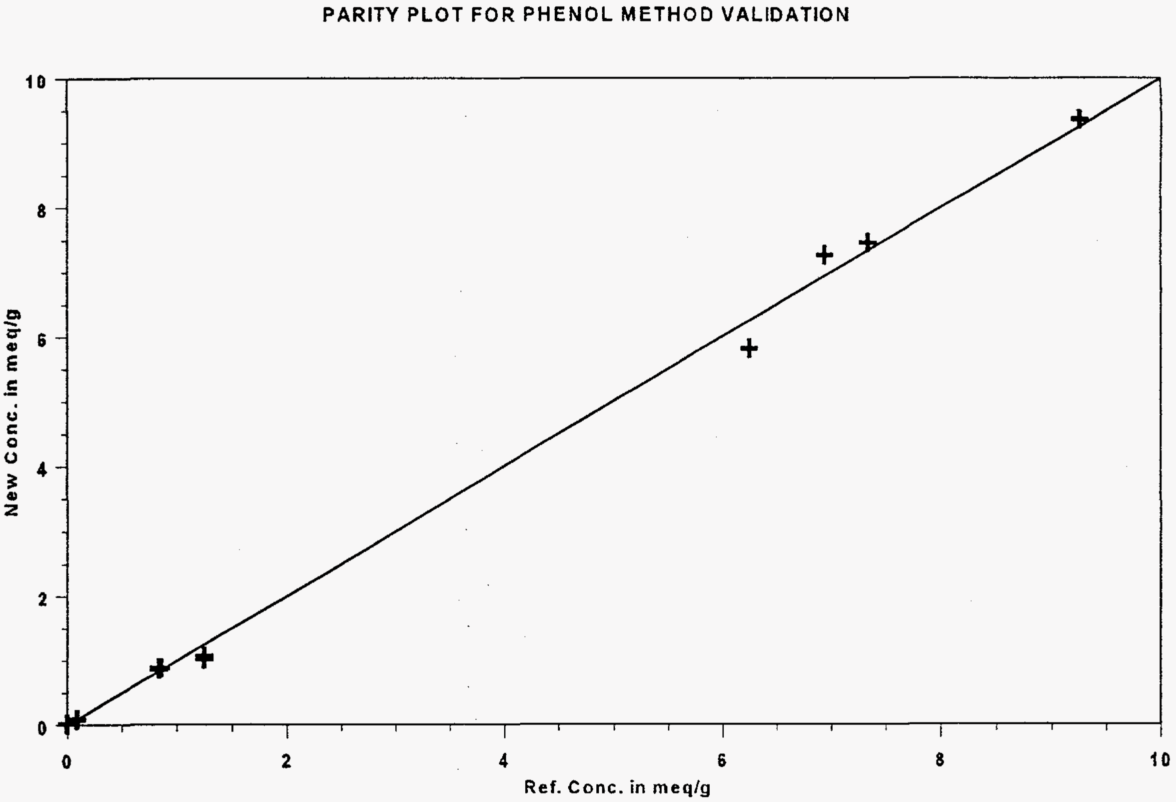


Figure A1-2. Parity Plot of Validation Results Obtained from New and Original Spectrometer and Calibration.

Listing A1-1 - Example Report Output for FTIR Spectroscopic Phenolic -OH Determination

```
 >>>>> START OF PHENOLIC -OH DETERMINATION <<<<<
TODAY'S DATE AND TIME: 4/1/97 10:32:15 AM
SPECTRUM FILENAME: C:\OMNIC\SPECTRA\PHENOLS\PHEN0190.SPA
SPECTRUM TITLE: HTI ALC-1 SOH 1STG OIL PER.25-WHOLE

Phenol peak height is .592 Abs. (Peak Height Method)
Phenol peak location is 3307.373 cm-1 (Peak Height Method)

MaxPeak: Normal Completion, 1 Peak(s) Found
Phenolic -OH peak height is .592 Abs.
Phenolic -OH peak location is 3307.72 cm-1
Max. Peak Position is 3307.72 cm-1
Max. Peak is .592 Abs.
Baseline is .00055 Abs., based on integration over 3750-3650 cm-1
Net absorbance is .5914 Abs.
Conc*PL for Phenolic -OH is 30.8783 mm meq/g
Solution Sample Volume is 2.00 mL
Sample Weight is 0.0463 g
IR Cell Pathlength is 1.00 mm
Amount of Phenolic -OH is 1.334 +/- 0.114 milliequivalents/gram of sample (meq/g)
>>>> END OF PHENOLIC -OH REPORT <<<<<
```

Listing A1-2 - Computer Program Listing for FTIR Spectroscopic Phenolic -OH Determination

Notes: Written in Visual Basic 3.0, Requires Nicolet Macros Pro and Nicolet OMNIC Software, Used on Nicolet Magna 550 Spectrometer

PHENCLC1.FRM - 1

```
VERSION 2.00
Begin Form Form1
   Caption         =   "Phenolic -OH Determination"
   ClientHeight    =   5865
   ClientLeft      =   60
   ClientTop       =   1680
   ClientWidth     =   7545
   Height          =   6270
   Left            =   0
   LinkTopic       =   "Form1"
   ScaleHeight     =   5865
   ScaleWidth      =   7545
   Top             =   1335
   Width           =   7665
   Begin CommandButton Command2
      Caption         =   "Calculate Results"
      Height          =   375
      Left            =   240
      TabIndex        =   10
      Top             =   4320
      Width           =   1815
   End
   Begin TextBox Text1
      Height          =   2175
      Left            =   240
      MultiLine       =   -1  'True
      ScrollBars      =   3  'Both
      TabIndex        =   9
      Text            =   "Text1"
      Top             =   120
      Width           =   7095
   End
   Begin TextBox Text8
      BackColor       =   &H0080FF80&
      Height          =   285
      Left            =   360
      TabIndex        =   8
      Text            =   "Text8"
      Top             =   3840
      Width           =   7095
   End
   Begin TextBox Text7
      BackColor       =   &H0080FFFF&
      Height          =   285
      Left            =   360
      TabIndex        =   7
      Text            =   "Text7"
      Top             =   3480
      Width           =   3495
```

```
End
Begin TextBox Text6
  Height        =  285
  Left        =  5760
  ScrollBars     =  2  'Vertical
  TabIndex      =  5
  Text          =  "Text6"
  Top           =  3480
  Width         =  1695
End
Begin TextBox Text5
  Height        =  285
  Left        =  3960
  TabIndex      =  4
  Text          =  "Text5"
```

PHENCLC1.FRM - 2

```
  Top           =  3480
  Width         =  1695
End
Begin TextBox Text4
  Height        =  285
  Left        =  3240
  TabIndex      =  3
  Text          =  "Text4"
  Top           =  3120
  Width         =  3495
End
Begin TextBox Text3
  Height        =  285
  Left        =  3240
  TabIndex      =  2
  Text          =  "Text3"
  Top           =  2760
  Width         =  3495
End
Begin TextBox Text2
  Height        =  285
  Left        =  3240
  TabIndex      =  1
  Text          =  "Text2"
  Top           =  2400
  Width         =  3495
End
Begin CommandButton Command1
  Caption       =  "Exit When Done"
  Height        =  375
  Left        =  4920
  TabIndex      =  0
  Top           =  4320
  Width         =  2535
End
Begin Label Label3
  Alignment     =  1  'Right Justify
  BackColor     =  &H000000C0&
  Caption       =  "Pathlength of IR Cell in mm:"
```

```
    ForeColor      =  &H00FFFFFF&
    Height       =  255
    Left        =  720
    TabIndex      =  6
    Top         =  3120
    Width        =  2415
  End
  Begin Label Label2
    Alignment      =  1  'Right Justify
    BackColor      =  &H000000C0&
    Caption       =  "Volume of Sample Solution in mL:"
    ForeColor      =  &H00FFFFFF&
    Height       =  255
    Left        =  240
    TabIndex      =  12
    Top         =  2400
    Width        =  2895
  End
  Begin Label Label1
    Alignment      =  1  'Right Justify
    BackColor      =  &H000000C0&
    Caption       =  "Weight of Sample in grams:"
    ForeColor      =  &H00FFFFFF&
    Height       =  255
    Left        =  720
```

PHENCLC1.FRM - 3

```
    TabIndex      =  11
    Top         =  2760
    Width        =  2415
  End
End
```

PHENCLC1.FRM - 1

```
Dim Results(100, 2), ResultStr, TotalLen, StartStr, EndStr, NumPeaks, MaxPeak, PeakPo
s
Dim NetAbs As Double, BslAbs As Double
Dim AmntPhen As Variant, ConcPL As Variant, ErrPhen As Variant
Dim SpecFile, SpecTitle

Sub GetResults ()
   StartStr = 1
   TotalLen = Len(ResultStr)
'  Error Trap for 0 peaks
If InStr(1, ResultStr, "No peaks found") > 0 GoTo NoPeaks
   StartStr = InStr(StartStr, ResultStr, "Position:")
   ResultStr = Mid(ResultStr, StartStr)
   StartStr = 10
   EndStr = InStr(StartStr, ResultStr, "Intensity:")
   Results(1, 1) = Val(Mid(ResultStr, StartStr, EndStr - StartStr))
   PeakPos = Results(1, 1)
   StartStr = EndStr
   ResultStr = Mid(ResultStr, StartStr)
   StartStr = 11
   EndStr = InStr(StartStr, ResultStr, "Position:")
```

```
   If EndStr = 0 Then EndStr = Len(ResultStr)
   Results(1, 2) = Val(Mid(ResultStr, StartStr, EndStr - StartStr))
   MaxPeak = Results(1, 2)
   StartStr = EndStr
   ResultStr = Mid(ResultStr, StartStr)
   TotalLen = Len(ResultStr)
   PeakNo = 1
' Stop here if only 1 peak
   If TotalLen < 20 GoTo LoopEnd
Loopstart:
   PeakNo = PeakNo + 1
   StartStr = 10
   EndStr = InStr(StartStr, ResultStr, "Intensity:")
   Results(PeakNo, 1) = Val(Mid(ResultStr, StartStr, EndStr - StartStr))
   StartStr = EndStr
   ResultStr = Trim(Mid(ResultStr, StartStr))
   StartStr = 11
   EndStr = InStr(StartStr, ResultStr, "Position:")
   If EndStr = 0 Then EndStr = Len(ResultStr)
   Results(PeakNo, 2) = Val(Mid(ResultStr, StartStr, EndStr - StartStr))
   If (Results(PeakNo, 2) > MaxPeak) Then
```

PHENCLC1.FRM - 2

```
   MaxPeak = Results(PeakNo, 2)
   PeakPos = Results(PeakNo, 1)
   End If
   ResultStr = Mid(ResultStr, EndStr)
   TotalLen = Len(ResultStr)
If (TotalLen > 30) GoTo Loopstart Else GoTo LoopEnd
NoPeaks:
   Text6.Text = "MaxPeak: No peaks were found"
   GoTo SubEnd
LoopEnd:
   Text6.Text = "MaxPeak: Normal Completion, " & PeakNo & " Peak(s) Found"
SubEnd:
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   Text6.Text = "Phenolic -OH peak height is " & MaxPeak & " Abs."
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   Text6.Text = "Phenolic -OH peak location is " & PeakPos & " cm-1"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   ExecuteOMNIC "CorrectedPeakArea 3800 3750 3650 3600"
   Result$ = GetOMNIC("Result Current")
    Text6.Text = "Max. Peak Position is " & PeakPos & " cm-1"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   Text6.Text = "Max. Peak is " & MaxPeak & " Abs."
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   BslAbs = GetVal(Result$, "Uncorrected:") / 100#
   Text6.Text = "Baseline is " & BslAbs & " Abs., based on integration over 3750-365
0 cm-1"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   NetAbs = Format(MaxPeak - BslAbs, "#0.00##")
   Text7.Text = "Net absorbance is " & NetAbs & " Abs."
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text7.Text
   ConcPL = Format((50.2 * NetAbs) + 1.19, "###0.00##")
   Text8.Text = "Conc*PL for Phenolic -OH is " & ConcPL & " mm meq/g"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text8.Text
```

```
   Text2.Text = "2.0"
   Text3.Text = "1.0"
   Text4.Text = "1.0"
End Sub

Sub Command1_Click ()
```

PHENCLC1.FRM - 3

```
"   ExecuteOMNIC "Select First"
"   ExecuteOMNIC "Set Display YAxisMode FullScale"
"  ExecuteOMNIC "CloseWindow No""Phenol sample"
"   ErrMsgBox
 End

End Sub

Sub Command2_Click ()
   SampleVol = Format(Val(Text2.Text), "#0.00")
   SampleWt = Format(Val(Text3.Text), "##0.00##")
   PathLen = Format(Val(Text4.Text), "#0.00")
   Text6.Text = "Solution Sample Volume is " & SampleVol & " mL"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   Text6.Text = "Sample Weight is " & SampleWt & " g"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   Text6.Text = "IR Cell Pathlength is " & PathLen & " mm"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   AmntPhen = ConcPL * SampleVol / (PathLen * SampleWt * 1000)
   AmntPhen = Format(AmntPhen, "##0.00#")
   ErrPhen = 2.63 * SampleVol / (PathLen * SampleWt * 1000)
   ErrPhen = Format(ErrPhen, "##0.00#")
   Text8.Text = "Amount of Phenolic -OH is " & AmntPhen & " +/- " & ErrPhen & " mill
iequivalents/gram of sample (meq/g)"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text8.Text
'
   Text6.Text = Chr(13) & Chr(10) & ">>>>> START OF PHENOLIC -OH DETERMINATION <<<<<
"
   Result$ = "TODAY'S DATE AND TIME: " & Date & " " & Time
   Text6.Text = Chr(13) & Chr(10) & Text6.Text & Chr(13) & Chr(10) & Result$
   Result$ = "SPECTRUM FILENAME: " & SpecFile
   Text6.Text = Chr(13) & Chr(10) & Text6.Text & Chr(13) & Chr(10) & Result$
   Result$ = "SPECTRUM TITLE: " & SpecTitle
   Text6.Text = Text6.Text & Chr(13) & Chr(10) & Result$ & Chr(13) & Chr(10)
   Text1.Text = Chr(13) & Chr(10) & Text6.Text & Chr(13) & Chr(10) & Text1.Text
   Text6.Text = ">>>> END OF PHENOLIC -OH REPORT <<<<<"
   Text1.Text = Text1.Text & Chr(13) & Chr(10) & Text6.Text
   Clipboard.Clear
   Clipboard.SetText Text1.Text
   ExecuteOMNIC "StartLogging""C:\OMNIC\LOG\PHENOLS.LOG"
   ExecuteOMNIC "LogDisplay On"
   ExecuteOMNIC "Paste"
   ErrMsgBox
   ExecuteOMNIC "StopLogging"
   ErrMsgBox
   LogFile = "C:\OMNIC\LOG\PHEN96aa.txt"
   TotalLen = Len(Result$)
   Open LogFile For Append Access Read Write Shared As #1 Len = TotalLen
```

```
    Result$ = Text1.Text
    Print #1, Result$;
    Close #1

    Text6.Text = "Last Error was " & Err
End Sub
```

PHENCLC1.FRM - 4

```
Sub Form_Load ()
    Load OmTalk
'
    I% = StartOMNIC(1, "")
    ExecuteOMNIC "DisplayLimits 4000 400 0.0 1.2"
    ExecuteOMNIC "Set Display ScaleAll True"
    SpecFile = GetOMNIC("Spectrum Filename")
    SpecTitle = GetOMNIC("Spectrum Title")
    ExecuteOMNIC "Set Display Mode OverlayMode"
    ExecuteOMNIC "Set Display YAxisMode FullScale"
    ExecuteOMNIC "DisplayLimits 4000 3000 0.0 1.2"
    ExecuteOMNIC "Set Display ScaleAll True"
    ExecuteOMNIC "Set Display YAxisMode FullScale"
    ExecuteOMNIC "CopySelectedSpectra"
    ExecuteOMNIC "LogDisplay On"
    ExecuteOMNIC "Paste"
    ExecuteOMNIC "Smooth 25"
    ExecuteOMNIC "PeakHeight 3300 Shift"
    ErrMsgBox
    Result$ = GetOMNIC("Result Current")
   PhenPeak = GetVal(Result$, "Y:")
   PhenPos = GetVal(Result$, "X:")
    Text1.Text = "Phenol peak height is " & PhenPeak & " Abs. (Peak Height Method)"
    Text1.Text = Text1.Text & Chr(13) & Chr(10) & "Phenol peak location is " & PhenPo
s & " cm-1 (Peak Height Method)"
    Text1.Text = Text1.Text & Chr(13) & Chr(10)
    ExecuteOMNIC "Set Display YAxisMode FullScale"
    ExecuteOMNIC "DisplayLimits 3400 3200 0.0 1.2"
    ExecuteOMNIC "Set Display YAxisMode FullScale"
    ExecuteOMNIC "MinMax"
    Result$ = GetOMNIC("Result Current")
    YMin = GetVal(Result$, "Min:")
    YMax = GetVal(Result$, "Max:")
    ExecuteOMNIC "CustomScale YMin YMax"
    ExecuteOMNIC "Display"
    ExecuteOMNIC "PeakPick 0.0 100"
'
    ErrMsgBox
    ResultStr = GetOMNIC("Result Current")
    Text2.Text = ""
    Text3.Text = ""
    Text6.Text = ""
    GetResults
End Sub
```

APPENDIX 2

SAMPLE REQUEST FOR HTI RUN PB-05

CONSOL Inc.
Research & Development
4000 Brownsville Road
Library, PA 15129-9566
412-854-6600
FAX: 412-854-6613
412-854-6748

August 2, 1996

Dr. V. Pradhan
Hydrocarbon Technologies, Inc.
P. O. Box 6047
New York and Puritan Avenues
Lawrenceville, NJ 08648

Dear Vivek:

Our sample request for Run PB-05 is described below. We are requesting material in sufficient quantities to allow for sample distribution to other research groups, as needed. We understand that you cannot always provide the requested amounts, and we can work with smaller quantities.

We would like to receive from each run condition: 1) 250 g of separator overhead (SOH) oil; 2) 250 g of atmospheric still overhead (ASOH); 3) 350 g of continuous atmospheric still (CAS) bottoms or its equivalent, such as O-6 bottoms; 4) 350 g of pressure-filter liquid (PFL); 5) 350 g of pressure-filter cake (PFC); 6) 350 g of feed slurry; and 7) 350 g of interstage sample (first-stage product). Please also include: 8) 250 g of the start-up/make-up oil from the beginning of the run; 9) 300 g sample of the feed coal; 10) 300 g sample of the feed resid; 11) 2 kg sample of the unprocessed feed auto-fluff; 12) 1 kg sample of the simulated MSW plastics; and 13) 250 g of SOH oil from any operating periods in which the in-line hydrotreater is bypassed.

For these samples, we prefer aliquots of the same samples HTI is using for material balance work-ups (i.e., from the last period of a run condition).

Let us know of any problem areas with this request. Thank you for your assistance.

Sincerely,

Gary
G. A. Robbins
Sr. Research Chemist

/ls

cc: R. M. Statnick
R. A. Winschel
S. D. Brandes
A. G. Comolli - HTI
M. A. Nowak - PETC
E. B. Klunder - PETC

APPENDIX 3

CRUDE OIL ASSAYS OF NET PRODUCTS OF HTI RUN PB-03

(Periods 6, 7, and 8 = Condition 2)
(Periods 9, 10, and 11 = Condition 3)

CONSOL Inc.
Research & Development
4000 Brownsville Road
Library, PA 15129-9566
412-854-6600
FAX: 412-854-6613
412-854-6683

November 15, 1996

Dr. M. A. Nowak
U.S. Department of Energy
Pittsburgh Energy Technology Center
P.O. Box 10940
Pittsburgh, PA 15236

Subject: DOE Contract DE-AC22-94PC93054

Dear Mike:

Enclosed are the reports on the crude oil assays of the two products of HTI Run PB-03. The on-line hydrotreater was in use during Periods 6, 7, and 8 (sample HTI PB-03-6, -7, and -8), and it was by-passed during periods 9, 10, and 11 (sample HTI PB-03-9, -10, and -11). CONSOL's preparation of the samples is described in the August 1996 status report for the subject contract.* These reports will be included in one of our Technical Progress Reports.

Sincerely,

Dick

*and in the main body of this report.

R. A. Winschel
Research Group Leader
Exploratory Research Group

/ls

cc: A. G. Comolli - HTI
P.-Z. Zhou - BRSC

NOV 1 4 1996 R.A.W.

Inchcape Testing Services
Caleb Brett

Consol, Inc.
4000 Brownsville Road
Library, PA 15129-9566

11/04/96

Attention: R.A. Winschell

Reference: Crude Assay on "HTI PB-03-6, 7, 8" received in September 1996

Mr. Winschell,

The following results are based on a composite of (2) separate distillations performed on this sample. The Distillation Data Report provides a breakdown of the separate distillations. Should you have any questions concerning this report, you may contact me at (713) 844-3311, or by fax at (713) 844-3330.

Sincerely,

Robert Kelly
Distillation Manager

Inchcape Testing Services
Caleb Brett

November 12, 1996

Houston, Texas

Our Reference : HO/96-005162
Your Reference : PO# 01-001-033668

Consol, Inc.
Research & Development
4000 Brownsville Road
Library, PA 15129-9566

ATTN: R.A. Winschel

Reference: To perform "CRUDE ASSAY" on "HT1 PB-03-6,7,8" received in September 1996.

Dear Mr. Winschel:

Please find enclosed the original report on the above referenced submitted sample(s) and our invoice for services rendered.

Should you have any questions regarding this report, please do not hesitate to contact us at your convenience.

We trust you find all in order and thank you for requesting our services.

Very Truly Yours,

INCHCAPE TESTING SERVICES
CALEB BRETT U.S.A.

John Mowrey

John Mowrey
Laboratory Manager

Enclosures:
JM/rm

Inchcape Testing Services
Caleb Brett

9809 Rowlett Road
Houston, TX 77075
Phone: (713) 946-2420
Fax: (713) 946-0545

Your Ref: PO# 01-001-033668

Date: 8-NOV-1996

Laboratory Report No. 96-005162-0-HOUS; 1

Consol, Inc.
4000 Brownsville Road
Library, PA 15129-9566

For the Attention of R. A. Winschel

SAMPLE DETAILS: 8 Sample(s) received on 16-SEP-1996

SOURCE : Consol, Inc.

DESCRIPTION :

HTI PB-03-6, 7, 8 CRUDE OIL	LAB REF
Sample As Received	001-00
IBP-70 Deg. F	002-00
70-180 Deg. F	003-00
180-350 Deg. F	004-00
350-400 Deg. F	005-00
400-550 Deg. F	006-00
550-650 Deg. F	007-00
650+ Deg. F	008-00

CONTAINERS : 5 Gallon Can

SEALS : NONE

RESULTS : SEE ATTACHED SHEETS

(TOTAL NUMBER OF PAGES 8)

Approved by: John Mowrey
John Mowrey

Laboratory Report No. 96-005162-0-HOUS; 1 - Page 2 of 8

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-001-00	Sample As Received

Test		Method	001-00
API Gravity @ 60/60 F		D4052	38.0
Specific Gravity @ 60/60 F		D4052	0.8347
Carbon	Wt. %	D5291	84.57
Hydrogen	Wt. %	D5291	12.92
Sulfur Content	Wt. %	D4294	0.06
Total Nitrogen	ppm	D4629	41.8
Methanol	Wt. %	D4815	<0.01
Ethanol	Wt. %	D4815	<0.01
t-Butanol	Wt. %	D4815	<0.01
Iso-Propanol	Wt. %	D4815	<0.01
n-Propanol	Wt. %	D4815	<0.01
Sec-Butanol	Wt. %	D4815	<0.01
Iso-Butanol	Wt. %	D4815	<0.01
n-Butanol	Wt. %	D4815	<0.01
MTBE	Wt. %	D4815	<0.01
ETBE	Wt. %	D4815	<0.01
DIPE	Wt. %	D4815	<0.01
TAME	Wt. %	D4815	<0.01
t-Pentanol	Wt. %	D4815	<0.01
Total Oxygenates	Wt. %	D4815	<0.01
Oxygen	Wt. %	By Difference	2.44
Ash Content for Digestion	Wt. %	D482	0.002
Vanadium	ppm	ICP	0.1
Nickel	ppm	ICP	0.1
Iron	ppm	ICP	0.2
Copper	ppm	ICP	0.1
Freezing Point	Deg. F	D2386	25.0
Microcarbon Residue	Wt. %	D4530	<0.1
N-Heptane Insolubles	Wt. %	D3279	0.05
Boiling Point Distribution		D5307	See Attached

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-002-00	IBP-70 Deg. F

Test	Method	002-00
API Gravity @ 60/60 F (Charge #1)	G.C.	113.3
API Gravity @ 60/60 F (Charge #2)	G.C.	109.2
Specific Gravity @ 60/60 F (Charge #1)	G.C.	0.5780
Specific Gravity @ 60/60 F (Charge #2)	G.C.	0.5880
DHA	GC-DHA	See Attached

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-003-00	70-180 Deg. F

Test		Method	003-00
API Gravity @ 60/60 F		D4052	65.7
Specific Gravity @ 60/60 F		D4052	0.7175
Carbon	Wt. %	D5291	84.48
Hydrogen	Wt. %	D5291	15.51
Sulfur Content	Wt. %	D4294	<0.01
Total Nitrogen	ppm	D4629	35.5
Vapor Pressure	psi	D323	8.4
Paraffins	Vol. %	G.C.	42.98
Olefins	Vol. %	G.C.	0.33
Naphthenes	Vol. %	G.C.	52.54
Aromatics	Vol. %	G.C.	1.15
Total N & A	Vol. %	G.C.	53.69
Benzene Content	Vol. %	G.C.	1.03
Total Acid Number	mgKOH/g	D974	0.13
Corrosion 3 hrs @ 122 F		D130	3b
Existent Gum	mg/100mL	D381	1
Oxidation Stability	min.	D525	>240
Research Octane Number		D2699	76.1
Motor Octane Number		D2700	73.6
Initial Boiling Point	Deg. F	D86	107
@ 5% Evaporated	Deg. F		132
@ 10% Evaporated	Deg. F		137
@ 20% Evaporated	Deg. F		143
@ 30% Evaporated	Deg. F		148
@ 40% Evaporated	Deg. F		153
@ 50% Evaporated	Deg. F		157
@ 60% Evaporated	Deg. F		161
@ 70% Evaporated	Deg. F		165
@ 80% Evaporated	Deg. F		168
@ 90% Evaporated	Deg. F		172
@ 95% Evaporated	Deg. F		174
Final Boiling Point	Deg. F		181
Recovery	Vol. %		99.0
Residue	Vol. %		0.6
Loss	Vol. %		0.4

Laboratory Report No. 96-005162-0-HOUS; 1 - Page 4 of 8

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-004-00	180-350 Deg. F

Test		Method	004-00
API Gravity @ 60/60 F		D4052	49.8
Specific Gravity @ 60/60 F		D4052	0.7804
Carbon	Wt. %	D5291	85.92
Hydrogen	Wt. %	D5291	14.44
Sulfur Content	Wt. %	D4294	0.03
Total Nitrogen	ppm	D4629	22.5
Mercaptan Sulfur Content	ppm	UOP163	25
Vapor Pressure	psi	D323	1.4
Paraffins	Vol. %	G.C.	18.56
Olefins	Vol. %	G.C.	<0.01
Naphthenes	Vol. %	G.C.	74.10
Aromatics	Vol. %	G.C.	7.34
Total N & A	Vol. %	G.C.	81.44
Total Acid Number	mgKOH/g	D974	0.14
Corrosion 3 hrs @ 122 F		D130	4a
Existent Gum	mg/100mL	D381	3
Oxidation Stability	min.	D525	>240
Research Octane Number		D2699	60.2
Motor Octane Number		D2700	58.0
Initial Boiling Point	Deg. F	D86	217
@ 5% Evaporated	Deg. F		226
@ 10% Evaporated	Deg. F		230
@ 20% Evaporated	Deg. F		236
@ 30% Evaporated	Deg. F		242
@ 40% Evaporated	Deg. F		250
@ 50% Evaporated	Deg. F		260
@ 60% Evaporated	Deg. F		273
@ 70% Evaporated	Deg. F		288
@ 80% Evaporated	Deg. F		304
@ 90% Evaporated	Deg. F		323
@ 95% Evaporated	Deg. F		336
Final Boiling Point	Deg. F		348
Recovery	Vol. %		97.3
Residue	Vol. %		1.0
Loss	Vol. %		1.7

Laboratory Report No. 96-005162-0-HOUS; 1 - Page 5 of 8

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-005-00	350-400 Deg. F

Test		Method	005-00
API Gravity @ 60/60 F		D4052	36.2
Specific Gravity @ 60/60 F		D4052	0.8437
Carbon	Wt. %	D5291	86.38
Hydrogen	Wt. %	D5291	13.17
Sulfur Content	Wt. %	D4294	0.03
Total Nitrogen	ppm	D4629	55.8
Mercaptan Sulfur Content	ppm	UOP163	16
Viscosity @ -20 C	cSt	D445	3.832
Viscosity @ 100 C	cSt	D445	0.569
Freezing Point	Deg. F	D2386	-76.0
Pour Point	Deg. F	D97	<-94.0
Flash Point, TCC	Deg. F	D56	135
Vapor Pressure	psi	D323	0.2
Luminometer Number		D1740	42
Smoke Point	mm	D1322	18
Paraffins	Vol. %	G.C.	13.13
Olefins	Vol. %	G.C.	2.40
Naphthenes	Vol. %	G.C.	60.58
Aromatics	Vol. %	G.C.	23.89
Total N & A	Vol. %	G.C.	84.47
Naphthalenes	Vol. %	D1840	0.36
Total Acid Number	mgKOH/g	D974	0.08
Corrosion 3 hrs @ 122 F		D130	4a
Existent Gum	mg/100mL	D381	6
Tube Rating		D3241	4.0
Pressure Drop	mmHg	D3241	>125.0
Oxidation Stability	min.	D525	>240
Research Octane Number		D2699	54.7
Motor Octane Number		D2700	52.1
Cetane Number (Apparent)		D613	29.9
Initial Boiling Point	Deg. F	D86	356
@ 5% Evaporated	Deg. F		360
@ 10% Evaporated	Deg. F		361
@ 20% Evaporated	Deg. F		361
@ 30% Evaporated	Deg. F		363
@ 40% Evaporated	Deg. F		365
@ 50% Evaporated	Deg. F		366
@ 60% Evaporated	Deg. F		368
@ 70% Evaporated	Deg. F		371
@ 80% Evaporated	Deg. F		374
@ 90% Evaporated	Deg. F		378
@ 95% Evaporated	Deg. F		382
Final Boiling Point	Deg. F		401
Recovery	Vol. %		99.0
Residue	Vol. %		1.0
Loss	Vol. %		0.0
Net Heat of Combustion	BTU/lb	D1405	18318

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-006-00	400-550 Deg. F

Test		Method	006-00
API Gravity @ 60/60 F		D4052	27.5
Specific Gravity @ 60/60 F		D4052	0.8898
Carbon	Wt. %	D5291	87.79
Hydrogen	Wt. %	D5291	12.14
Sulfur Content	Wt. %	D4294	0.02
Total Nitrogen	ppm	D4629	31.8
Basic Nitrogen	ppm	UOP269	22
Mercaptan Sulfur Content	ppm	UOP163	17
Viscosity @ -20 C	cSt	D445	12.65
Viscosity @ 40 C	cSt	D445	2.203
Viscosity @ 100 C	cSt	D445	0.952
Freezing Point	Deg. F	D2386	-43.0
Pour Point	Deg. F	D97	-49.0
Aniline Point	Deg. F	D611	91.0
Flash Point, TCC	Deg. F	D56	195
Luminometer Number		D1740	23
Smoke Point	mm	D1322	11
Paraffins	Vol. %	G.C.	11.52
Olefins	Vol. %	G.C.	13.50
Naphthenes	Vol. %	G.C.	39.10
Aromatics	Vol. %	G.C.	35.88
Total N & A	Vol. %	G.C.	74.98
Naphthalenes	Vol. %	D1840	5.11
Corrosion 3 hrs @ 122 F		D130	3a
Existent Gum	mg/100mL	D381	8
Tube Rating		D3241	>4.0
Pressure Drop	mmHg		>125.0
Cetane Number		D613	31.6
Initial Boiling Point	Deg. F	D86	428
@ 5% Evaporated	Deg. F		436
@ 10% Evaporated	Deg. F		442
@ 20% Evaporated	Deg. F		446
@ 30% Evaporated	Deg. F		451
@ 40% Evaporated	Deg. F		457
@ 50% Evaporated	Deg. F		464
@ 60% Evaporated	Deg. F		471
@ 70% Evaporated	Deg. F		480
@ 80% Evaporated	Deg. F		491
@ 90% Evaporated	Deg. F		503
@ 95% Evaporated	Deg. F		508
Final Boiling Point	Deg. F		524
Recovery	Vol. %		99.0
Residue	Vol. %		0.9
Loss	Vol. %		0.1
Net Heat of Combustion	BTU/lb	D1405	17937
Total Acid Number	mgKOH/g	D974	0.03

Laboratory Report No. 96-005162-0-HOUS; 1 - Page 7 of 8

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-007-00	550-650 Deg. F

Test		Method	007-00
API Gravity @ 60/60 F		D4052	20.9
Specific Gravity @ 60/60 F		D4052	0.9285
Carbon	Wt. %	D5291	88.65
Hydrogen	Wt. %	D5291	12.19
Sulfur Content	Wt. %	D4294	0.02
Total Nitrogen	ppm	D4629	19.0
Basic Nitrogen	ppm	UOP269	9
Viscosity @ 40 C	cSt	D445	7.080
Viscosity @ 100 C	cSt	D445	1.938
Pour Point	Deg. F	D97	10.4
Aniline Point	Deg. F	D611	109.0
Flash Point (Method A)	Deg. F	D93	>200
Paraffins	Vol. %	G.C.	20.89
Olefins	Vol. %	G.C.	23.10
Naphthenes	Vol. %	G.C.	8.26
Aromatics	Vol. %	G.C.	47.75
Total N & A	Vol. %	G.C.	56.01
Bromine Number		D1159	0.2
Corrosion 3 hrs @ 122 F		D130	1a
Cetane Number		D613	34.8
Simulated Distillation		D2887	See Attached

Inchcape Testing Services
Caleb Brett

Laboratory Report No. 96-005162-0-HOUS; 1 - Page 8 of 8

Sample ID	Description
	HTI PB-03-6, 7, 8 CRUDE OIL
96-005162-0-HOUS-008-00	650+ Deg. F

Test		Method	008-00
API Gravity @ 60/60 F		D4052	21.4
Specific Gravity @ 60/60 F		D4052	0.9254
Carbon	Wt. %	D5291	87.69
Hydrogen	Wt. %	D5291	12.29
Sulfur Content	Wt. %	D4294	0.61
Total Nitrogen	ppm	D4629	79.0
Basic Nitrogen	ppm	UOP269	34
Viscosity @ 40 C	cSt	D445	20.31
Viscosity @ 100 C	cSt	D445	3.791
Pour Point	Deg. F	D97	84.0
Aniline Point	Deg. F	D611	169.0
Flash Point (Method A)	Deg. F	D93	>200
Microcarbon Residue	Wt. %	D4530	<0.1
Corrosion 3 hrs @ 122 F		D130	1b
Cetane Number		D613	N/A
Initial Boiling Point	Deg. F	D1160	655
@ 5% Recovery	Deg. F		675
@ 10% Recovery	Deg. F		678
@ 20% Recovery	Deg. F		683
@ 30% Recovery	Deg. F		684
@ 40% Recovery	Deg. F		690
@ 50% Recovery	Deg. F		697
@ 60% Recovery	Deg. F		708
@ 70% Recovery	Deg. F		724
@ 80% Recovery	Deg. F		740
@ 90% Recovery	Deg. F		761
@ 95% Recovery	Deg. F		779
Final Boiling Point	Deg. F		839
Recovery	Vol. %		99.0
Residue + Loss	Vol. %		1.0

SAMPLE: 96-5162-1R
FILE: c:\tc4\gc10\5162-1r
PARAMETER: d2887

OCT 15, 1996 - 13:31:13
SIMDIS EXPERT V5.0
Page 2

Boiling Point Distribution

ASTM D-2887

%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)
IBP	108.1	42.3	**40**	358.1	181.2	**80**	559.0	292.8
1	118.3	47.9	**41**	361.6	183.1	**81**	564.5	295.8
2	147.1	63.9	**42**	368.8	187.1	**82**	569.5	298.6
3	159.7	70.9	**43**	374.7	190.4	**83**	575.3	301.8
4	168.0	75.6	**44**	379.6	193.1	**84**	579.2	304.0
5	177.4	80.8	**45**	384.6	195.9	**85**	584.5	306.9
6	184.8	84.9	**46**	389.6	198.7	**86**	590.8	310.4
7	189.3	87.4	**47**	393.8	201.0	**87**	597.4	314.1
8	194.6	90.3	**48**	396.0	202.2	**88**	602.5	316.9
9	202.1	94.5	**49**	401.4	205.2	**89**	609.3	320.7
10	211.2	99.6	**50**	408.2	209.0	**90**	617.0	325.0
11	216.1	102.3	**51**	414.0	212.2	**91**	624.9	329.4
12	217.8	103.2	**52**	418.5	214.7	**92**	631.9	333.3
13	219.4	104.1	**53**	423.5	217.5	**93**	641.6	338.7
14	221.4	105.2	**54**	430.3	221.3	**94**	650.5	343.6
15	226.4	108.0	**55**	433.3	222.9	**95**	664.5	351.4
16	240.7	115.9	**56**	439.1	226.2	**96**	678.1	358.9
17	245.5	118.6	**57**	444.7	229.3	**97**	696.1	368.9
18	252.7	122.6	**58**	450.2	232.3	**98**	721.9	383.3
19	260.0	126.7	**59**	454.4	234.7	**99**	756.6	402.6
20	262.8	128.2	**60**	458.5	236.9	**FBP**	789.7	420.9
21	269.7	132.1	**61**	464.0	240.0			
22	271.0	132.8	**62**	468.4	242.4			
23	272.0	133.3	**63**	474.4	245.8			
24	276.1	135.6	**64**	480.0	248.9			
25	284.5	140.3	**65**	484.2	251.2			
26	292.6	144.8	**66**	488.2	253.4			
27	296.7	147.1	**67**	492.4	255.8			
28	304.6	151.4	**68**	497.9	258.8			
29	308.3	153.5	**69**	502.4	261.3			
30	313.0	156.1	**70**	507.7	264.3			
31	315.8	157.7	**71**	511.7	266.5			
32	321.6	160.9	**72**	517.5	269.7			
33	329.9	165.5	**73**	521.4	271.9			
34	332.7	167.1	**74**	526.0	274.4			
35	335.7	168.7	**75**	531.8	277.7			
36	341.0	171.7	**76**	537.1	280.6			
37	345.8	174.3	**77**	542.2	283.4			
38	350.6	177.0	**78**	548.4	286.9			
39	354.3	179.1	**79**	553.1	289.5			

Start Time: 0.2 minutes
End Time: 24.3 minutes
Area: 139559232.0
Slice Width: 0.80 sec
Baseline Subtracted: c:\tc4\gc10\1014b

Sample Offset: 21859.9
Baseline Offset: 22729.7
Calibration File: 1014rt
Calibration Date: 10/16/96

Boiling Point Distribution

ASTM D-2887

%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)
IBP	531.2	277.3	**40**	600.3	315.7	**80**	651.9	344.4
1	539.0	281.7	**41**	601.5	316.4	**81**	653.1	345.1
2	545.9	285.5	**42**	602.8	317.1	**82**	654.4	345.8
3	550.3	287.9	**43**	604.1	317.8	**83**	655.7	346.5
4	553.2	289.6	**44**	605.3	318.5	**84**	656.9	347.2
5	555.6	290.9	**45**	606.6	319.2	**85**	658.2	347.9
6	557.7	292.1	**46**	607.9	319.9	**86**	659.4	348.6
7	559.4	293.0	**47**	609.1	320.6	**87**	660.7	349.3
8	560.9	293.8	**48**	610.4	321.3	**88**	662.0	350.0
9	562.3	294.6	**49**	611.7	322.1	**89**	663.2	350.7
10	563.6	295.3	**50**	613.0	322.8	**90**	664.5	351.4
11	564.8	296.0	**51**	614.2	323.4	**91**	665.7	352.1
12	566.1	296.7	**52**	615.5	324.2	**92**	667.0	352.8
13	567.3	297.4	**53**	616.8	324.9	**93**	668.4	353.6
14	568.6	298.1	**54**	618.0	325.6	**94**	669.9	354.4
15	569.8	298.8	**55**	619.3	326.3	**95**	671.7	355.4
16	571.1	299.5	**56**	620.6	327.0	**96**	673.5	356.4
17	572.3	300.2	**57**	621.9	327.7	**97**	675.5	357.5
18	573.6	300.9	**58**	623.1	328.4	**98**	678.3	359.1
19	574.8	301.6	**59**	624.4	329.1	**99**	682.6	361.4
20	576.1	302.3	**60**	625.7	329.8	**FBP**	686.8	363.8
21	577.3	302.9	**61**	627.0	330.6			
22	578.5	303.6	**62**	628.3	331.3			
23	579.7	304.3	**63**	629.6	332.0			
24	580.9	304.9	**64**	630.9	332.7			
25	582.1	305.6	**65**	632.2	333.4			
26	583.3	306.3	**66**	633.5	334.2			
27	584.6	307.0	**67**	634.9	334.9			
28	585.8	307.7	**68**	636.2	335.7			
29	587.0	308.3	**69**	637.5	336.4			
30	588.2	309.0	**70**	638.8	337.1			
31	589.4	309.7	**71**	640.1	337.8			
32	590.6	310.3	**72**	641.4	338.6			
33	591.8	311.0	**73**	642.7	339.3			
34	593.0	311.7	**74**	644.1	340.1			
35	594.2	312.3	**75**	645.4	340.8			
36	595.4	313.0	**76**	646.7	341.5			
37	596.7	313.7	**77**	648.0	342.2			
38	597.9	314.4	**78**	649.3	342.9			
39	599.1	315.1	**79**	650.6	343.7			

Start Time: 0.7 minutes
End Time: 16.4 minutes
Area: 329832608.0
Slice Width: 0.80 sec
Baseline Subtracted: c:\tc4\sd6890\1111b

Sample Offset: 8544.0
Baseline Offset: 8792.0
Calibration File: 1111rt
Calibration Date: 11/11/96

Inchcape Testing Services
Caleb Brett

WinAssay '95
Version 1.00

Final Reports

Client Name:	**Consol Inc.**
Sample ID:	**HTI PB-03-6,7,8 (Charge #1)**
Laboratory ID:	**96-005162**
Date:	**9/25/96**
Operator:	**Robert Kelly**

Distillation Summary Report

Prepared For: Consol Inc.
Sample ID: HTI PB-03-6,7,8 (Charge #1)
Date: 9/25/96

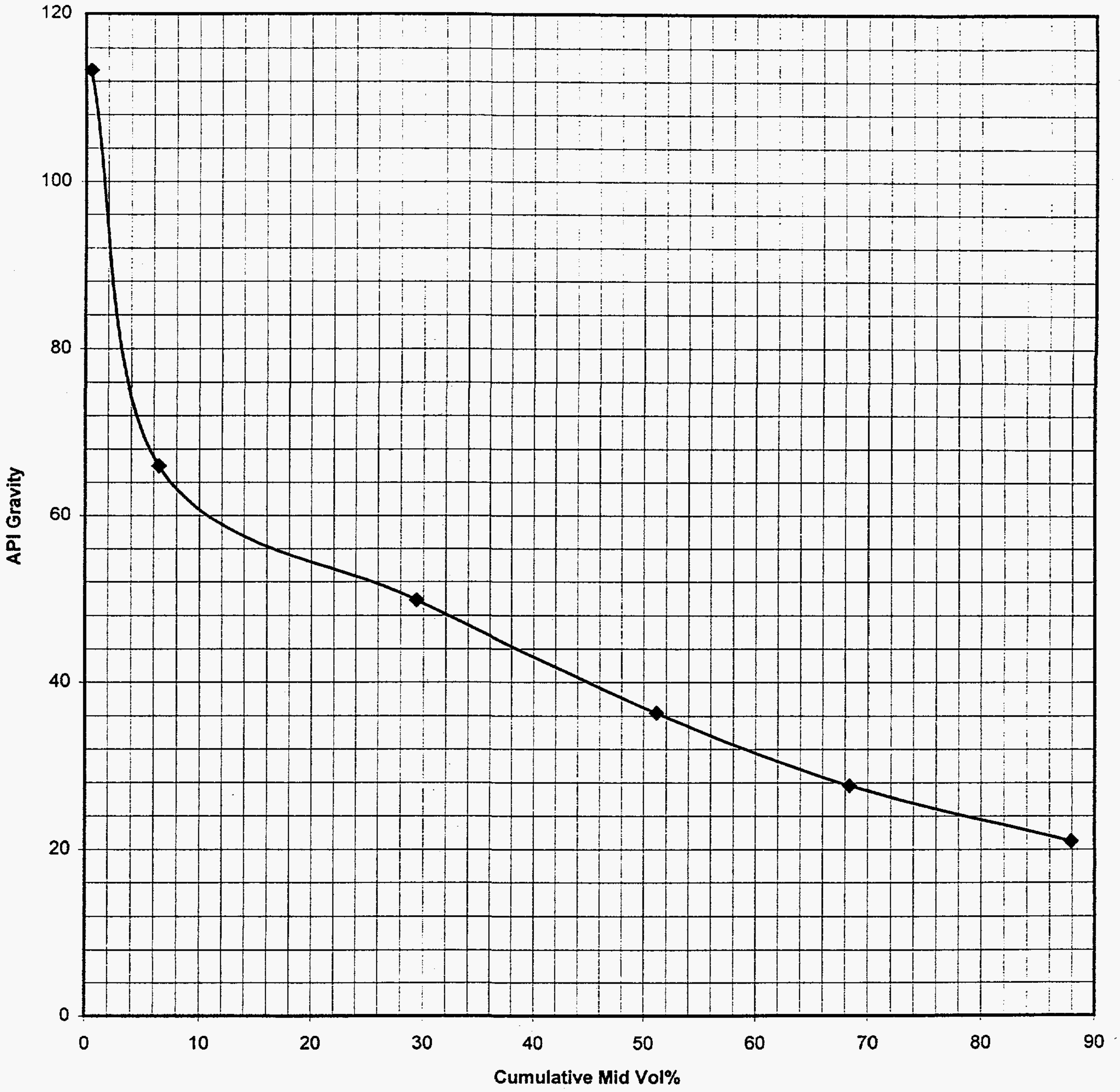
Cut Temp	Degrees F	DUMP WT(g)	Specific Gravity	MLS	LIQ VOL%	CUM. LIQ VOL%	WT%	CUM WT%	API GRAVITY	MID LIQ VOL%
TO										
ASTM D2892 Distillation Yields										
IBP	70	61.80	0.5780	106.92	0.94	0.94	0.65	0.65	113.31	0.47
70	180	896.90	0.7166	1251.60	11.00	11.94	9.45	10.10	65.96	6.44
180	350	3092.80	0.7803	3963.60	34.85	46.79	32.58	42.67	49.84	29.37
350	400	829.60	0.8430	984.10	8.65	55.44	8.74	51.41	36.35	51.12
400	550	2613.00	0.8892	2938.60	25.84	81.28	27.52	78.94	27.63	68.36
550	650	1399.90	0.9282	1508.19	13.26	94.54	14.75	93.68	20.95	87.91
650+		600.00	0.9248	648.79	5.70	100.24	6.32	100.00	21.51	

Loss (Grams): 9.8 (0.10 Wt.%)
Distribution: (2/3) 6.5 g to IBP-70 F
(1/3) 3.3 g to 70-180 F

WinAssay '95 Quality Control Applications

Cum. Mid Vol% v. API Gravity

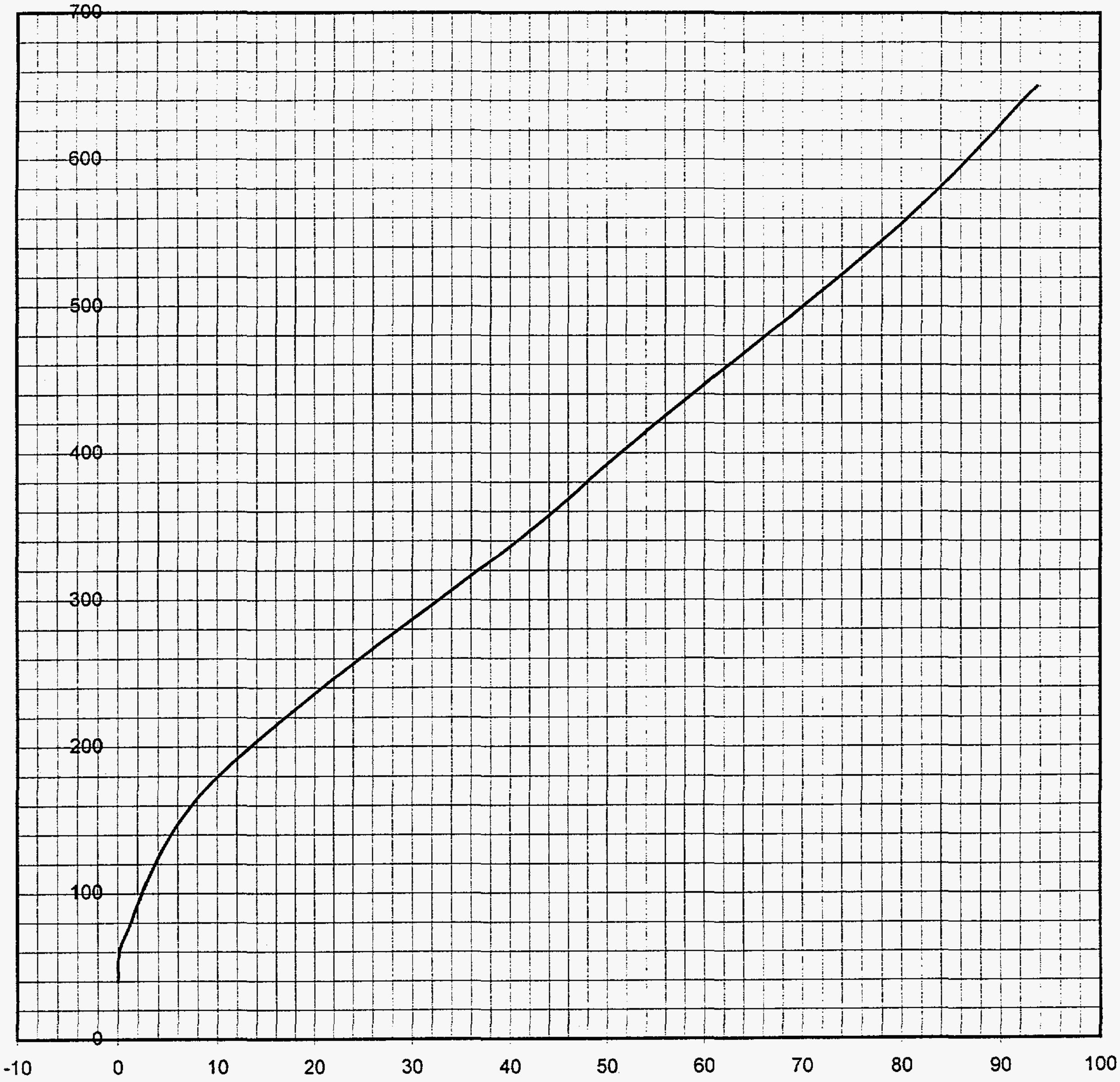
HTI PB-03-6,7,8 (Charge #1)



WinAssay '95
True Boiling Point Curve
Vaporline Temperature v. Cumulative Wt% Yield

Sample ID

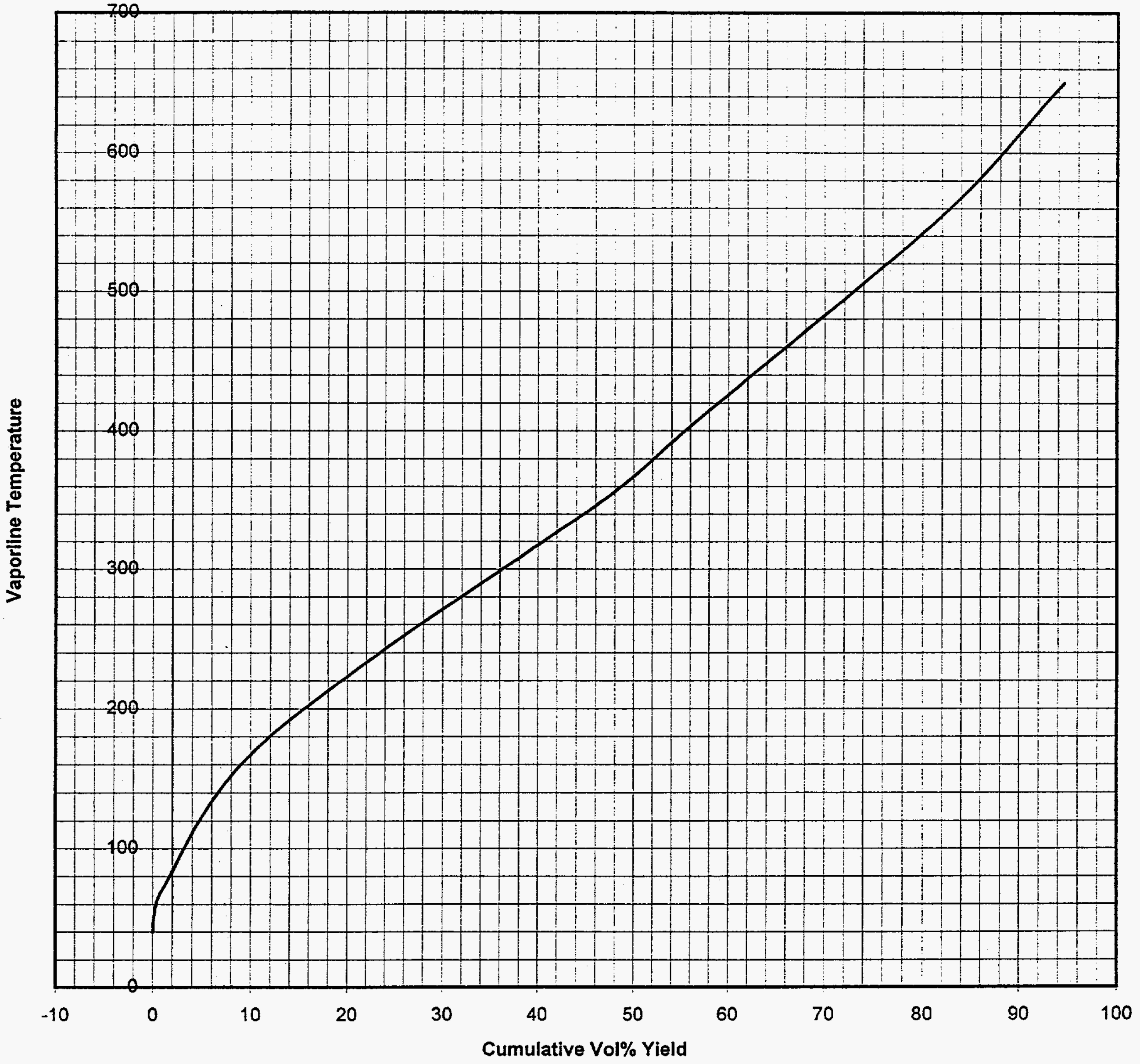
HTI PB-03-6,7,8 (Charge #1)



WinAssay '95
True Boiling Point Curve vs Cumulative Vol% Yield

Sample ID

HTI PB-03-6,7,8 (Charge #1)



ITS- Caleb Brett Distillation Group

ASTM D2892/D5236 CHARGE INFORMATION

Lab ID:	96-005162
Client Name:	Consol Inc.
Sample ID:	HTI PB-03-6,7,8 (Charge #1)
Date:	9/25/96

Operator: Robert Kelly

Charge Mass D2892(g):	9494.0
Charge S.G D2892 (60/60F):	0.8347
Charge Mass D5236(g):	0.0
Charge S.G. D5236 (60/60F):	0.0000

Water Weight Removed (g):	0.0
Initial Vapor Temp:	40
Whole Crude Sulfur Wt%:	

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 5:03 PM
Reported: 09-23-1996 11:06:08
Normalized to 100.00%

Components Listed in Chromatographic Order

Min.	INDEX	Component	Wt%	Vol%	Mol%
8.746	200.0	ethane	0.027	0.046	0.053
9.242	294.1	propylene	0.014	0.016	0.019
9.298	300.0	propane	8.488	9.812	11.350
10.322	366.3	i-butane	5.925	6.153	6.011
10.991	391.0	butene-1	0.091	0.088	0.095
11.293	400.0	n-butane	63.634	63.611	64.556
11.608	411.1	t-butene-2	0.206	0.197	0.216
11.701	414.2	2,2-dimethylpropane	0.091	0.089	0.074
12.121	427.0	c-butene-2	0.123	0.114	0.129
13.491	460.3	3-methylbutene-1	0.007	0.006	0.006
13.726	465.0	?	0.058	0.054	0.049
14.398	477.6	i-pentane	10.862	10.143	8.877
15.156	490.1	pentene-1	0.036	0.032	0.030
15.545	495.9	2-methylbutene-1	0.014	0.013	0.012
15.828	500.0	n-pentane	10.326	9.541	8.439
16.239	509.0	t-pentene-2	0.044	0.040	0.037
16.668	517.9	c-pentene-2	0.018	0.016	0.015
16.940	523.3	2-methylbutene-2	0.013	0.011	0.011
17.781	538.9	2,2-dimethylbutane	0.004	0.003	0.003
18.785	555.8	cyclopentene	0.021	0.016	0.018

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 5:03 PM
Reported: 09-23-1996 11:06:08
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Weight Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.027	0.000	0.000	0.000	0.000	0.027
C3:	8.488	0.000	0.000	0.000	0.014	8.502
C4:	63.634	5.925	0.000	0.000	0.419	69.979
C5:	10.326	10.953	0.000	0.000	0.152	21.431
C6:	0.000	0.004	0.000	0.000	0.000	0.004
C7:	0.000	0.000	0.000	0.000	0.000	0.000
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	82.474	16.882	0.000	0.000	0.585	99.942

Oxygenates: 0.000 Total C14+: 0.000 Total Unknowns: 0.058
Grand Total: 100.000

Molecular Weight and Relative Density Data

Group:	Ave. Mw.:	Ave. Rel. Density:
C1:	0.000	0.000
C2:	30.070	0.340
C3:	44.094	0.501
C4:	58.112	0.577
C5:	72.134	0.623
C6:	86.178	0.649
C7:	0.000	0.000
C8:	0.000	0.000
C9:	0.000	0.000
C10:	0.000	0.000
C11:	0.000	0.000
C12:	0.000	0.000
C13:	0.000	0.000
C14:	0.000	0.000
Total Sample:	58.932	0.578

File: 5174A2.DHA

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 5:03 PM
Reported: 09-23-1996 11:06:08
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Volume Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.046	0.000	0.000	0.000	0.000	0.046
C3:	9.812	0.000	0.000	0.000	0.016	9.828
C4:	63.611	6.153	0.000	0.000	0.400	70.163
C5:	9.541	10.232	0.000	0.000	0.133	19.905
C6:	0.000	0.003	0.000	0.000	0.000	0.003
C7:	0.000	0.000	0.000	0.000	0.000	0.000
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	83.010	16.388	0.000	0.000	0.548	99.946

Oxygenates: 0.000 Total C14+: 0.000 Total Unknowns: 0.054
Grand Total: 100.000

(in Mole Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.053	0.000	0.000	0.000	0.000	0.053
C3:	11.350	0.000	0.000	0.000	0.019	11.369
C4:	64.556	6.011	0.000	0.000	0.441	71.008
C5:	8.439	8.951	0.000	0.000	0.128	17.518
C6:	0.000	0.003	0.000	0.000	0.000	0.003
C7:	0.000	0.000	0.000	0.000	0.000	0.000
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	84.397	14.965	0.000	0.000	0.588	99.951

Oxygenates: 0.000 Total C14+: 0.000 Total Unknowns: 0.049
Grand Total: 100.000

File: 5174A2.DHA

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 5:03 PM
Reported: 09-23-1996 11:06:08
Normalized to 100.00%

Boiling Point Distribution Data

	Wt. Percent Off: deg.C.:	Wt. Percent Off: deg.F.:	Vol. Percent Off: deg.C.:	Vol. Percent Off: deg.F.:
IBP (0.5%)	-42.04	-43.67	-42.04	-43.67
5.0%	-42.04	-43.67	-42.04	-43.67
10.0%	-11.72	10.90	-11.72	10.90
15.0%	-0.50	31.10	-11.72	10.90
20.0%	-0.50	31.10	-0.50	31.10
25.0%	-0.50	31.10	-0.50	31.10
30.0%	-0.50	31.10	-0.50	31.10
35.0%	-0.50	31.10	-0.50	31.10
40.0%	-0.50	31.10	-0.50	31.10
45.0%	-0.50	31.10	-0.50	31.10
50.0%	-0.50	31.10	-0.50	31.10
55.0%	-0.50	31.10	-0.50	31.10
60.0%	-0.50	31.10	-0.50	31.10
65.0%	-0.50	31.10	-0.50	31.10
70.0%	-0.50	31.10	-0.50	31.10
75.0%	-0.50	31.10	-0.50	31.10
80.0%	27.84	82.11	3.72	38.70
85.0%	27.84	82.11	27.84	82.11
90.0%	36.06	96.91	27.84	82.11
95.0%	36.06	96.91	36.06	96.91
FBP (99.5%)	36.06	96.91	36.06	96.91

Research Octane Number =107.33
(Calculated from Individual Component Values)

Contribution to Total by:

Paraffins:	88.70
Iso-paraffins:	17.97
Aromatics:	0.00
Naphthenes:	0.00
Olefins:	0.61
Oxygenates:	0.00

File: 5174A2.DHA

Inchcape Testing Services
Caleb Brètt

WinAssay '95

Version 1.00

Final Reports

Client Name:	**Consol Inc.**
Sample ID:	**HTI PB-03-6,7,8 (Charge #2)**
Laboratory ID:	**96-005162**
Date:	**9/25/96**
Operator:	**Robert Kelly**

Distillation Summary Report

Prepared For: Consol Inc.
Sample ID: HTI PB-03-6,7,8 (Charge #2)
Date: 9/25/96

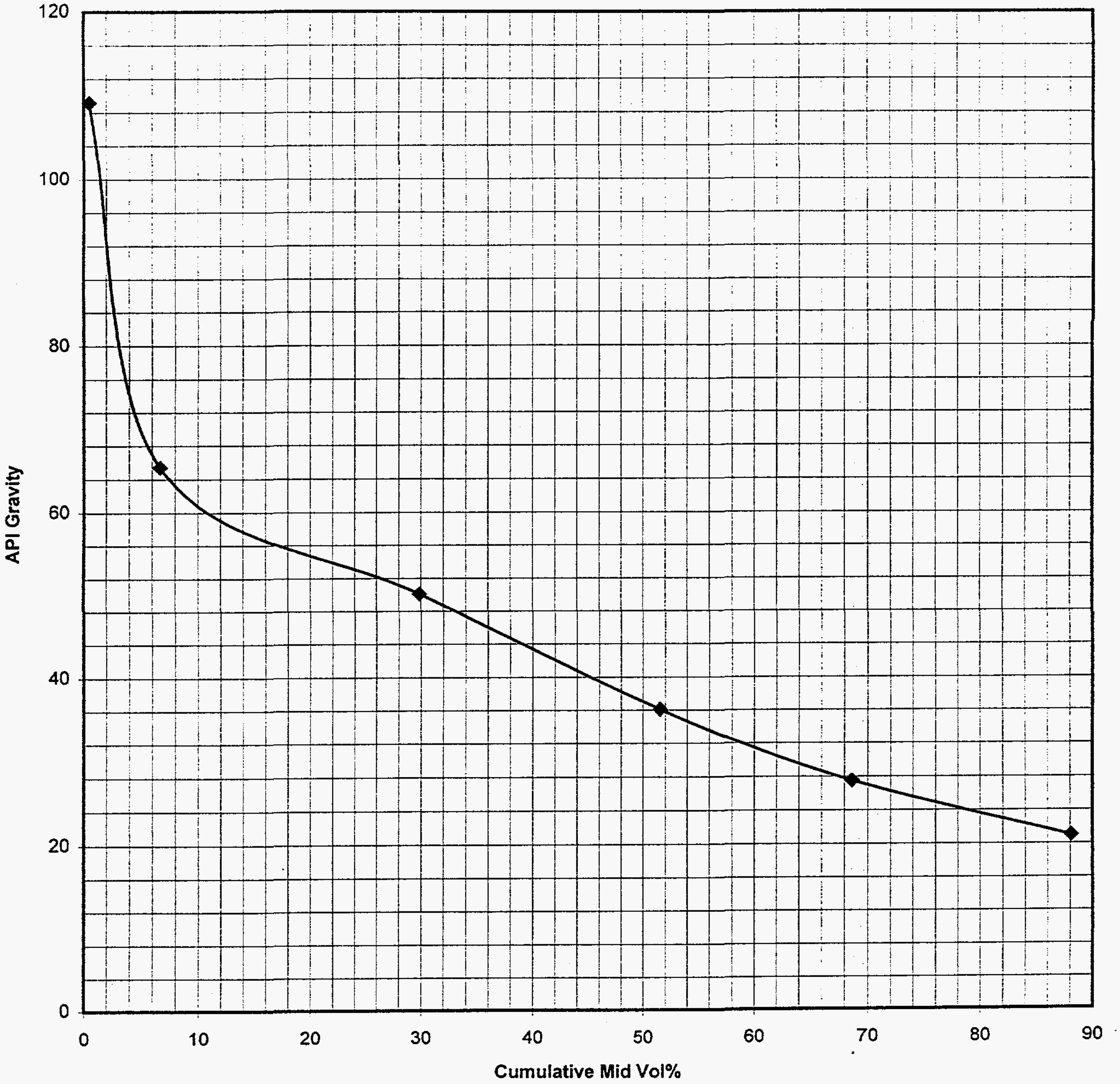
Cut Temp	Degrees F	DUMP	Specific		LIQ	CUM. LIQ	WT%	CUM	API	MID
TO		WT(g)	Gravity	MLS	VOL%	VOL%		WT%	GRAVITY	LIQ VOL%
ASTM D2892 Distillation Yields										
IBP	70	58.30	0.5880	99.15	0.94	0.94	0.66	0.66	109.15	0.47
70	180	874.20	0.7185	1216.70	11.51	12.45	9.91	10.57	65.44	6.70
180	350	2866.60	0.7792	3678.90	34.82	47.27	32.50	43.07	50.10	29.86
350	400	756.20	0.8444	895.55	8.48	55.74	8.57	51.65	36.07	51.51
400	550	2426.70	0.8899	2726.94	25.81	81.55	27.51	79.16	27.51	68.65
550	650	1282.60	0.9280	1382.11	13.08	94.63	14.54	93.70	20.98	88.09
650+		555.40	0.9260	599.78	5.68	100.31	6.30	100.00	21.31	

Loss (Grams): 17.8 (0.20 Wt.%)
Distribution: (2/3) 11.9 g to IBP-70 F
(1/3) 5.9 g to 70-180 F

WinAssay '95 Quality Control Applications

Cum. Mid Vol% v. API Gravity

HTI PB-03-6,7,8 (Charge #2)



WinAssay '95

True Boiling Point Curve

Vaporline Temperature v. Cumulative Wt% Yield

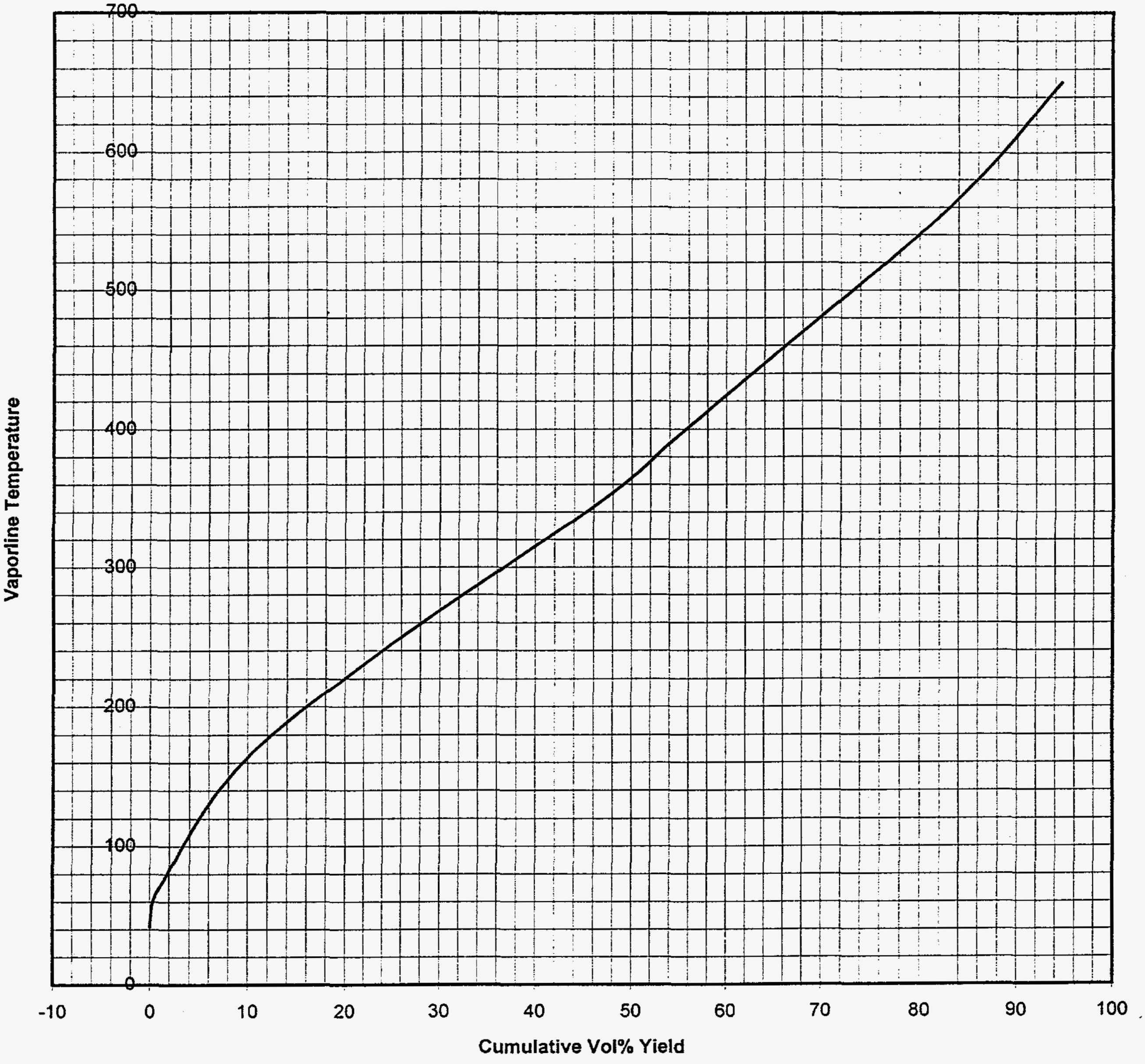
Sample ID

HTI PB-03-6,7,8 (Charge #2)

WinAssay '95
True Boiling Point Curve vs Cumulative Vol% Yield

Sample ID

HTI PB-03-6,7,8 (Charge #2)



ITS- Caleb Brett Distillation Group

ASTM D2892/D5236 CHARGE INFORMATION

Lab ID: 96-005162
Client Name: Consol Inc.
Sample ID: HTI PB-03-6,7,8 (Charge #2)
Date: 9/25/96

Operator: Robert Kelly

Charge Mass D2892(g): 8820.0
Charge S.G D2892 (60/60F): 0.8347

Charge Mass D5236(g): 0.0
Charge S.G. D5236 (60/60F): 0.0000

Water Weight Removed (g): 0.0
Initial Vapor Temp: 42
Whole Crude Sulfur Wt%:

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 6:06 PM
Reported: 09-23-1996 11:09:47
Normalized to 100.00%

Components Listed in Chromatographic Order

Min.	INDEX	Component	Wt%	Vol%	Mol%
9.308	300.0	propane	0.213	0.251	0.295
10.324	366.0	i-butane	6.759	7.137	7.083
10.994	391.0	butene-1	0.108	0.107	0.117
11.293	400.0	n-butane	69.245	70.395	72.569
11.616	411.4	t-butene-2	0.246	0.240	0.267
11.708	414.4	2,2-dimethylpropane	0.111	0.110	0.094
12.125	427.0	c-butene-2	0.149	0.141	0.162
13.497	460.2	3-methylbutene-1	0.006	0.006	0.005
13.740	465.1	?	0.033	0.031	0.029
14.409	477.5	i-pentane	9.621	9.137	8.123
15.161	489.9	pentene-1	0.032	0.029	0.027
15.554	495.8	2-methylbutene-1	0.011	0.010	0.009
15.850	500.0	n-pentane	11.015	10.351	9.300
16.246	508.7	?	0.047	0.044	0.039
16.674	517.6	?	0.019	0.017	0.016
16.945	522.9	2-methylbutene-2	0.014	0.012	0.012
17.787	538.6	?	0.010	0.008	0.008
18.797	555.7	cyclopentene	0.011	0.008	0.009
19.435	565.6	cyclopentane	1.039	0.820	0.902
19.582	567.8	4-methyl-c-pentene-2	0.050	0.044	0.036
19.882	572.2	2-methylpentane	0.422	0.380	0.298
20.776	584.7	3-methylpentane	0.221	0.196	0.156
21.973	600.0	n-hexane	0.361	0.322	0.255
23.884	625.5	methylcyclopentane	0.259	0.203	0.187

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 6:06 PM
Reported: 09-23-1996 11:09:47
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Weight Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.000	0.000	0.000	0.000	0.000	0.000
C3:	0.213	0.000	0.000	0.000	0.000	0.213
C4:	69.245	6.759	0.000	0.000	0.503	76.507
C5:	11.015	9.732	0.000	1.039	0.073	21.859
C6:	0.361	0.643	0.000	0.259	0.050	1.313
C7:	0.000	0.000	0.000	0.000	0.000	0.000
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	80.835	17.134	0.000	1.297	0.626	99.892

Oxygenates: 0.000 Total C14+: 0.000 Total Unknowns: 0.108
Grand Total: 100.000

Molecular Weight and Relative Density Data

Group:	Ave. Mw.:	Ave. Rel. Density:
C1:	0.000	0.000
C2:	0.000	0.000
C3:	44.097	0.501
C4:	58.110	0.577
C5:	72.045	0.628
C6:	85.695	0.675
C7:	0.000	0.000
C8:	0.000	0.000
C9:	0.000	0.000
C10:	0.000	0.000
C11:	0.000	0.000
C12:	0.000	0.000
C13:	0.000	0.000
C14:	0.000	0.000
Total Sample:	60.848	0.588

File: 5164B2.DHA

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 6:06 PM
Reported: 09-23-1996 11:09:47
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Volume Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.000	0.000	0.000	0.000	0.000	0.000
C3:	0.251	0.000	0.000	0.000	0.000	0.251
C4:	70.395	7.137	0.000	0.000	0.488	78.020
C5:	10.351	9.248	0.000	0.820	0.065	20.483
C6:	0.322	0.576	0.000	0.203	0.044	1.145
C7:	0.000	0.000	0.000	0.000	0.000	0.000
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	81.319	16.961	0.000	1.023	0.596	99.899

Oxygenates: 0.000 Total C14+: 0.000 Total Unknowns: 0.101
Grand Total: 100.000

(in Mole Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.000	0.000	0.000	0.000	0.000	0.000
C3:	0.295	0.000	0.000	0.000	0.000	0.295
C4:	72.569	7.083	0.000	0.000	0.546	80.198
C5:	9.300	8.217	0.000	0.902	0.063	18.482
C6:	0.255	0.454	0.000	0.187	0.036	0.933
C7:	0.000	0.000	0.000	0.000	0.000	0.000
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	82.418	15.754	0.000	1.089	0.646	99.908

Oxygenates: 0.000 Total C14+: 0.000 Total Unknowns: 0.092
Grand Total: 100.000

File: 5164B2.DHA

CALEB BRETT HOUSTON

TID: 96-005162-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-6,7,8 CRUDE
OIL/IBP-70 F
NID: 51851 Date: 16-SEP-1996

Analyzed: 9/20/96 6:06 PM
Reported: 09-23-1996 11:09:47
Normalized to 100.00%

Boiling Point Distribution Data

	Wt. Percent Off: deg.C.:	Wt. Percent Off: deg.F.:	Vol. Percent Off: deg.C.:	Vol. Percent Off: deg.F.:
IBP (0.5%)	-11.72	10.90	-11.72	10.90
5.0%	-11.72	10.90	-11.72	10.90
10.0%	-0.50	31.10	-0.50	31.10
15.0%	-0.50	31.10	-0.50	31.10
20.0%	-0.50	31.10	-0.50	31.10
25.0%	-0.50	31.10	-0.50	31.10
30.0%	-0.50	31.10	-0.50	31.10
35.0%	-0.50	31.10	-0.50	31.10
40.0%	-0.50	31.10	-0.50	31.10
45.0%	-0.50	31.10	-0.50	31.10
50.0%	-0.50	31.10	-0.50	31.10
55.0%	-0.50	31.10	-0.50	31.10
60.0%	-0.50	31.10	-0.50	31.10
65.0%	-0.50	31.10	-0.50	31.10
70.0%	-0.50	31.10	-0.50	31.10
75.0%	-0.50	31.10	-0.50	31.10
80.0%	27.84	82.11	27.84	82.11
85.0%	27.84	82.11	27.84	82.11
90.0%	36.06	96.91	36.06	96.91
95.0%	36.06	96.91	36.06	96.91
FBP (99.5%)	68.73	155.71	68.73	155.71

Research Octane Number =105.62
(Calculated from Individual Component Values)

Contribution to Total by:

Paraffins:	85.41
Iso-paraffins:	18.33
Aromatics:	0.00
Naphthenes:	1.15
Olefins:	0.65
Oxygenates:	0.00

File: 5164B2.DHA

Inchcape Testing Services
Caleb Brett

NOV 1 4 1996 R.A.W.

Consol, Inc.
4000 Brownsville Road
Library, PA 15129-9566

11/04/96

Attention: R.A. Winschell

<u>Reference: Crude Assay on "HTI PB-03-9, 10, 11" received in September 1996</u>

Mr. Winschell,

The following results are based on a composite of (2) separate distillations performed on this sample. The Distillation Data Report provides a breakdown of the separate distillations. Should you have any questions concerning this report, you may contact me at (713) 844-3311, or by fax at (713) 844-3330.

Sincerely,

Robert Kelly
Distillation Manager

Inchcape Testing Services
Caleb Brett

November 12, 1996

Houston, Texas

Our Reference : HO/96-005170
Your Reference : PO# 01-001-033668

Consol, Inc.
Research & Development
4000 Brownsville Road
Library, PA 15129-9566

ATTN: R.A. Winschel

Reference: To perform "CRUDE ASSAY" on "HT1 PB-03-9,10,11" received in September 1996.

Dear Mr. Winschel:

Please find enclosed the original report on the above referenced submitted sample(s) and our invoice for services rendered.

Should you have any questions regarding this report, please do not hesitate to contact us at your convenience.

We trust you find all in order and thank you for requesting our services.

Very Truly Yours,

INCHCAPE TESTING SERVICES
CALEB BRETT U.S.A.

John Mowrey
Laboratory Manager

Enclosures:
JM/rm

Inchcape Testing Services
Caleb Brett

9809 Rowlett Road
Houston, TX 77075
Phone: (713) 946-2420
Fax: (713) 946-0545

Your Ref: PO# 01-001-033668

Date: 8-NOV-1996

Laboratory Report No. 96-005170-0-HOUS; 1

Consol, Inc.
4000 Brownsville Road
Library, PA 15129-9566

For the Attention of R.A. Winschel

SAMPLE DETAILS: 8 Sample(s) received on 16-SEP-1996

SOURCE : Consol, Inc.

DESCRIPTION :

	LAB REF
HTI PB-03-9, 10, 11 CRUDE OIL	
Sample As Received	001-00
IBP-70 Deg. F	002-00
70-180 Deg. F	003-00
180-350 Deg. F	004-00
350-400 Deg. F	005-00
400-550 Deg. F	006-00
550-650 Deg. F	007-00
650+ Deg. F	008-00

CONTAINERS : 5 Gallon Can

SEALS : NONE

RESULTS : SEE ATTACHED SHEETS

(TOTAL NUMBER OF PAGES 8)

Approved by: John Mowrey

Laboratory Report No. 96-005170-0-HOUS; 1 - Page 2 of 8

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-001-00	Sample As Received

Test		Method	001-00
API Gravity @ 60/60 F		D4052	24.1
Specific Gravity @ 60/60 F		D4052	0.9091
Carbon	Wt. %	D5291	83.53
Hydrogen	Wt. %	D5291	10.99
Sulfur Content	Wt. %	D4294	0.31
Total Nitrogen	ppm	D4629	6716.0
Methanol	Wt. %	D4815	<0.01
Ethanol	Wt. %	D4815	<0.01
t-Butanol	Wt. %	D4815	<0.01
Iso-Propanol	Wt. %	D4815	<0.01
n-Propanol	Wt. %	D4815	<0.01
Sec-Butanol	Wt. %	D4815	<0.01
Iso-Butanol	Wt. %	D4815	<0.01
n-Butanol	Wt. %	D4815	<0.01
MTBE	Wt. %	D4815	<0.01
ETBE	Wt. %	D4815	<0.01
DIPE	Wt. %	D4815	<0.01
TAME	Wt. %	D4815	<0.01
t-Pentanol	Wt. %	D4815	<0.01
Total Oxygenates	Wt. %	D4815	<0.01
Oxygen	Wt. %	By Difference	4.49
Ash Content for Digestion	Wt. %	D482	0.004
Vanadium	ppm	ICP	<0.1
Nickel	ppm	ICP	<0.1
Iron	ppm	ICP	17.7
Copper	ppm	ICP	0.1
Freezing Point	Deg. F	D2386	Too Dark
Microcarbon Residue	Wt. %	D4530	<0.1
N-Heptane Insolubles	Wt. %	D3279	0.09
Boiling Point Distribution		D5307	See Attached

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-002-00	IBP-70 Deg. F

Test	Method	002-00
API Gravity @ 60/60 F(Charge #1)	G.C.	109.2
API Gravity @ 60/60 F(Charge #2)	G.C.	113.3
Specific Gravity @ 60/60 F (Charge #1)	G.C.	0.5880
Specific Gravity @ 60/60 F (Charge #2)	G.C.	0.5780
DHA	G.C.	See Attached

Laboratory Report No. 96-005170-0-HOUS; 1 - Page 3 of 8

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-003-00	70-180 Deg. F

Test		Method	003-00
API Gravity @ 60/60 F		D4052	65.7
Specific Gravity @ 60/60 F		D4052	0.7174
Carbon	Wt. %	D5291	84.39
Hydrogen	Wt. %	D5291	15.35
Sulfur Content	Wt. %	D4294	0.19
Total Nitrogen	ppm	D4629	3542.0
Vapor Pressure	psi	D323	9.0
Paraffins	Vol. %	G.C.	42.20
Olefins	Vol. %	G.C.	7.23
Naphthenes	Vol. %	G.C.	47.51
Aromatics	Vol. %	G.C.	3.06
Total N & A	Vol. %	G.C.	50.57
Benzene Content	Vol. %	G.C.	2.55
Total Acid Number	mgKOH/g	D974	0.13
Corrosion 3 hrs @ 122 F		D130	4b
Existent Gum	mg/100mL	D381	<1
Oxidation Stability	min.	D525	>240
Research Octane Number		D2699	75.7
Motor Octane Number		D2700	72.2
Initial Boiling Point	Deg. F	D86	107
@ 5% Evaporated	Deg. F		126
@ 10% Evaporated	Deg. F		131
@ 20% Evaporated	Deg. F		137
@ 30% Evaporated	Deg. F		142
@ 40% Evaporated	Deg. F		147
@ 50% Evaporated	Deg. F		151
@ 60% Evaporated	Deg. F		155
@ 70% Evaporated	Deg. F		160
@ 80% Evaporated	Deg. F		164
@ 90% Evaporated	Deg. F		168
@ 95% Evaporated	Deg. F		174
Final Boiling Point	Deg. F		189
Recovery	Vol. %		99.5
Residue	Vol. %		0.2
Loss	Vol. %		0.3

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-004-00	180-350 Deg. F

Test		Method	004-00
API Gravity @ 60/60 F		D4052	43.7
Specific Gravity @ 60/60 F		D4052	0.8076
Carbon	Wt. %	D5291	80.45
Hydrogen	Wt. %	D5291	12.74
Sulfur Content	Wt. %	D4294	0.21
Total Nitrogen	ppm	D4629	2005.0
Mercaptan Sulfur Content	ppm	UOP163	669
Vapor Pressure	psi	D323	1.7
Paraffins	Vol. %	G.C.	22.15
Olefins	Vol. %	G.C.	5.01
Naphthenes	Vol. %	G.C.	55.26
Aromatics	Vol. %	G.C.	17.58
Total N & A	Vol. %	G.C.	72.84
Corrosion 3 hrs @ 122 F		D130	2d
Existent Gum	mg/100mL	D381	50
Oxidation Stability	min.	D525	>240
Research Octane Number		D2699	65.6
Motor Octane Number		D2700	66.5
Initial Boiling Point	Deg. F	D86	176
@ 5% Evaporated	Deg. F		184
@ 10% Evaporated	Deg. F		234
@ 20% Evaporated	Deg. F		243
@ 30% Evaporated	Deg. F		251
@ 40% Evaporated	Deg. F		260
@ 50% Evaporated	Deg. F		271
@ 60% Evaporated	Deg. F		284
@ 70% Evaporated	Deg. F		298
@ 80% Evaporated	Deg. F		311
@ 90% Evaporated	Deg. F		328
@ 95% Evaporated	Deg. F		341
Final Boiling Point	Deg. F		352
Recovery	Vol. %		96.6
Residue	Vol. %		0.6
Loss	Vol. %		1.1
Total Acid Number	mgKOH/g	D664	0.14

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-005-00	350-400 Deg. F

Test		Method	005-00
API Gravity @ 60/60 F		D4052	21.2
Specific Gravity @ 60/60 F		D4052	0.9268
Carbon	Wt. %	D5291	81.77
Hydrogen	Wt. %	D5291	10.54
Sulfur Content	Wt. %	D4294	0.17
Total Nitrogen	ppm	D4629	5897.0
Mercaptan Sulfur Content	ppm	UOP163	597
Viscosity @ -20 C	cSt	D445	12.42
Viscosity @ 100 C	cSt	D445	0.750
Freezing Point	Deg. F	D2386	-56.0
Pour Point	Deg. F	D97	<-76.0
Flash Point, TCC	Deg. F	D56	150
Vapor Pressure	psi	D323	0.2
Luminometer Number		D1740	33
Smoke Point	mm	D1322	13
Paraffins	Vol. %	G.C.	21.46
Olefins	Vol. %	G.C.	9.20
Naphthenes	Vol. %	G.C.	11.83
Aromatics	Vol. %	G.C.	57.51
Total N & A	Vol. %	G.C.	69.34
Naphthalenes	Vol. %	D1840	6.34
Corrosion 3 hrs @ 122 F		D130	3a
Existent Gum	mg/100mL	D381	63
Tube Rating		D3241	4.0
Pressure Drop	mmHg	D3241	>125.0
Oxidation Stability	min.	D525	>240
Research Octane Number		D2699	106.6
Motor Octane Number		D2700	91.2
Cetane Number		D613	<18.3
Initial Boiling Point	Deg. F	D86	351
@ 5% Evaporated	Deg. F		360
@ 10% Evaporated	Deg. F		361
@ 20% Evaporated	Deg. F		365
@ 30% Evaporated	Deg. F		367
@ 40% Evaporated	Deg. F		369
@ 50% Evaporated	Deg. F		370
@ 60% Evaporated	Deg. F		372
@ 70% Evaporated	Deg. F		376
@ 80% Evaporated	Deg. F		378
@ 90% Evaporated	Deg. F		383
@ 95% Evaporated	Deg. F		388
Final Boiling Point	Deg. F		403
Recovery	Vol. %		99.0
Residue	Vol. %		0.5
Loss	Vol. %		0.5
Net Heat of Combustion	BTU/lb	D1405	18158
Total Acid Number	mgKOH/g	D664	0.70

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-006-00	400-550 Deg. F

Test		Method	006-00
API Gravity @ 60/60 F		D4052	16.8
Specific Gravity @ 60/60 F		D4052	0.9540
Carbon	Wt. %	D5291	83.85
Hydrogen	Wt. %	D5291	10.15
Sulfur Content	Wt. %	D4294	0.12
Total Nitrogen	ppm	D4629	8159.0
Basic Nitrogen	ppm	UOP269	5972
Mercaptan Sulfur Content	ppm	UOP163	126
Viscosity @ -20 C	cSt	D445	73.47
Viscosity @ 40 C	cSt	D445	4.216
Viscosity @ 100 C	cSt	D445	1.199
Freezing Point	Deg. F	D2386	-19.0
Pour Point	Deg. F	D97	-33.0
Aniline Point	Deg. F	D611	18.5
Flash Point, TCC	Deg. F	D56	>200
Luminometer Number		D1740	22
Smoke Point	mm	D1322	9
Paraffins	Vol. %	G.C.	13.95
Olefins	Vol. %	G.C.	4.40
Naphthenes	Vol. %	G.C.	20.97
Aromatics	Vol. %	G.C.	60.68
Total N & A	Vol. %	G.C.	81.65
Naphthalenes	Vol. %	D1840	14.92
Corrosion 3 hrs @ 122 F		D130	1a
Existent Gum	mg/100mL	D381	40
Tube Rating		D3241	4.0
Pressure Drop	mmHg	D3241	<1.0
Cetane Number		D613	<18.3
Initial Boiling Point	Deg. F	D86	412
@ 5% Evaporated	Deg. F		433
@ 10% Evaporated	Deg. F		439
@ 20% Evaporated	Deg. F		444
@ 30% Evaporated	Deg. F		450
@ 40% Evaporated	Deg. F		457
@ 50% Evaporated	Deg. F		462
@ 60% Evaporated	Deg. F		471
@ 70% Evaporated	Deg. F		480
@ 80% Evaporated	Deg. F		484
@ 90% Evaporated	Deg. F		504
@ 95% Evaporated	Deg. F		514
Final Boiling Point	Deg. F		532
Recovery	Vol. %		99.0
Residue	Vol. %		0.5
Loss	Vol. %		0.5
Net Heat of Combustion	BTU/lb	D1405	18190
Total Acid Number	mgKOH/g	D974	0.74

Laboratory Report No. 96-005170-0-HOUS; 1 - Page 7 of 8

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-007-00	550-650 Deg. F

Test		Method	007-00
API Gravity @ 60/60 F		D4052	12.9
Specific Gravity @ 60/60 F		D4052	0.9800
Carbon	Wt. %	D5291	88.72
Hydrogen	Wt. %	D5291	10.58
Sulfur Content	Wt. %	D4294	0.25
Total Nitrogen	ppm	D4629	7197.0
Basic Nitrogen	ppm	UOP269	4547
Viscosity @ 40 C	cSt	D445	11.12
Viscosity @ 100 C	cSt	D445	7.456
Pour Point	Deg. F	D97	5.0
Aniline Point	Deg. F	D611	48.0
Flash Point (Method A)	Deg. F	D93	>200
Paraffins	Vol. %	G.C.	15.57
Olefins	Vol. %	G.C.	4.40
Naphthenes	Vol. %	G.C.	5.59
Aromatics	Vol. %	G.C.	74.44
Total N & A	Vol. %	G.C.	80.03
Bromine Number		D1159	29
Corrosion 3 hrs @ 122 F		D130	1a
Cetane Number		D613	<18.3
Simulated Distillation		D2887	See Attached

Laboratory Report No. 96-005170-0-HOUS; 1 - Page 8 of 8

Sample ID	Description
	HTI PB-03-9, 10, 11 CRUDE OIL
96-005170-0-HOUS-008-00	650+ Deg. F

Test		Method	008-00
API Gravity @ 60/60 F		D4052	6.4
Specific Gravity @ 60/60 F		D4052	1.0261
Carbon	Wt. %	D5291	88.13
Hydrogen	Wt. %	D5291	9.44
Sulfur Content	Wt. %	D4294	0.62
Total Nitrogen	ppm	D4629	8584.0
Basic Nitrogen	ppm	UOP269	4768
Viscosity @ 40 C	cSt	D445	145.5
Viscosity @ 100 C	cSt	D445	7.456
Pour Point	Deg. F	D97	59.0
Aniline Point	Deg. F	D611	113.0
Flash Point (Method A)	Deg. F	D93	>200
Microcarbon Residue	Wt. %	D4530	<0.1
Corrosion 3 hrs @ 122 F		D130	1a
Cetane Number		D613	N/A
Initial Boiling Point	Deg. F	D1160	656
@ 5% Recovery	Deg. F		677
@ 10% Recovery	Deg. F		678
@ 20% Recovery	Deg. F		692
@ 30% Recovery	Deg. F		700
@ 40% Recovery	Deg. F		709
@ 50% Recovery	Deg. F		724
@ 60% Recovery	Deg. F		743
@ 70% Recovery	Deg. F		773
@ 80% Recovery	Deg. F		871
@ 90% Recovery	Deg. F		880
Final Boiling Point	Deg. F		880
Recovery	Vol. %		84.0
Residue + Loss	Vol. %		16.0

SAMPLE: 96-5170-7
FILE: c:\tc4\sd6890\5170-7
PARAMETER: 2887

NOV 11, 1996 - 18:24:30
SIMDIS EXPERT V5.0
Page 3

Boiling Point Distribution

ASTM D-2887

%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)
IBP	519.1	270.6	**40**	603.5	317.5	**80**	648.5	342.5
1	538.5	281.4	**41**	604.5	318.1	**81**	649.8	343.2
2	550.2	287.9	**42**	605.6	318.7	**82**	651.1	343.9
3	555.9	291.1	**43**	606.6	319.2	**83**	652.2	344.6
4	559.7	293.2	**44**	607.7	319.8	**84**	653.3	345.2
5	562.0	294.4	**45**	608.7	320.4	**85**	654.3	345.7
6	563.6	295.3	**46**	609.8	321.0	**86**	655.4	346.3
7	565.1	296.2	**47**	610.8	321.6	**87**	656.4	346.9
8	566.5	296.9	**48**	611.9	322.2	**88**	657.4	347.4
9	567.8	297.7	**49**	612.9	322.7	**89**	658.7	348.2
10	569.2	298.4	**50**	614.0	323.3	**90**	660.2	349.0
11	570.6	299.2	**51**	615.0	323.9	**91**	661.9	349.9
12	572.1	300.1	**52**	616.1	324.5	**92**	663.6	350.9
13	573.6	300.9	**53**	617.2	325.1	**93**	665.5	351.9
14	575.1	301.7	**54**	618.3	325.7	**94**	667.8	353.2
15	576.5	302.5	**55**	619.4	326.3	**95**	669.9	354.4
16	577.8	303.2	**56**	620.5	326.9	**96**	672.3	355.7
17	578.9	303.8	**57**	621.5	327.5	**97**	675.6	357.6
18	579.9	304.4	**58**	622.6	328.1	**98**	677.6	358.7
19	580.9	304.9	**59**	623.6	328.7	**99**	685.4	363.0
20	581.9	305.5	**60**	624.7	329.3	**FBP**	701.8	372.1
21	582.9	306.1	**61**	625.7	329.8			
22	584.0	306.7	**62**	626.8	330.4			
23	585.2	307.3	**63**	628.0	331.1			
24	586.4	308.0	**64**	629.1	331.7			
25	587.7	308.7	**65**	630.2	332.3			
26	588.9	309.4	**66**	631.3	332.9			
27	590.1	310.1	**67**	632.3	333.5			
28	591.2	310.7	**68**	633.4	334.1			
29	592.3	311.3	**69**	634.5	334.7			
30	593.4	311.9	**70**	635.8	335.4			
31	594.4	312.4	**71**	637.1	336.2			
32	595.4	313.0	**72**	638.5	336.9			
33	596.4	313.6	**73**	639.9	337.7			
34	597.4	314.1	**74**	641.2	338.4			
35	598.4	314.7	**75**	642.4	339.1			
36	599.4	315.2	**76**	643.6	339.8			
37	600.4	315.8	**77**	644.8	340.4			
38	601.4	316.3	**78**	646.0	341.1			
39	602.4	316.9	**79**	647.2	341.8			

Start Time: 0.7 minutes
End Time: 17.8 minutes
Area: 272058272.0
Slice Width: 0.80 sec
Baseline Subtracted: c:\tc4\sd6890\1111b

Sample Offset: 8638.0
Baseline Offset: 8792.0
Calibration File: 1111rt
Calibration Date: 11/11/96

SAMPLE: 96-5170-1
FILE: C:\TC4\SD6890\5170-1
PARAMETER: 2887

OCT 14, 1996 - 10:58:41
SIMDIS EXPERT V5.0

Boiling Point Distribution

ASTM D-2887

%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)	%Off	BP(F)	BP(C)
IBP	157.4	69.7	40	421.3	216.3	80	610.5	321.4
1	166.0	74.4	41	427.9	219.9	81	617.1	325.1
2	179.7	82.1	42	433.5	223.1	82	623.2	328.4
3	185.9	85.5	43	438.7	225.9	83	629.6	332.0
4	191.2	88.4	44	444.1	228.9	84	635.6	335.3
5	198.9	92.7	45	446.8	230.4	85	642.8	339.3
6	209.7	98.7	46	451.6	233.1	86	649.1	342.8
7	215.5	101.9	47	456.2	235.7	87	657.8	347.7
8	219.2	104.0	48	459.1	237.3	88	666.3	352.4
9	222.4	105.8	49	462.7	239.3	89	673.1	356.2
10	236.4	113.6	50	466.8	241.6	90	683.1	361.7
11	246.7	119.3	51	470.9	243.8	91	691.6	366.4
12	256.1	124.5	52	474.7	245.9	92	702.0	372.2
13	264.7	129.3	53	479.8	248.8	93	713.6	378.7
14	269.7	132.1	54	483.8	251.0	94	725.2	385.1
15	280.2	137.9	55	487.8	253.2	95	739.1	392.8
16	291.2	144.0	56	492.4	255.8	96	754.6	401.4
17	300.4	149.1	57	496.6	258.1	97	775.2	412.9
18	309.6	154.2	58	500.9	260.5	98	804.4	429.1
19	316.9	158.3	59	505.8	263.2	99	854.2	456.8
20	324.0	162.2	60	511.1	266.2	FBP	905.9	485.5
21	326.1	163.4	61	514.9	268.3			
22	330.4	165.8	62	519.4	270.8			
23	337.3	169.6	63	525.2	274.0			
24	343.7	173.2	64	530.9	277.2			
25	353.3	178.5	65	536.6	280.3			
26	358.6	181.4	66	542.7	283.7			
27	363.2	184.0	67	547.5	286.4			
28	367.3	186.3	68	551.3	288.5			
29	369.5	187.5	69	554.5	290.3			
30	372.9	189.4	70	559.3	292.9			
31	379.4	193.0	71	564.8	296.0			
32	383.5	195.3	72	570.6	299.2			
33	393.1	200.6	73	574.5	301.4			
34	400.0	204.4	74	580.2	304.6			
35	403.3	206.3	75	584.1	306.7			
36	406.1	207.8	76	589.3	309.6			
37	408.2	209.0	77	594.9	312.7			
38	413.1	211.7	78	598.9	314.9			
39	417.8	214.3	79	604.4	318.0			

Start Time: 0.2 minutes
End Time: 26.0 minutes
Area: 39163064.0
Slice Width: 0.80 sec
Baseline Subtracted: C:\TC4\SD6890\1013B

Sample Offset: 7366.0
Baseline Offset: 7234.0
Calibration File: 1013rt
Calibration Date: 10/13/96

Inchcape Testing Services
Caleb Brett

WinAssay '95
Version 1.00

Final Reports

Client Name:	Consol Inc.
Sample ID:	HTI PB-03-9,10,11 (Charge #1)
Laboratory ID:	96-005170
Date:	10/01/96
Operator:	Robert Kelly

Distillation Summary Report

Prepared For: Consol Inc.
Sample ID: HTI PB-03-9,10,11 (Charge #1)
Date: 10/01/96

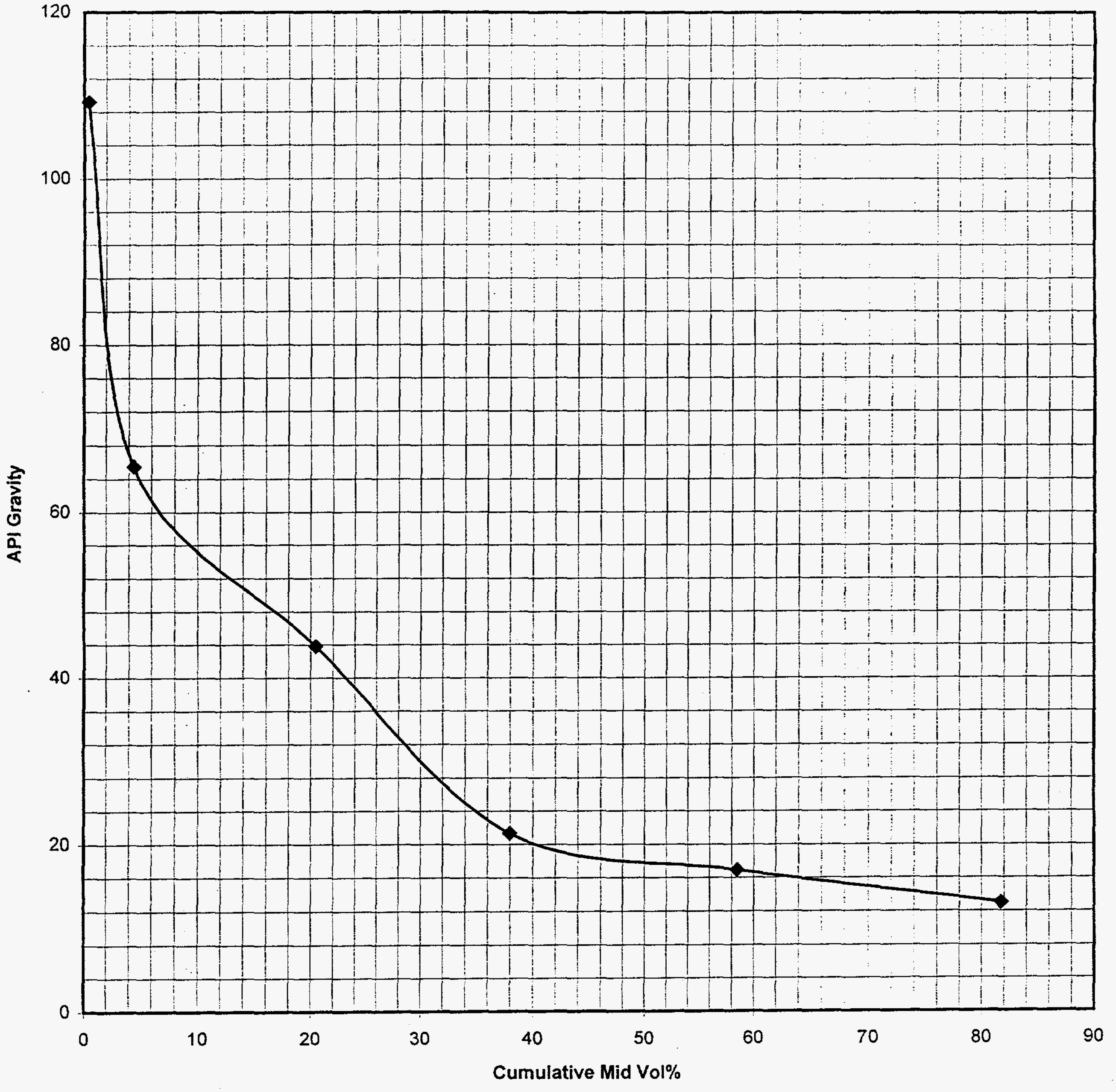
Cut Temp	Degrees F	DUMP WT(g)	Specific Gravity	MLS	LIQ VOL%	CUM. LIQ VOL%	WT%	CUM WT%	API GRAVITY	MID LIQ VOL%
TO										
ASTM D2892 Distillation Yields										
IBP	70	45.50	0.5880	77.38	0.81	0.81	0.53	0.53	109.15	0.41
70	180	490.20	0.7183	682.44	7.18	7.99	5.67	6.20	65.49	4.40
180	350	1910.40	0.8071	2366.99	24.90	32.90	22.11	28.31	43.82	20.45
350	400	894.10	0.9258	965.76	10.16	43.06	10.35	38.66	21.34	37.98
400	550	2797.10	0.9534	2933.82	30.87	73.92	32.37	71.03	16.92	58.49
550	650	1448.00	0.9793	1478.61	15.56	89.48	16.76	87.79	12.99	81.70
650+		1054.60	1.0246	1029.28	10.83	100.31	12.21	100.00	6.60	

Loss (Grams): 9.2 (0.11 Wt.%)
Distribution: (2/3) 6.1 g to IBP-70 F
(1/3) 3.1 g to 70-180 F

WinAssay '95 Quality Control Applications

Cum. Mid Vol% v. API Gravity

HTI PB-03-9,10,11 (Charge #1)

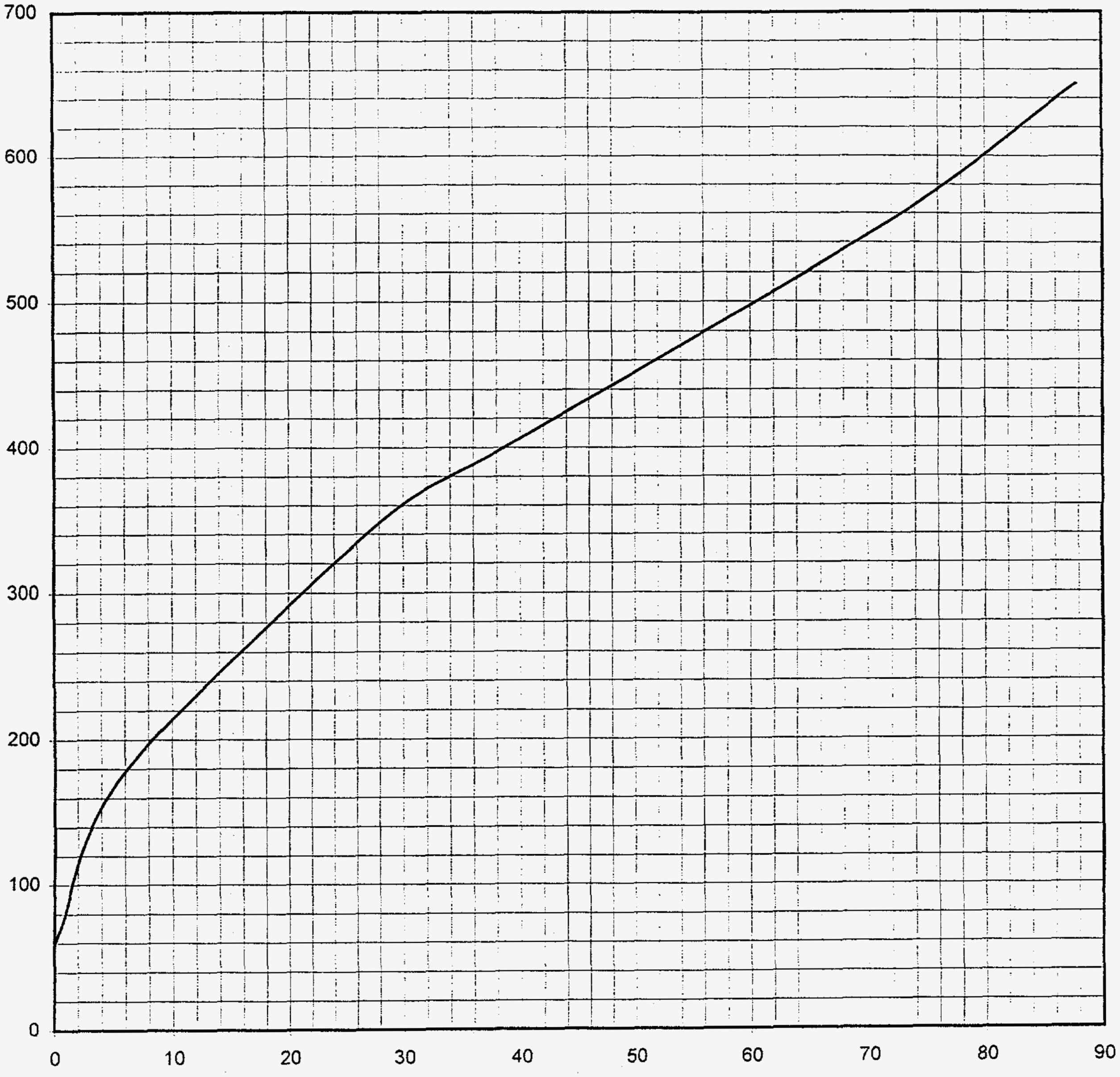


API CURVE Chart 1

WinAssay '95
True Boiling Point Curve
Vaporline Temperature v. Cumulative Wt% Yield

Sample ID

HTI PB-03-9,10,11 (Charge #1)

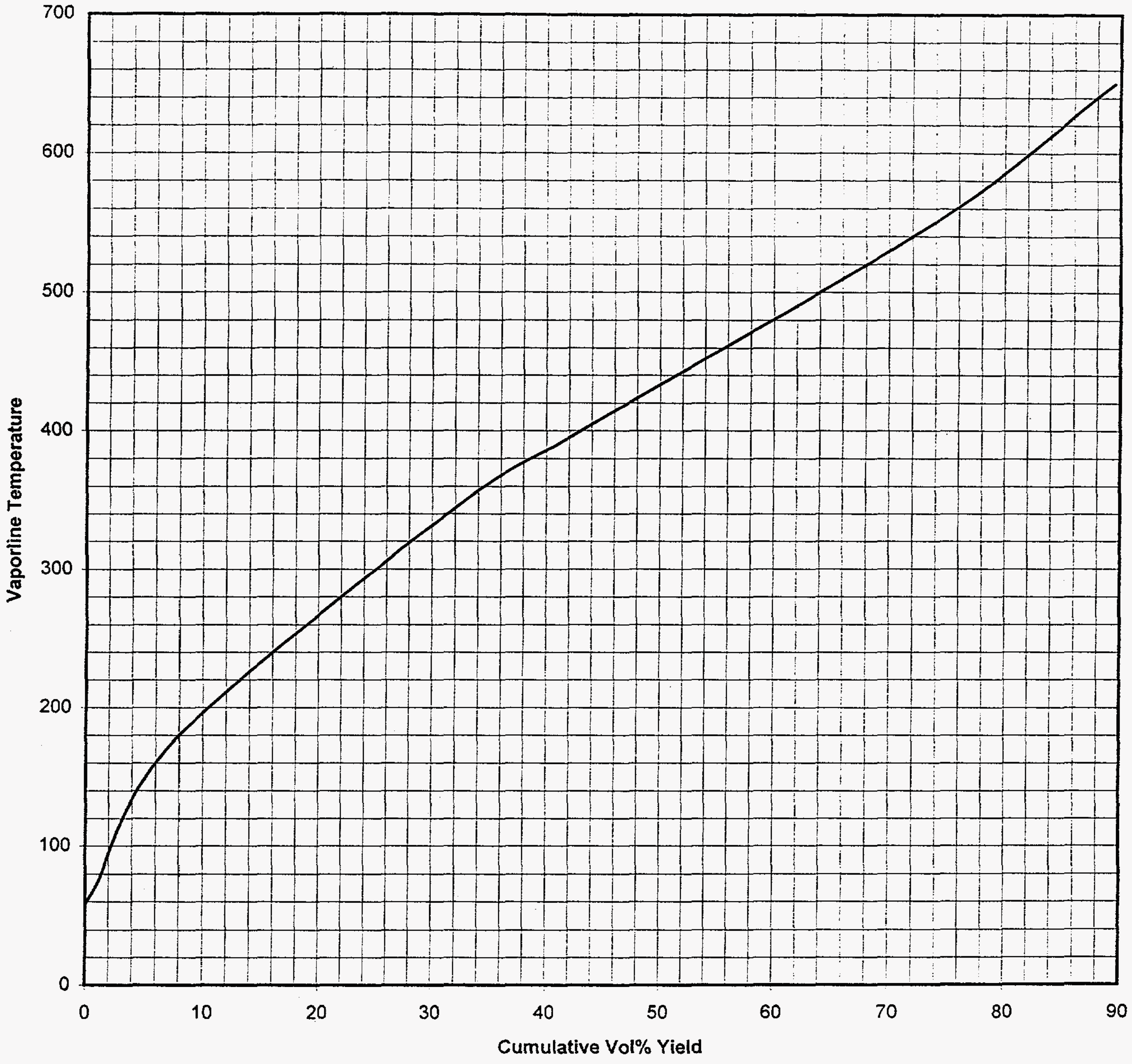


Wt Curve Chart 1

WinAssay '95
True Boiling Point Curve vs Cumulative Vol% Yield

Sample ID

HTI PB-03-9,10,11 (Charge #1)



TBP CURVE Chart 1

ASTM D2892/D5236 CHARGE INFORMATION

Lab ID: 96-005170

Client Name: Consol Inc.

Sample ID: HTI PB-03-9,10,11 (Charge #1)

Date: 10/01/96

Operator: Robert Kelly

Charge Mass D2892(g): 8702.0

Charge S.G D2892 (60/60F): 0.9090

Charge Mass D5236(g): 0.0

Charge S.G. D5236 (60/60F): 0.0000

Water Weight Removed (g): 62.1

Initial Vapor Temp: 58

Whole Crude Sulfur Wt%:

CALEB BRETT HOUSTON

TID: 96-005170-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-9,10,11 CRUDE
OIL/IBP-70 F
NID: 51870 Date: 16-SEP-1996

Analyzed: 10/2/96 8:25 AM
Reported: 10-03-1996 13:00:05
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Weight Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.009	0.000	0.000	0.000	0.000	0.009
C3:	2.051	0.000	0.000	0.000	0.052	2.103
C4:	62.570	7.306	0.000	0.000	5.875	75.751
C5:	8.407	7.611	0.000	1.239	1.541	18.797
C6:	0.727	0.837	0.041	1.101	0.207	2.914
C7:	0.021	0.037	0.000	0.110	0.027	0.196
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	73.784	15.791	0.041	2.450	7.702	99.769

Oxygenates: 0.017 Total C14+: 0.000 Total Unknowns: 0.214
Grand Total: 100.000

Molecular Weight and Relative Density Data

Group:	Ave. Mw.:	Ave. Rel. Density:
C1:	0.000	0.000
C2:	30.070	0.340
C3:	44.045	0.501
C4:	57.962	0.579
C5:	71.833	0.632
C6:	85.077	0.700
C7:	98.782	0.731
C8:	0.000	0.000
C9:	0.000	0.000
C10:	0.000	0.000
C11:	0.000	0.000
C12:	0.000	0.000
C13:	0.000	0.000
C14:	0.000	0.000
Total Sample:	60.242	0.588

File: 5170S1.DHA

CALEB BRETT HOUSTON

TID: 96-005170-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-9,10,11 CRUDE
OIL/IBP-70 F
NID: 51870 Date: 16-SEP-1996

Analyzed: 10/2/96 8:25 AM
Reported: 10-03-1996 13:00:05
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Volume Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.015	0.000	0.000	0.000	0.000	0.015
C3:	2.416	0.000	0.000	0.000	0.060	2.476
C4:	63.715	7.728	0.000	0.000	5.721	77.164
C5:	7.912	7.242	0.000	0.979	1.389	17.523
C6:	0.650	0.751	0.028	0.853	0.173	2.454
C7:	0.018	0.032	0.000	0.085	0.022	0.158
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	74.726	15.753	0.028	1.917	7.365	99.789

Oxygenates: 0.012 Total C14+: 0.000 Total Unknowns: 0.199
Grand Total: 100.000

(in Mole Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.017	0.000	0.000	0.000	0.000	0.017
C3:	2.809	0.000	0.000	0.000	0.074	2.883
C4:	64.999	7.589	0.000	0.000	6.323	78.912
C5:	7.035	6.369	0.000	1.066	1.329	15.800
C6:	0.509	0.587	0.032	0.790	0.150	2.068
C7:	0.013	0.022	0.000	0.068	0.017	0.120
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	75.383	14.568	0.032	1.924	7.893	99.800

Oxygenates: 0.017 Total C14+: 0.000 Total Unknowns: 0.184
Grand Total: 100.000

File: 5170S1.DHA

CALEB BRETT HOUSTON

TID: 96-005170-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-9,10,11 CRUDE
OIL/IBP-70 F
NID: 51870 Date: 16-SEP-1996

Analyzed: 10/2/96 8:25 AM
Reported: 10-03-1996 13:00:05
Normalized to 100.00%

Boiling Point Distribution Data

	Wt. Percent Off:		Vol. Percent Off:	
	deg.C.:	deg.F.:	deg.C.:	deg.F.:
IBP (0.5%)	-42.04	-43.67	-42.04	-43.67
5.0%	-11.72	10.90	-11.72	10.90
10.0%	-6.25	20.75	-11.72	10.90
15.0%	-0.50	31.10	-0.50	31.10
20.0%	-0.50	31.10	-0.50	31.10
25.0%	-0.50	31.10	-0.50	31.10
30.0%	-0.50	31.10	-0.50	31.10
35.0%	-0.50	31.10	-0.50	31.10
40.0%	-0.50	31.10	-0.50	31.10
45.0%	-0.50	31.10	-0.50	31.10
50.0%	-0.50	31.10	-0.50	31.10
55.0%	-0.50	31.10	-0.50	31.10
60.0%	-0.50	31.10	-0.50	31.10
65.0%	-0.50	31.10	-0.50	31.10
70.0%	-0.50	31.10	-0.50	31.10
75.0%	0.88	33.58	-0.50	31.10
80.0%	27.84	82.11	20.05	68.09
85.0%	27.84	82.11	27.84	82.11
90.0%	36.06	96.91	36.06	96.91
95.0%	36.34	97.41	36.06	96.91
FBP (99.5%)	80.72	177.30	80.72	177.30

Research Octane Number =106.27
(Calculated from Individual Component Values)

Contribution to Total by:

Paraffins:	78.57
Iso-paraffins:	17.09
Aromatics:	0.04
Naphthenes:	2.14
Olefins:	8.20
Oxygenates:	0.02

File: 5170S1.DHA

CALEB BRETT HOUSTON

TID: 96-005170-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-9,10,11 CRUDE
OIL/IBP-70 F
NID: 51870 Date: 16-SEP-1996

Analyzed: 10/2/96 8:25 AM
Reported: 10-03-1996 13:00:05
Normalized to 100.00%

Components Listed in Chromatographic Order

Min.	INDEX	Component	Wt%	Vol%	Mol%
8.734	200.0	ethane	0.009	0.015	0.017
9.230	293.9	propylene	0.052	0.060	0.074
9.289	300.0	propane	2.051	2.416	2.809
10.309	366.4	i-butane	7.306	7.728	7.589
10.973	391.1	butene-1	2.383	2.360	2.564
11.271	400.0	n-butane	62.570	63.715	64.999
11.596	411.5	t-butene-2	1.795	1.751	1.932
11.689	414.5	2,2-dimethylpropane	0.050	0.050	0.042
11.959	422.9	?	0.002	0.002	0.002
12.105	427.2	c-butene-2	1.698	1.610	1.827
13.472	460.3	3-methylbutene-1	0.174	0.164	0.150
13.695	464.8	?	0.169	0.159	0.145
14.380	477.5	i-pentane	7.561	7.192	6.328
14.906	486.3	?	0.000	0.000	0.000
15.139	490.0	pentene-1	0.392	0.360	0.337
15.380	493.7	i-propanol	0.017	0.012	0.017
15.528	495.9	2-methylbutene-1	0.147	0.133	0.127
15.818	500.0	n-pentane	8.407	7.912	7.035
16.225	508.9	t-pentene-2	0.365	0.332	0.315
16.652	517.7	c-pentene-2	0.190	0.171	0.163
16.923	523.1	2-methylbutene-2	0.160	0.142	0.138
17.765	538.7	?	0.010	0.009	0.008
18.684	554.2	?	0.030	0.027	0.026
18.774	555.7	cyclopentene	0.113	0.086	0.100
19.056	560.1	4-methylpentene-1	0.009	0.008	0.007
19.420	565.7	cyclopentane	1.239	0.979	1.066
19.562	567.8	4-methyl-c-pentene-2	0.049	0.043	0.035
19.865	572.2	2-methylpentane	0.542	0.489	0.380
19.977	573.8	4-methyl-t-pentene-2	0.010	0.009	0.007
20.608	582.6	1,5-hexadiene	0.030	0.026	0.022
20.758	584.6	3-methylpentane	0.296	0.262	0.207
21.122	589.4	2-methylpentene-1	0.004	0.003	0.003
21.596	595.5	O11	0.002	0.002	0.001
21.661	596.3	?	0.000	0.000	0.000
21.963	600.0	n-hexane	0.727	0.650	0.509
22.135	602.5	t-hexene-3	0.006	0.006	0.005
22.316	605.0	t-hexene-2	0.019	0.016	0.014
22.483	607.3	2-methylpentene-2	0.007	0.006	0.005
22.687	610.1	3-methylcyclopentene	0.006	0.004	0.004
22.789	611.5	O13	0.007	0.006	0.005
22.980	614.1	c-hexene-2	0.010	0.008	0.007
23.444	620.2	3,3-dimethylpentene-1	0.007	0.006	0.004
23.707	623.5	2,2-dimethylpentane	0.001	0.001	0.001

Components Listed in Chromatographic Order

Min.	INDEX	Component	Wt%	Vol%	Mol%
23.872	625.6	methylcyclopentane	0.620	0.488	0.445
24.211	629.8	2,3,3-trimethylbutene-1	0.005	0.004	0.003
25.374	643.6	?	0.002	0.001	0.001
25.776	648.2	1-methylcyclopentene	0.016	0.012	0.012
25.909	649.6	benzene	0.041	0.028	0.032
26.652	657.7	cyclohexane	0.482	0.365	0.346
27.178	663.2	2-ethyl-3-methylbutene-1	0.001	0.001	0.000
27.537	666.9	2-methylhexane	0.015	0.013	0.009
27.683	668.4	2,3-dimethylpentane	0.005	0.004	0.003
27.950	671.0	1,1-dimethylcyclopentane	0.003	0.002	0.002
28.212	673.6	cyclohexene	0.032	0.023	0.023
28.397	675.4	3-methylhexane	0.016	0.014	0.010
29.050	681.6	1c,3-dimethylcyclopentane	0.012	0.010	0.007
29.348	684.4	1t,3-dimethylcyclopentane	0.010	0.008	0.006
29.477	685.6	?	0.002	0.001	0.001
29.642	687.1	1t,2-dimethylcyclopentane	0.018	0.014	0.011
31.101	700.0	n-heptane	0.021	0.018	0.013
33.631	718.3	methylcyclohexane	0.059	0.045	0.036
34.591	724.7	O35	0.015	0.012	0.009
35.287	729.2	ethylcyclopentane	0.008	0.006	0.005

WinAssay '95

Version 1.00

Final Reports

Client Name:	**Consol Inc.**
Sample ID:	**HTI PB-03-9,10,11 (Charge #2)**
Laboratory ID:	**96-005170**
Date:	**10/01/96**
Operator:	**Robert Kelly**

Distillation Summary Report

Prepared For: Consol Inc.
Sample ID: HTI PB-03-9,10,11 (Charge #2)
Date: 10/01/96

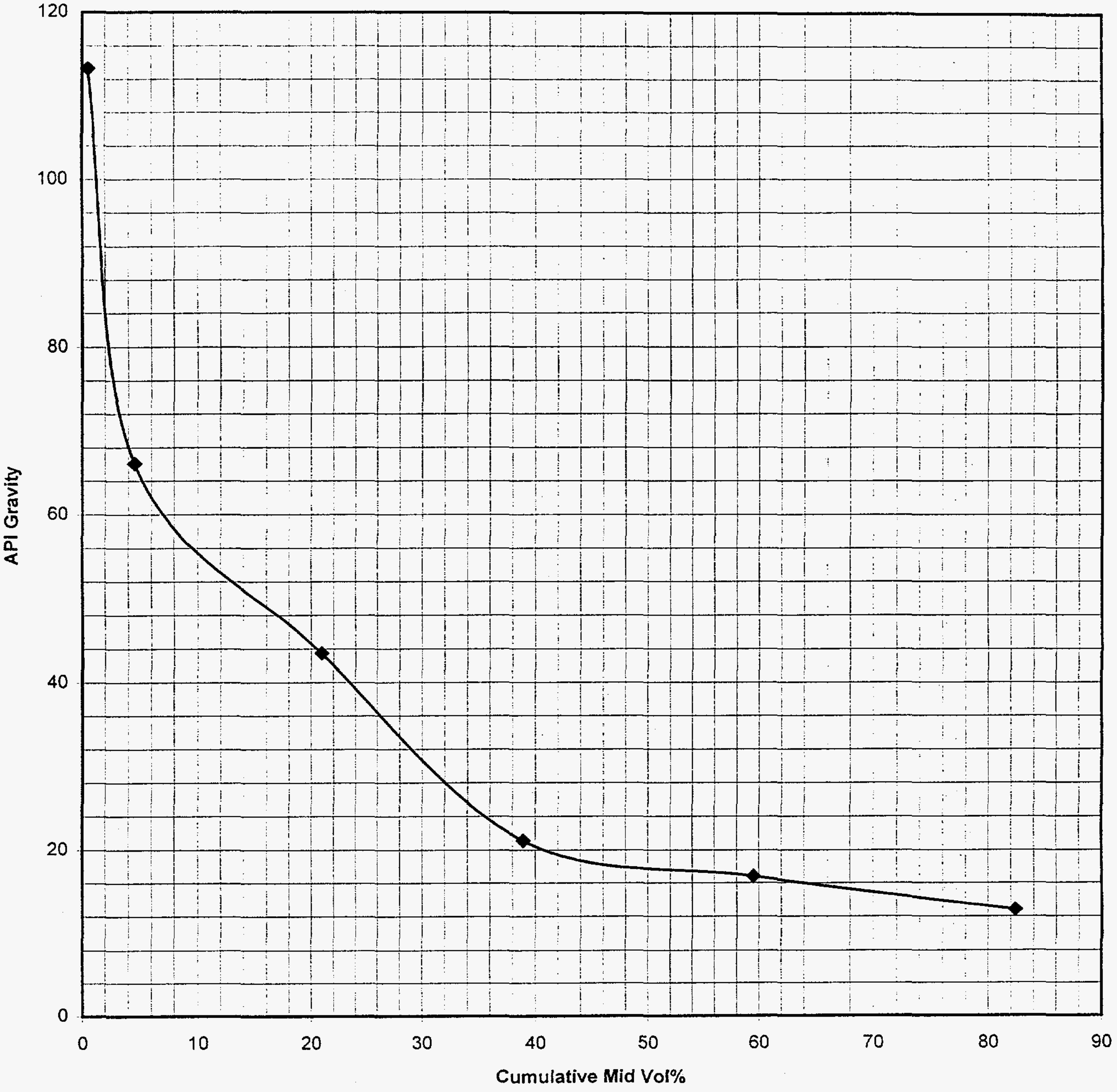
Cut Temp	Degrees F	DUMP	Specific		LIQ	CUM. LIQ	WT%	CUM	API	MID
TO		WT(g)	Gravity	MLS	VOL%	VOL%		WT%	GRAVITY	LIQ VOL%
ASTM D2892 Distillation Yields										
IBP	70	47.50	0.5780	82.18	0.98	0.98	0.62	0.62	113.31	0.49
70	180	425.70	0.7162	594.39	7.10	8.08	5.59	6.22	66.07	4.53
180	350	1736.60	0.8086	2147.66	25.66	33.74	22.82	29.04	43.49	20.91
350	400	805.10	0.9274	868.13	10.37	44.11	10.58	39.62	21.08	38.92
400	550	2453.30	0.9542	2571.05	30.71	74.82	32.24	71.86	16.79	59.47
550	650	1240.00	0.9804	1264.79	15.11	89.93	16.30	88.16	12.83	82.38
650+		901.10	1.0298	875.02	10.45	100.38	11.84	100.00	5.91	

Loss (Grams): 16.2 (0.21 Wt.%)
Distribution: (2/3) 10.8 g to IBP-70 F
(1/3) 5.4 g to 70-180 F

WinAssay '95 Quality Control Applications

Cum. Mid Vol% v. API Gravity

HTI PB-03-9,10,11 (Charge #2)

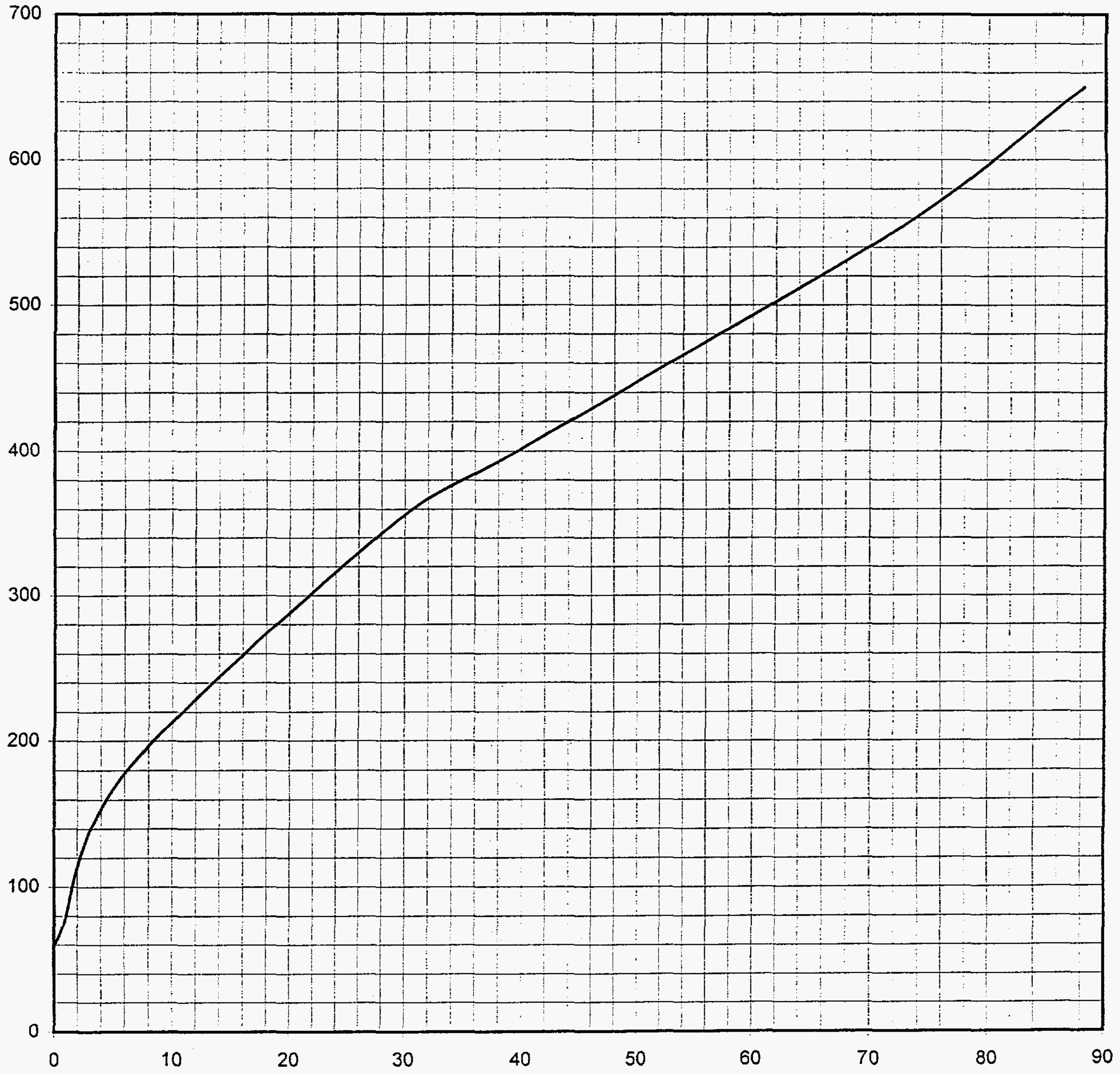


API CURVE Chart 1

WinAssay '95
True Boiling Point Curve
Vaporline Temperature v. Cumulative Wt% Yield

Sample ID

HTI PB-03-9,10,11 (Charge #2)

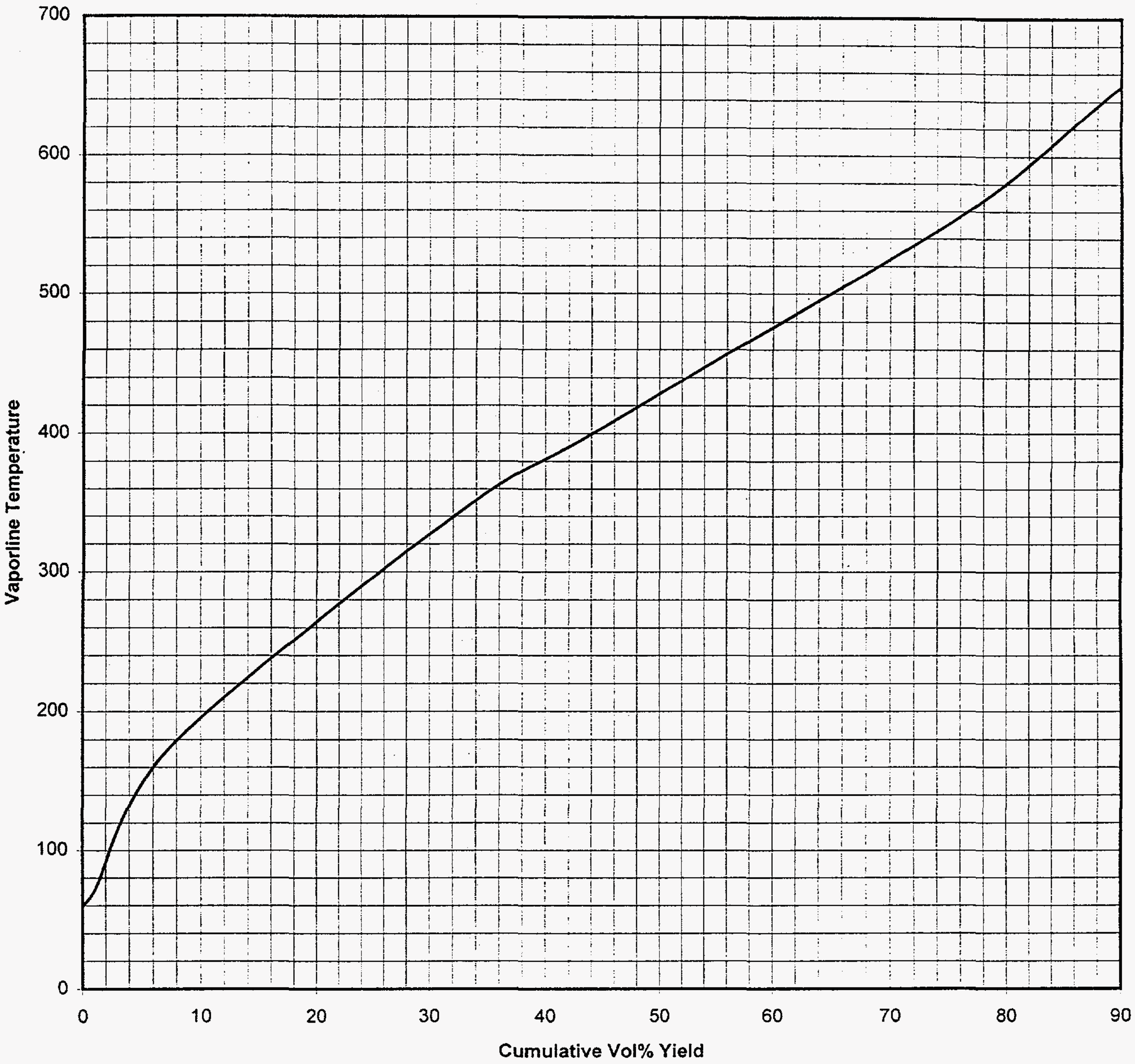


Wt Curve Chart 1

WinAssay '95
True Boiling Point Curve vs Cumulative Vol% Yield

Sample ID

HTI PB-03-9,10,11 (Charge #2)



ITS- Caleb Brett Distillation Group

ASTM D2892/D5236 CHARGE INFORMATION

Lab ID:	96-005170
Client Name:	Consol Inc.
Sample ID:	HTI PB-03-9,10,11 (Charge #2)
Date:	10/01/96

Operator: Robert Kelly

Charge Mass D2892(g):	7669.0
Charge S.G D2892 (60/60F):	0.9090

Charge Mass D5236(g):	0.0
Charge S.G. D5236 (60/60F):	0.0000

Water Weight Removed (g):	59.7
Initial Vapor Temp:	59
Whole Crude Sulfur Wt%:	

CALEB BRETT HOUSTON

TID: 96-005170-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-9,10,11 CRUDE
OIL/IBP-70 F
NID: 51870 Date: 16-SEP-1996

Analyzed: 10/1/96 3:56 PM
Reported: 10-03-1996 13:02:33
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Weight Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.018	0.000	0.000	0.000	0.000	0.018
C3:	8.900	0.000	0.000	0.000	0.277	9.177
C4:	58.508	9.557	0.000	0.000	5.651	73.716
C5:	6.364	5.081	0.000	1.119	1.125	13.688
C6:	0.694	0.783	0.028	1.370	0.219	3.094
C7:	0.000	0.035	0.000	0.003	0.008	0.046
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	74.483	15.456	0.028	2.491	7.280	99.739

Oxygenates: 0.018 Total C14+: 0.000 Total Unknowns: 0.244
Grand Total: 100.000

Molecular Weight and Relative Density Data

Group:	Ave. Mw.:	Ave. Rel. Density:
C1:	0.000	0.000
C2:	30.070	0.340
C3:	44.033	0.501
C4:	57.964	0.578
C5:	71.796	0.634
C6:	85.025	0.705
C7:	99.725	0.689
C8:	0.000	0.000
C9:	0.000	0.000
C10:	0.000	0.000
C11:	0.000	0.000
C12:	0.000	0.000
C13:	0.000	0.000
C14:	0.000	0.000
Total Sample:	58.258	0.578

File: 5170.DHA

CALEB BRETT HOUSTON

TID: 96-005170-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-9,10,11 CRUDE
OIL/IBP-70 F
NID: 51870 Date: 16-SEP-1996

Analyzed: 10/1/96 3:56 PM
Reported: 10-03-1996 13:02:33
Normalized to 100.00%

Composite Report
Totals by Group Type & Carbon Number
(in Volume Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.030	0.000	0.000	0.000	0.000	0.030
C3:	10.313	0.000	0.000	0.000	0.318	10.631
C4:	58.626	9.947	0.000	0.000	5.428	74.001
C5:	5.894	4.758	0.000	0.870	0.993	12.515
C6:	0.611	0.691	0.019	1.041	0.186	2.546
C7:	0.000	0.030	0.000	0.002	0.006	0.039
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	75.473	15.426	0.019	1.913	6.931	99.762

Oxygenates: 0.013 Total C14+: 0.000 Total Unknowns: 0.225
Grand Total: 100.000

(in Mole Percent)

	Paraffins:	I-paraffins:	Aromatics:	Naphthenes:	Olefins:	Total:
C1:	0.000	0.000	0.000	0.000	0.000	0.000
C2:	0.034	0.000	0.000	0.000	0.000	0.034
C3:	11.789	0.000	0.000	0.000	0.385	12.173
C4:	58.796	9.604	0.000	0.000	5.883	74.283
C5:	5.152	4.113	0.000	0.932	0.940	11.136
C6:	0.471	0.530	0.021	0.951	0.153	2.126
C7:	0.000	0.020	0.000	0.002	0.005	0.027
C8:	0.000	0.000	0.000	0.000	0.000	0.000
C9:	0.000	0.000	0.000	0.000	0.000	0.000
C10:	0.000	0.000	0.000	0.000	0.000	0.000
C11:	0.000	0.000	0.000	0.000	0.000	0.000
C12:	0.000	0.000	0.000	0.000	0.000	0.000
C13:	0.000	0.000	0.000	0.000	0.000	0.000
C14:	0.000	0.000	0.000	0.000	0.000	0.000
Total:	76.242	14.268	0.021	1.884	7.365	99.780

Oxygenates: 0.017 Total C14+: 0.000 Total Unknowns: 0.203
Grand Total: 100.000

File: 5170.DHA

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        CALEB BRETT HOUSTON

      TID: 96-005170-0-HOUS-002-00
      CID: CONSOLINC                          Analyzed: 10/1/96 3:56 PM
      SID: HTI PB-03-9,10,11 CRUDE           Reported: 10-03-1996 13:02:33
           OIL/IBP-70 F                       Normalized to 100.00%
      NID: 51870        Date: 16-SEP-1996


               Boiling Point Distribution Data

                   Wt.  Percent Off:           Vol. Percent Off:
                 deg.C.:        deg.F.:      deg.C.:        deg.F.:

    IBP  (0.5%)    -42.04        -43.67       -42.04         -43.67
          5.0%     -42.04        -43.67       -42.04         -43.67
         10.0%     -11.72         10.90       -42.04         -43.67
         15.0%     -11.72         10.90       -11.72          10.90
         20.0%      -6.25         20.75       -11.72          10.90
         25.0%      -0.50         31.10        -0.50          31.10
         30.0%      -0.50         31.10        -0.50          31.10
         35.0%      -0.50         31.10        -0.50          31.10
         40.0%      -0.50         31.10        -0.50          31.10
         45.0%      -0.50         31.10        -0.50          31.10
         50.0%      -0.50         31.10        -0.50          31.10
         55.0%      -0.50         31.10        -0.50          31.10
         60.0%      -0.50         31.10        -0.50          31.10
         65.0%      -0.50         31.10        -0.50          31.10
         70.0%      -0.50         31.10        -0.50          31.10
         75.0%      -0.50         31.10        -0.50          31.10
         80.0%       0.88         33.58        -0.50          31.10
         85.0%      27.84         82.11        20.05          68.09
         90.0%      36.06         96.91        31.15          88.07
         95.0%      36.06         96.91        36.06          96.91
    FBP (99.5%)     80.72        177.30        80.72         177.30


                 Research Octane Number =108.79
                (Calculated from Individual Component Values)

                   Contribution to Total by:
                     Paraffins:        81.07
                     Iso-paraffins:    17.32
                     Aromatics:         0.03
                     Naphthenes:        2.19
                     Olefins:           7.92
                     Oxygenates:        0.02


    File: 5170.DHA
```

CALEB BRETT HOUSTON

TID: 96-005170-0-HOUS-002-00
CID: CONSOLINC
SID: HTI PB-03-9,10,11 CRUDE
OIL/IBP-70 F
NID: 51870 Date: 16-SEP-1996

Analyzed: 10/1/96 3:56 PM
Reported: 10-03-1996 13:02:33
Normalized to 100.00%

Components Listed in Chromatographic Order

Min.	INDEX	Component	Wt%	Vol%	Mol%
8.737	200.0	ethane	0.018	0.030	0.034
9.231	294.2	propylene	0.277	0.318	0.385
9.287	300.0	propane	8.900	10.313	11.789
10.312	366.4	i-butane	9.557	9.947	9.604
10.979	391.0	butene-1	2.701	2.632	2.812
11.282	400.0	n-butane	58.508	58.626	58.796
11.601	411.3	t-butene-2	1.573	1.510	1.638
11.695	414.4	2,2-dimethylpropane	0.040	0.039	0.033
12.112	427.1	c-butene-2	1.377	1.285	1.433
13.480	460.4	3-methylbutene-1	0.108	0.100	0.090
13.712	465.1	?	0.244	0.225	0.203
14.371	477.4	i-pentane	5.041	4.718	4.081
15.145	490.2	pentene-1	0.259	0.235	0.216
15.401	494.1	i-propanol	0.018	0.013	0.017
15.535	496.1	2-methylbutene-1	0.102	0.091	0.085
15.809	500.0	n-pentane	6.364	5.894	5.152
16.229	509.2	t-pentene-2	0.276	0.247	0.230
16.658	518.0	c-pentene-2	0.145	0.129	0.121
16.929	523.4	2-methylbutene-2	0.127	0.111	0.105
17.773	539.0	2,2-dimethylbutane	0.008	0.007	0.005
18.779	555.8	cyclopentene	0.108	0.081	0.092
19.063	560.3	4-methylpentene-1	0.023	0.020	0.016
19.423	565.8	cyclopentane	1.119	0.870	0.932
19.569	567.9	4-methyl-c-pentene-2	0.044	0.038	0.030
19.869	572.3	2-methylpentane	0.503	0.447	0.341
20.642	583.1	1,5-hexadiene	0.012	0.010	0.008
20.762	584.7	3-methylpentane	0.271	0.237	0.184
21.197	590.4	hexene-1	0.031	0.026	0.021
21.967	600.0	n-hexane	0.694	0.611	0.471
22.141	602.5	t-hexene-3	0.016	0.014	0.011
22.322	605.0	t-hexene-2	0.021	0.018	0.015
22.489	607.3	2-methylpentene-2	0.011	0.010	0.008
22.563	608.3	3-methyl-c-pentene-2	0.016	0.013	0.011
22.694	610.1	3-methylcyclopentene	0.007	0.005	0.005
22.795	611.5	O13	0.008	0.006	0.005
22.985	614.0	c-hexene-2	0.011	0.009	0.008
23.451	620.1	3,3-dimethylpentene-1	0.008	0.006	0.005
23.878	625.5	methylcyclopentane	0.677	0.525	0.470
24.217	629.7	2,4-dimethylpentane	0.007	0.006	0.004
25.781	648.0	diolefin	0.020	0.016	0.014
25.915	649.5	benzene	0.028	0.019	0.021
26.663	657.6	cyclohexane	0.692	0.516	0.481
27.543	666.7	2-methylhexane	0.021	0.018	0.012

Components Listed in Chromatographic Order

Min.	INDEX	Component	Wt%	Vol%	Mol%
27.690	668.1	2,3-dimethylpentane	0.008	0.006	0.004
27.958	670.8	1,1-dimethylcyclopentane	0.003	0.002	0.002

APPENDIX 4

UNIVERSITY OF DELAWARE QUARTERLY REPORT

THE KINETICS OF COAL LIQUEFACTION DISTILLATION RESID CONVERSION

QUARTERLY REPORT
7/16/96-10/15/96

Michael T. Klein
Principal Investigator

William H. Calkins
Co-Principal Investigator

He Huang
Research Associate

and

Shaojie Wang
Visiting Scientist

Center For Catalytic Science and Technology
Department of Chemical Engineering
University of Delaware
Newark, Delaware 19716

Date Published
October 29,1996

Subcontract from CONSOL under DOE Contract DE-AC22-94PC9305

EXECUTIVE SUMMARY

Hydroconversion runs on 15 resids supplied by CONSOL, Inc. have been completed using 3 to 1 tetralin to resid wt. ratio with and without 3 wt% sulfided molybdenum naphthenate catalyst at 420 °C for 30 minutes. Two of the resids from the Princeton laboratory and two catalyzed Wilsonville resids have still to be analyzed. The other resids showed 30.2 to 44.9 % conversion to material boiling below 850°F in 30 minutes in the catalyzed experiments. This can be compared to uncatalyzed hydroconversion of the same materials of 15 - 23 wt % under the same conditions. The difference in these resids which can account for the variation of 30 up to 45 % has not yet been investigated. Previous work using a different work up procedure showed that 0.9 wt % sulfided molybdenum naphthenate at the same times and temperature showed only a slight increase in conversion over the uncatalyzed system.

TGA tests run on solubilized and unsolubilized resid from both the catalyzed and thermal hydroconversion experiments show that there is little of no conversion of the unsolubilized residue to material boiling below 454 °C (850 °F). However, there is substantial conversion of the solubilized resid to lower boiling material.

The molecular modeling work is proceeding satisfactorily and the algorithm for the resid structure is in place. Necessary analytical work on the VPO molecular weights on the SARA fractions is almost complete.

QUARTERLY REPORT

HYDROCONVERSION OF COAL DERIVED RESIDS

Hydroconversion of resids

Hydroconversion experiments have now been completed on all 15 resids supplied by CONSOL Inc. under the conditions agreed upon with CONSOL on May 17, 1996. Two of the resids from the Princeton laboratory and two catalyzed Wilsonville resids have still to be analyzed. The hydroconversion conditions were 30 minute runs at 420°C in 3 to 1 tetralin to resid wt ratio and 1500 psig hydrogen with and without using sulfided molybdenum naphthenate catalyst (as 3 wt% molybdenum based on resid charged). Each resid was also run at ambient temperature for comparative purposes.

Hydroconversion experiments using Shell Ni/Mo catalyst are still indeterminate pending the finding of a suitable method of conversion determination. Experiments have shown that the Shell catalyst itself changes under hydroprocessing conditions, making the use of ash tracer as a way of following conversion unreliable. We are considering alternative approaches to determining conversion when using that catalyst.

Work up Procedure for Hydroprocessing Experiments

The work-up of the runs is an extensive process. The reaction mixture is filtered and the filter cake is washed with cold methylene chloride into the filtrate. The methylene chloride is then distilled out of the filtrate at low temperature (below 42°C). The low concentration of the tetralin in the methylene chloride distillate is determined by gas chromatography. The methylene chloride remaining and the tetralin in the resid filtrate are also determined by gas chromatography. SimDis TGA is performed on both the filtrate and the filter cake to allow the determination of the resid conversion to product boiling below 850 °F.

Resid Conversion Kinetics

Since the conversion is determined as the product of the amount of resid solubilized and the percentage of that solubilized product boiling below 850°F, the conversion calculation is based on Equation:

$$Conversion(<850F) = TSF \times (1 - \frac{850F+}{RSF})$$

where the Tetralin Soluble Fraction (TSF) of resid (dry-ash-free basis) is determined by ash content in the solid residue after resid hydroprocessing; the Resid Soluble Fraction (RSF) in tetralin and the 850°F$^+$ fraction of the RSF are determined by running GC and SimDis TGA on the liquid products, respectively.

Table 1 shows the thermal and catalytic reactor runs made and the conditions used. Table 2 shows the solubilization of the thermal and catalyzed runs as well as the room temperature controls. Table 3 shows the increase in solubilization over the room temperature controls in the thermal and catalytic runs. There is a substantial solubility of all the resids in tetralin even at room temperature. The increases in solubilization over the room temperature experiments are only of the order of 10 to 20 %. The presence of a catalyst increases the solubilization only a small amount (1 to 4%). Tables 4 and 5 show the conversions of resid to material boiling below 850°F for the thermal and catalyzed runs, respectively.

TGA on the solid residues using the ramp method shows that all the insoluble portion of the resids or reactor solid residues shows no material boiling below 850°F. The hydroconversion of the resid apparently occurs only on the solublized resid. This conversion is only about 15 - 20 % for the thermal hydroconversion to 850°F$^-$ with the tetralin donor solvent alone. On the other hand, in the presence of 3% molybdenum naphthenate catalyst, the conversion increases to 30.2 to 44.9 % depending upon the resid. All of the 13 Wilsonville resids and two HTI resids have been run under thermal and catalytic conditions and have been analyzed. The recovery in processing the two very sticky HTI resids was relatively poor. Two more of the Wilsonville resids converted in the presence of catalyst and two HTI resids have not yet been completely analyzed.

Several difficulties have been experienced with the gas chromatograph, which is used in the analytical scheme. The latest problem relates to the drifting response factor of the FID detector. We are trying to avoid this problem by using an internal standard, and all the samples of filtrate and distillate are now being analyzed in this way. We believe, however, that the experimental errors in the conversion data presented in this report cannot be more than 5% (relative). With the use of internal standards, we expect to minimize them.

MODELING OF RESID HYDROCONVERSION

Analysis of SARA Samples

To date molecular weights have been determined for each of the aromatic, aliphatic and preasphaltene fractions of the SARA separations (Table 6). However, the resin samples which were dissolved in dichlorobenzene yielded results which are too high and these samples are unrecoverable. It will be assumed that these samples have a MW of 500. The value of 500 falls within 2 experimental standard deviations of every measured resin, so this assumption should not lead to much error. To account for this approximation, the constraint in the structural model for these resids which requires the molecular weight of the resin fraction to match the experimentally determined value will be relaxed.

The molecular weights for the first six saturate fractions have also been determined. Only the remaining nine saturate fractions need to be determined. All other analytical information has been supplied by CONSOL, Inc.

Modeling

A molecular structure model for coal resid has been assembled (see Table 7 and Figure 1). Currently, a molecular representation of the first resid (W258 V-131B) is being optimized to the analytical properties of the initial feedstock. The molecules have been defined by their basic structural attributes (e.g. number of aromatic rings, number of thiophenic rings, number of sidechains, number of unit sheets, etc.) and a construction algorithm is presented in Figure 1. The attributes are represented by probability distribution functions (pdf's) which give the probability of finding a value or less of a given attribute. Table 7 lists the distributions used in this algorithm.

By stochastic (Monte Carlo) sampling of the pdf's, a molecular sample of a resid can be constructed. The properties of this representation can be compared to experimental values. The parameters of the pdf's are optimized to minimize the difference between the predicted and experimental values. The objective function used to optimize these parameters is:

$$\begin{aligned}\chi^2 = {} & \left(\frac{Hwt_{\exp} - Hwt_{pred}}{0.0016}\right)^2 + \left(\frac{Cwt_{\exp} - Cwt_{pred}}{0.0064}\right)^2 + \left(\frac{Owt_{\exp} - Owt_{pred}}{0.0150}\right)^2 + \\ & \left(\frac{Nwt_{\exp} - Nwt_{pred}}{0.0011}\right)^2 + \left(\frac{Swt_{\exp} - Swt_{pred}}{0.0010}\right)^2 + \\ & \left(\frac{1}{\#\ Comps}\right)\sum_{i=1}^{\#\,Comps}\left(\frac{SaraWt_{\exp} - SaraWt_{pred}}{0.03}\right)^2 + \left(\frac{Harno_{\exp} - Harno_{pred}}{0.02}\right)^2 + \\ & \left(\frac{Hcarno_{\exp} - Hcarno_{pred}}{0.02}\right)^2 + \left(\frac{Haano_{\exp} - Haano_{pred}}{0.02}\right)^2 + \\ & \left(\frac{Hcano_{\exp} - Hcano_{pred}}{0.02}\right)^2 + \left(\frac{Habno_{\exp} - Habno_{pred}}{0.02}\right)^2 + \\ & \left(\frac{Hcbno_{\exp} - Hcbno_{pred}}{0.02}\right)^2 + \left(\frac{Hgno_{\exp} - Hgno_{pred}}{0.02}\right)^2 + \left(\frac{Phox_{\exp} - Phox_{pred}}{0.02}\right)^2\end{aligned}$$

The numerator of each term represents the residual between the measured and predicted values. The denominator is a weighting factor equal to approximately one standard deviation. The weighting factor ensures that values which are measured with higher accuracy are represented more closely than values measure with lower accuracy.

The model will be fine-tuned with the first few resids. In particular, an investigation into the importance of conditional probability and into the approximation of the intersheet linkages will be made. It may be necessary to make some slight alterations to the current model to get an optimal representation. Later, some reaction product data may also be added to the objective function.

Sample molecules from this representation will be examined with a view to selecting model structures for determining hydroconversion rate constants in the SCTBR reactor. Such data is not essential to the functioning of the model. However, it could provide a useful starting point for the reaction rate constants.

SUMMARY AND CONCLUSIONS

1. A distinct increase in the production of material boiling below 850°F is observed in the presence of 3 wt% sulfided molybdenum naphthenate catalyst under the conditions used. This is substantially higher than that observed in hydroprocessing in the absence of catalyst (thermal) or when only 0.9 wt% of the same catalyst is used.

2. Little or no conversion of the unsolubilized resid to lower boiling material appears to occur even in the presence of the catalyst.

3. The solubilization of the resid in tetralin is substantial even at room temperature. It increases only 10 to 20 wt% under hydroprocessing conditions without catalyst. There is very little increase in solubilization in the presence of catalyst. However, the breakdown of the soluble resid into low boiling material depends strongly on the catalyst.

4. Conversions of various resids under catalytic conditions have varied from 30 to 45 wt%. These values are being correlated with analytical data of the resids.

5. A molecular structure model for coal derived resid has been assembled and is being optimized.

6. Molecular weight determination of the various SARA fractions is almost complete.

FUTURE PROGRAM

1. In consultation with CONSOL Inc., one or more selected resids will be hydroprocessed over a range of conditions, i.e., time, temperature, tetralin to resid ratio, catalyst concentration, hydrogen pressure and solvent type (including Wilsonville recycle solvent) to establish what conversion of recycle resid can be expected under actual process conditions.

2. An effort will be made to correlate conversion data with the available structural information (NMR, elemental analysis etc.).

3. If time permits, an effort will be made to relate hydroprocessing performance in the presence of molybdenum naphthenate catalyst to that of the use of sulfided Ni-Mo/Al_2O_3 catalysts used at Wilsonville and the other promising catalysts developed in recent years.

4. If time permits, some rate constants of selected model compounds will be determined for hydroprocessing to assist in the molecular modeling work.

PUBLICATIONS

Three papers were presented to the New Orleans National Meeting of the American Chemical Society in the Spring of 1996 that relate in part to work done under this project. They are:

1. He Huang, Keyu Wang, Shaojie Wang, M.T. Klein, and W.H. Calkins 'Applications of the Thermogravimetric Analysis in the Study of Fossil Fuels', *Prepr. Pap. - Am. Chem. Soc., Div. Fuel Chem.* **1996**, 41(1), 1.

2. Keyu Wang, Shaojie Wang, He Huang, M.T. Klein, and W.H. Calkins 'A Novel Smoothing Routine for the Data Processing in Thermogravimetric Analysis', *Prepr. Pap. - Am. Chem. Soc., Div. Fuel Chem.* **1996**, 41(1), 27.

3. He Huang, Keyu Wang, Shaojie Wang, M.T. Klein, and W.H. Calkins 'Distillation of Liquid Fuels by Thermogravimetry', *Prepr. Pap. - Am. Chem. Soc., Div. Fuel Chem.* **1996**, 41(1), 87.

Table 1 Reactor Runs

Sample	Name		Thermal			Catalytic	
			Run #	T, C		Run #	T, C
Resid A	W258V-131B		C096	425		C109	422
Resid B	W259R-1235		C097	421		C110	417
Resid C	W259V-131B		C098	422		C123	419
Resid D	W261V-131B		C099	426		C124	420
Resid E	W262R-1235		C100	420		C125	421
Resid F	W262V-1067		C101	422		C126	418
Resid G	W262V-131B		C102	422		C127	420
Resid H	W260V-131B		C103	420		C128	419
Resid I	W261V-1067		C104	420		C129	420
Resid J	W259V-1067		C105	421		C130	420
Resid K	W260R-1235		C106	421		C131	419
Resid L	W260V-1067		C107	418		C132	421
Resid M	W261R-1235		C108	418		C133	418
Resid N	HTI POC-01, O-43		C134	423		C137	421
Resid O	HTI POC-02, O-43		C135	422		C136	423
Thermal: 30 min; 1500 psig H2							
Catalytic: 30 min; 1500 psig H2; 3 wt% Mo (molybdenum naphthenate) based on the amount of							
the resid chaged; ca. 3 g of methyl disulfide							

Table 2 Tetralin Soluble Fraction of Resid

Sample	Name	Ash	Thermal			Catalytic			Control		
			Run #	Ash	TSF	Run #	Ash	TSF	Run #	Ash	TSF
Resid A	W258V-131B	17.0	C096	50.1	79.7	C109	51.5	80.7	C091	40.2	69.6
Resid B	W259R-1235	8.7	C097	33.5	81.0	C110	40.0	85.6	C111	21.9	65.8
Resid C	W259V-131B	8.5	C098	35.0	82.7	C123	41.4	86.8	C112	24.6	71.5
Resid D	W261V-131B	9.9	C099	45.8	87.1	C124	54.9	91.0	C113	29.7	74.1
Resid E	W262R-1235	15.6	C100	43.5	76.0	C125	44.2	76.6	C114	32.6	61.7
Resid F	W262V-1067	17.5	C101	47.8	76.9	C126	49.4	78.3	C115	38.6	66.4
Resid G	W262V-131B	15.9	C102	46.5	78.3	C127	48.3	79.8	C116	39.1	70.6
Resid H	W260V-131B	15.2	C103	46.6	79.5	C128	50.9	82.7	C117	38.3	71.2
Resid I	W261V-1067	15.9	C104	50.8	81.8	C129	56.6	85.5	C118	37.9	69.2
Resid J	W259V-1067	10.2	C105	38.7	82.0	C130	43.8	85.4	C119	29.0	72.2
Resid K	W260R-1235	17.2	C106	49.0	78.4	C131	52.1	80.8	C120	35.6	62.4
Resid L	W260V-1067	16.7	C107	51.3	81.0	C132	53.5	82.6	C121	37.4	66.6
Resid M	W261R-1235	13.7	C108	45.6	81.1	C133	53.7	86.3	C122	30.9	64.5
Resid N	HTI POC-01, O-43	0.4	C134	33.0	99.2	C137	36.4	99.3	C139	33.4	99.2
Resid O	HTI POC-02, O-43	4.1	C135	38.0	93.1	C136	48.3	95.4	C138	37.4	92.9

Thermal: 420 C; 30 min; 1500 psig H2

Catalytic: 420 C; 30 min; 1500 psig H2; 3 wt% Mo

Control: 25 C; 10 min; 1500 psig H2

TSF: Tetralin Soluble Fraction of resid, wt% (daf basis)

Table 3 Differences of the Tetralin Soluble Fraction of resid at various processing conditions

Sample	Name		Tetralin Soluble Fraction of resid, daf basis		
			Thermal - Control	Catalytic - Control	Catalytic - Thermal
Resid A	W258V-131B		10.1	11.2	1.1
Resid B	W259R-1235		15.2	19.8	4.6
Resid C	W259V-131B		11.2	15.3	4.1
Resid D	W261V-131B		13.0	16.9	4.0
Resid E	W262R-1235		14.3	14.9	0.6
Resid F	W262V-1067		10.5	11.9	1.4
Resid G	W262V-131B		7.8	9.3	1.5
Resid H	W260V-131B		8.3	11.5	3.2
Resid I	W261V-1067		12.6	16.4	3.8
Resid J	W259V-1067		9.8	13.2	3.4
Resid K	W260R-1235		15.9	18.4	2.5
Resid L	W260V-1067		14.4	16.1	1.6
Resid M	W261R-1235		16.5	21.8	5.3
Resid N	HTI POC-01, O-43		0.0	0.1	0.1
Resid O	HTI POC-02, O-43		0.2	2.6	2.4
Thermal: 420 C; 30 min; 1500 psig H2					
Catalytic: 420 C; 30 min; 1500 psig H2; 3 wt% of Mo					
Control: 25 C; 10 min; 1500 psig H2					

Table 4 Conversion of thermal hydroprocessing of resid

Resid			Solid Residue		Liquid Residue			Conversion to 850 F-
Sample	Name	Ash	Ash	TSF	Tetralin	SRF	850 F+	
Resid A	W258V-131B	17.0	50.1	79.7	88.0	12.0	9.6	16.1
Resid B	W259R-1235	8.7	33.5	81.0	88.1	11.9	9.6	15.3
Resid C	W259V-131B	8.5	35.0	82.7	86.6	13.4	10.8	15.8
Resid D	W261V-131B	9.9	45.8	87.1	88.1	12.0	9.7	16.3
Resid E	W262R-1235	15.6	43.5	76.0	88.8	11.2	8.6	17.8
Resid F	W262V-1067	17.5	47.8	76.9	87.8	12.2	9.7	15.8
Resid G	W262V-131B	15.9	46.5	78.3	88.6	11.4	8.7	18.7
Resid H	W260V-131B	15.2	46.6	79.5	87.6	12.4	9.0	21.8
Resid I	W261V-1067	15.9	50.8	81.8	88.6	11.4	8.5	21.1
Resid J	W259V-1067	10.2	38.7	82.0	86.1	13.9	11.5	14.4
Resid K	W260R-1235	17.2	49.0	78.4	87.2	12.8	9.8	18.4
Resid L	W260V-1067	16.7	51.3	81.0	89.3	10.7	8.3	18.1
Resid M	W261R-1235	13.7	45.6	81.1	90.2	9.8	7.9	15.4
Resid N	HTI POC-01, O-43	0.4	33.0	99.2				
Resid O	HTI POC-02, O-43	4.1	38.0	93.1				
Thermal: 420 C; 30 min; 1500 psig H2								
Catalytic: 420 C; 30 min; 1500 psig H2; 3 wt% Mo								
Control: 25 C; 10 min; 1500 psig H2								
TSF: Tetralin Soluble Fraction of resid, wt% (daf basis)								
RSF: Resid Soluble Fraction in tetralin, wt%								
850 F+: fraction of boiling above 850 F								

Table 5 Conversion of catalytic hydroprocessing of resid

Resid			Solid Residue		Liquid Residue			Conversion
Sample	Name	Ash	Ash	SF	Tetralin	SR in Tetralin	850 F+	
Resid A	W258V-131B	17.0	51.5	80.7	82.4	17.6	9.9	35.3
Resid B	W259R-1235	8.7	40.0	85.6	80.3	19.7	11.2	36.9
Resid C	W259V-131B	8.5	41.4	86.8	81.9	18.1	11.5	31.7
Resid D	W261V-131B	9.9	54.9	91.0	77.3	22.7	14.1	34.4
Resid E	W262R-1235	15.6	44.2	76.6	81.4	18.6	11.3	30.2
Resid F	W262V-1067	17.5	49.4	78.3	80.2	19.8	10.7	36.2
Resid G	W262V-131B	15.9	48.3	79.8	80.0	20.0	11.5	34.1
Resid H	W260V-131B	15.2	50.9	82.7	75.4	24.6	11.3	44.9
Resid I	W261V-1067	15.9	56.6	85.5	76.0	24.0	11.9	43.0
Resid J	W259V-1067	10.2	43.8	85.4	77.2	22.8	11.9	40.8
Resid K	W260R-1235	17.2	52.1	80.8	78.9	21.1	12.4	33.5
Resid L	W260V-1067	16.7	53.5	82.6				
Resid M	W261R-1235	13.7	53.7	86.3				
Resid N	HTI POC-01, O-43	0.4	36.4	99.3				
Resid O	HTI POC-02, O-43	4.1	48.3	95.4				
Thermal: 420 C; 30 min; 1500 psig H2								
Catalytic: 420 C; 30 min; 1500 psig H2; 3 wt% Mo								
Control: 25 C; 10 min; 1500 psig H2								
TSF: Tetralin Soluble Fraction, wt% (daf basis)								
RSF: Resid Soluble Fraction in tetralin, wt%								
850 F+: fraction of boiling above 850 F								

Table 6: VPO results

Sample Number	Saturate MW	Aromatic MW	Resin MW	Asphaltene MW	Pre-Asphaltene MW
1	288	320	676* (500)	778	988
2	250	274	613* (500)	926	1060
3	295	319	539	680	919
4	305	362	718* (500)	595	988
5	443	493	789* (500)	864	1037
6	347	420	(500)	789	1201
7		348	426	708	1202
8		425	535	686	1025
9		323	499	759	1087
10		430	520	657	895
11		386	479	760	829
12		413	502	653	1114
13		336	542	745	1107
14		356	501	718	1087
15		345	515	743	997

* MWs determined using dichlorobenzene as solvent, results likely too high.

Table 7 Attribute distributions used to construct a molecular representation for coal resid.

Distribution	**Name**	**Used to construct fractions:**
Paraffin Length	npdis	Paraffins
# of Naphthenic Rings	nrdis	Naphthenics
# of Sidechains	nscdis	Naphthenics, Aromatics/Resins, Asphaltenes, Preasphaltenes
Length of Naphthenic Sidechains	nsldis	Naphthenics
Length of Aromatic Sidechains	asldis	Aromatics/Resins, Asphaltenes, Preasphaltenes
Naphthenic # of Unit Sheets	ndpdis	Naphthenics
Resin # of Unit Sheets	rdpdis	Aromatics/Resins
Asphaltene # of Unit Sheets	adpdis	Asphaltenes
Preasphaltene # of Unit Sheets	pdpdis	Preasphaltenes
# of Aromatic Rings	ardis	Aromatics/ Resins, Asphaltenes, Preasphaltenes
# of Naphthenic Rings on an Aromatic Core	nardis	Aromatics/ Resins, Asphaltenes, Preasphaltenes
# of Phenolic Oxygens per unit sheet	frphh	Aromatics/Resins, Asphaltenes, Preasphaltenes
Intersheet Connections		
Fraction of Oxygen Connections	0.25	Resins, Asphaltenes, Preasphaltenes
Fraction of Sulfur Connections	0.25	Resins, Asphaltenes, Preasphaltenes
Fraction of Methylene Connections	0.50:0.25	Napthenics:Aromatics/ Resins, Asphaltenes, Preasphaltenes
Fraction of Biphenyl Connections	0.50:0.25	Napthenics:Aromatics/ Resins, Asphaltenes, Preasphaltenes
Heteroatomic Rings		
Fraction of Oxygen Rings	frorings	Resins, Asphaltenes, Preasphaltenes
Fraction of Sulfur Rings	frsrings	Resins, Asphaltenes, Preasphaltenes
Fraction of Nitrogen Rings (5 and 6 members in a 2 to 1 ratio)	frnrings	Resins, Asphaltenes, Preasphaltenes
Fraction of no Heteroatomic Rings	frhcrings	Resins, Asphaltenes, Preasphaltenes

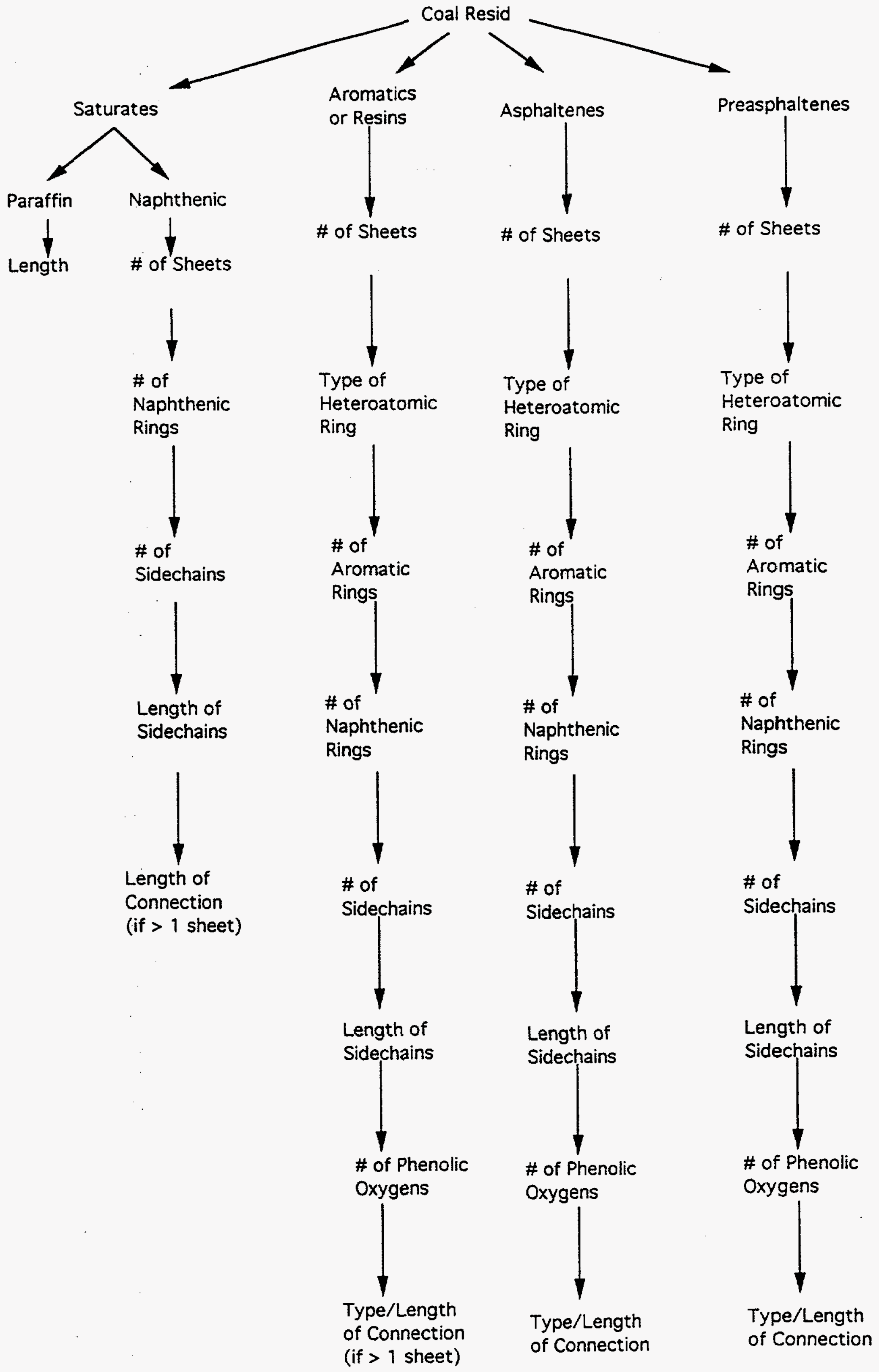

Figure 1: Coal Resid Construction Algorithm