Upgrading of Coal Liquids for Use as Power Generation Fuels

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Work Period: February 1977 - January 1978

Prepared by

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ABSTRACT

Residual coal liquids were hydroprocessed in a fixed bed hydroprocessing unit to upgrade them to high quality power generation fuels. Greater than 95, 90, and 80 percent of the sulfur, oxygen, and nitrogen, respectively, were removed at hydrogen consumptions as high as 4300 scf/B. The conversion of the resins and asphaltenes to saturates, oils, and light liquids appears to be strongly dependent upon the oxygen removal. SRC products produced at short contact times were upgraded to the same extent as regular SRC when processed at similar conditions. Trace metal contaminants in the SRC products deposit on the hydroprocessing catalysts and contribute to catalyst deactivation. A kinetic model linking the rates of conversion and heteroatom removal to the operating conditions was developed.

A 30-day aging study was conducted on an H-Coal distillate product. A relatively low aging rate of 5-10°F/month indicates that acceptable cycles can be obtained with this feed.

Tests in a small scale turbine combustor at Westinghouse Research indicate that H-Coal and SRC distillate liquids would be unacceptable in today's gas turbines. However, when hydroprocessed, these liquids gave results comparable to a No. 2 petroleum fuel oil.

EPRI PERSPECTIVE

PROJECT DESCRIPTION

Heavy residual coal liquids, which can represent a large proportion of the products resulting from primary coal liquefaction processes, exhibit properties that do not especially qualify them as high-quality power generation fuels for use in combustion turbines and in some boiler applications. The properties involved include low hydrogen content, high viscosity, and high concentrations of sulfur and nitrogen. During the reporting period, the project investigated (with laboratory-scale continuous equipment) the use of standard petroleum-refining techniques as a means of upgrading residual liquids obtained from the H-Coal (RP238) and solvent-refined coal (RP1234) projects to higher quality utility fuels.

Another aspect of the recent work was the investigation of the use of these same petroleum-refining techniques for upgrading the products of short-residence-time solvent refining (SRT-SRC) to acceptable utility fuel products. The products were produced at the Wilsonville SRC pilot plant. By uncoupling the coal dissolution step from a subsequent catalytic hydroprocessing step, the potential for reduced overall hydrogen consumption is created.

In addition, observations on changes in the performance obtained in a small-scale turbine combustor at Westinghouse Electric Corp. were made for a number of different coal liquids that had been hydroprocessed to a variety of hydrogen contents.

This report is the second annual report on RP361-2. A final report on the overall project is projected for the spring of 1979.

PROJECT OBJECTIVE

The overall objective of this project is to evaluate various petroleum-refining operations as a means of upgrading a variety of coal liquids to acceptable turbine and boiler fuels. As part of this effort, predictive kinetic process models for each processing scheme are being developed, small-scale turbine combustor tests have been run on upgraded liquids to obtain performance data, and data have been provided for preliminary economic evaluations that relate the level of upgrading to performance and quality parameters.

CONCLUSIONS AND RECOMMENDATIONS

During this reporting period, the project demonstrated that residual coal liquids can, through the choice of the proper catalysts, be hydroprocessed to higher quality power generation fuels having greatly reduced sulfur and nitrogen content and substantially improved handling properties. The product quality of the upgraded fuels would appear to conform to many of the requirements of turbine and high-quality boiler fuels. Hydrogen usage, however, is considerable and adds considerably to fuel cost.

The results of the small-scale turbine combustor tests carried out by Westinghouse under a subcontract from Mobil Research and Development Corp., identified hydrogen content as a key parameter for achieving acceptable combustion performance. However, subsequent investigations carried out at Westinghouse (RP989) using a larger test combustor (referred to as a subscale unit) have demonstrated that acceptable combustion performance from upgraded coal-derived distillate fuels is achieved at hydrogen levels significantly below those identified in this report. Since the cost of upgrading these raw distillates is closely tied to product hydrogen content, it appears that less catalytic upgrading is required than the results presented in this report may indicate. This of course reduces the potential cost of acceptable fuel.

As an approach to reduce overall hydrogen consumption, prior work at Mobil (RP410) and Battelle (RP779-5) has indicated that SRT-SRC can be achieved with a very low hydrogen consumption, but this produces an equally low-quality product. The present study has demonstrated that catalytic hydroprocessing may be used to upgrade this low-quality residual product to the same extent as the conventional SRC at similar levels of processing severities. This demonstrates the initial technical feasibility of a process concept that can achieve a desirable level of product quality while offering the potential for reduced hydrogen usage, which has a significant cost advantage. Actual integration of the dissolution step with the subsequent upgrading step is required to further assess this processing approach.

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Section 1

SUMMARY

Coal liquefaction products from the H-Coal and SRC processes were hydroprocessed in a bench scale fixed bed catalytic pilot unit. Emphasis was placed on high concentration blends of SRC in process derived recycle solvent. These included SRC products from Illinois No. 6 and Wyodak coals produced at regular and short contact times at the SRC pilot plant in Wilsonville, Alabama. A short hydroprocessing study was also made on an H-Coal fuel oil and a distillate H-Coal liquid produced in the HRI Process Development Unit in Trenton. Samples of the raw and hydroprocessed H-Coal distillate were successfully burned in a small scale gas turbine combustor at the Westinghouse Research Center in Pittsburgh.

Residual containing coal liquids can be significantly upgraded by hydroprocessing over commercial NiMo and CoMo on alumina catalysts. Liquids containing high concentrations of residual coal liquids yield two phase products when processed over small pore catalysts. Processing over a large pore catalyst gave one phase products. Apparently the small pore catalysts upgrade the distillate fraction of these liquids to the point where it is no longer compatible with the residual fraction.

Blends of SRC in process derived recycle solvent containing as much as 90% SRC were processed in our unit. Greater than 95, 90, and 80 percent removal of the sulfur, oxygen, and nitrogen were achieved at hydrogen consumptions as high as 4300 scf/B. Gradient Elution Chromatographic analyses of the charge and product showed conversion of up to 95% of the resins and asphaltenes. The polar asphaltenes are converted to asphaltenes and resins and then to saturates, oils, and light liquids. Oxygen removal appears to be an important step in the conversion of these resins and asphaltenes. A kinetic model linking the rates of conversion and heteroatom removal to the operating conditions was developed.

Short contact time products from Wilsonville were analyzed extensively. These liquids represent a step back on the reaction path toward coal. They are significantly higher in sulfur content as well as asphaltenes and resins. The conversion to lighter products and removal of heteroatoms from these liquids occur at about the

same rate as for regular SRC. When processed at the same conditions, regular and short contact time SRC's give very similar products in terms of elemental composition, GEC analyses and physical properties.

The trace metals in the SRC liquids are deposited on the hydroprocessing catalyst. This removes these contaminants from the liquids. However, the deposition of these metals contributes to catalyst deactivation. The data reported here were obtained in short (≈10 days) process variable studies and no aging studies were made. However, the data indicate that the catalyst deactivation rate may be significant.

The concentration of SRC in the feed blend appears to affect the rate of conversion. When processing a given feed rate of SRC, optimum conversion occurs when the SRC is diluted to about 50-70 wt % concentration in recycle solvent. Apparently the SRC inhibits its own conversion.

A 34-day aging study on an H-Coal distillate showed a deactivation rate of about 5-10°F/month. This rate should give an acceptable cycle length in a fixed bed unit. The products from the two different operating severities in this study were collected and evaluated by Westinghouse Research in their small scale combustor. Combustion results from these liquids, and from the raw and hydroprocessed SRC recycle solvents produced last year, show that the raw coal liquids are unacceptable for burning in present-day gas turbines. They form coke deposits in the combustor, give increased NO_x emissions and result in higher combustor wall temperatures. The raw H-Coal distillate was significantly better than the SRC recycle solvent; however, it was still inferior to the No. 2 petroleum fuel oil used as a base fuel. Hydroprocessing upgraded these liquids by decreasing the fuel bound nitrogen and aromatic content such that their performance in the laboratory combustor was nearly equivalent to the base fuel.

The principal conclusions of this work are:

- Small pore (≲90 Å) catalysts give two phase products when hydroprocessing blends of residual coal liquids. Larger pore catalysts do not.
- Greater than 95, 90, and 85 percent removal of sulfur, oxygen, and nitrogen, respectively, can be achieved by hydroprocessing residual coal liquids. Hydrogen consumptions are as high as 4300 scf/B.
- When hydroprocessing residual coal liquids, polar asphaltenes are converted to resins and then to aromatic oils, saturates, and lighter liquids. Oxygen removal is important for this conversion.

- Short contact time SRC can be hydroprocessed at about the same conditions as regular SRC to yield very similar products.
- Catalyst aging with H-Coal distillate is about 5-10°F/month. Aging with residual coal liquids is much faster due to coking and metal deposition.
- When burned in a small-scale combustor, raw coal liquids give increased wall temperature and NO_x emissions relative to petroleum-derived No. 2 fuel oil. Hydroprocessing significantly reduces these effects.

Section 2

INTRODUCTION

Coal liquefaction processes currently under development are directed at producing boiler fuels for stationary power plants and synthetic crudes for subsequent refining to gasoline and distillate fuels. Although these coal liquids are lower in sulfur and ash content than the coals from which they are derived, they will undoubtedly require further processing to enable them to be used as gas turbine fuels. Presently, gas turbines account for about five percent of installed power generation capacity. Most of this capacity is dedicated to peak load generation where high reliability and the ability to put the capacity on-line rapidly are of critical importance. In the future, gas turbine capacity is expected to grow for both peaking load and for intermediate and base load as high temperature combined cycle systems are brought on-line.

Currently, gas turbines operate commercially on both gaseous and liquid fuels. The liquid fuels are predominantly petroleum-derived kerosenes and light distillates and to a lesser extent heavy distillates and residual fuel oil. All of these liquids are characterized by stringent specifications defining fluidity properties, combustion characteristics, low emissions of nitrogen and sulfur oxides, and absence of trace metals.

In February 1975, a study was initiated by Mobil Research and Development Corporation for the Electric Power Research Institute on the potential use of coal liquids as gas turbine fuels. Results from this study were reported in the Phase I Report under EPRI Project 361-1 (2-1). It was found that products from current coal liquefaction processes require considerable upgrading to meet specifications for gas turbine fuels. Detailed physical and chemical characterization of products from the Solvent Refined Coal, H-Coal, and Synthoil liquefaction processes show that these materials are extremely deficient in hydrogen and contain considerably more nitrogen, oxygen, and trace metals than petroleum-derived fuels currently used in gas turbines. Based on C^{13}-NMR and Gradient Elution Chromatography analyses, the coal liquids are significantly more aromatic and contain more polar asphaltenes of lower molecular weight than petroleum vacuum residua. These chemical differences cause strong incompatibilities in blends of coal liquids with petroleum fuels. Exploratory process studies in a shaker bomb reactor suggest that petroleum refining processes such as hydroprocessing and solvent deasphalting can be used to upgrade coal liquids sufficiently for use as gas turbine fuels.

In February 1976, Mobil Research and Development Corporation signed a contract (RP 361-2) with the Electric Power Research Institute to conduct a two-year program to upgrade coal liquids to gas turbine fuel. The objective of this project is to evaluate hydroprocessing, vacuum distillation, and solvent deasphalting, in bench-scale laboratory units, as means of upgrading coal liquids to proposed turbine fuel specifications. A secondary objective is to define chemical, physical and compositional properties of the upgraded coal liquids in terms of processing conditions and to relate these properties to the compatibility of coal liquids with petroleum fuels and to the burning characteristics of coal liquids in turbine combustors.

Work during the first year of this contract (RP 361-2) was directed primarily at the upgrading of light naphtha and distillate coal liquids. It was found that hydroprocessing could upgrade these liquids to quality levels approaching existing petroleum-derived turbine fuels. The required operating conditions were somewhat more severe and the hydrogen consumptions significantly higher than for hydroprocessing petroleum-derived liquids with a similar boiling range. A limited amount of work on the hydroprocessing of low concentration blends of SRC's in process-derived recycle solvent showed these heavy coal liquids to require even higher severity operation and higher hydrogen consumption. The raw and hydroprocessed distillate coal liquids were found to be compatible with petroleum fuels. The raw residual liquids were incompatible; however, hydroprocessing significantly reduced this incompatibility. A kinetic model was developed for heteroatom removal and aromatic saturation in the distillate coal liquids.

This report covers the work completed during the second year of this contract, February 1977 through January 1978. Coal liquids, obtained from the Wilsonville SRC Pilot Plant and from the H-Coal Process Development unit processing Illinois No. 6 Burning Star, Monterey and Wyodak coals, have been analyzed and processed in fixed-bed bench-scale hydroprocessing units. A number of commercial and

Mobil proprietary hydrotreating catalysts were used in these studies. Whereas last year's work concentrated primarily on the naphtha and distillate boiling range liquids, more effort this year was placed on the heavier residual-containing liquids, particularly SRC. High concentration blends ($\leq$90%) of SRC in process-derived recycle solvent were successfully processed in a pilot unit specifically designed for these viscous refractory stocks. The effects of coal source and SRC blend concentration were determined. In addition, samples of SRC produced at short contact times at Wilsonville were analyzed and processed to determine their processibility relative to regular SRC. A small sample of H-Coal fuel oil product was also analyzed and processed. The products from this processing were analyzed and their compatibility with typical petroleum fuels was determined. A kinetic model relating the processing conditions to heteroatom removal and SRC conversion was developed.

Samples of H-Coal distillate liquid, hydroprocessed to different severity levels, were prepared. These samples, along with hydroprocessed samples of SRC recycle solvent which were prepared last year, were burned in a small scale gas turbine combustor at Westinghouse Research Laboratories in Pittsburgh. The fuel atomization, combustion performance, emissions characteristics and possible erosion or corrosion effects of the combustion products were determined.

Section 3

DISTILLATE COAL LIQUIDS

Of the various products that coal liquefaction processes can potentially produce, the distillate boiling range liquids are the best suited for use in combustion gas turbines. Their physical and chemical properties most closely approach current turbine fuel specifications, providing the best starting materials for turbine fuel manufacture in terms of minimal additional processing required. Sulfur and nitrogen, important parameters that influence emissions of SO_x and NO_x upon combustion, are less concentrated in distillate coal liquids and exist in molecular structures that best afford their removal. Metal contaminants, which may lead to severe corrosion, erosion, and deposition on turbine blades, are significantly less than found in resid containing coal liquids. Higher hydrogen and lower aromatic carbon content, lower viscosity and the virtual absence of materials yielding Conradson Carbon Residue provide the distillate coal liquids with better burning characteristics. Both capital investment and operating costs are lower for upgrading distillate coal liquids to projected turbine fuel quality specifications because of the lower severity hydrotreating required and the lower net chemical hydrogen consumption. The future availability of these materials for use as turbine fuels will be better known as coal liquefaction processes become commercialized.

Work performed during the first year of the current program on upgrading coal liquids for use as gas turbine fuels showed that several distillate coal liquids could be upgraded by hydrotreating over commercially available catalysts to quality levels approaching existing petroleum-derived turbine fuels. Processing severities necessary to convert naphtha-kerosine boiling range SRC light organic liquid and heavy distillates from both the Solvent Refined Coal and H-Coal processes to a turbine fuel containing less than 0.1 wt % nitrogen and 12.0 wt % hydrogen were determined. Required hydrogen consumptions ranged from 500-3300 scf/B depending on the distillate used.

One of the objectives of this work was to extend the knowledge of distillate coal liquid hydroprocessing by investigation of the upgrading of additional distillate boiling range materials and by further characterization of the coal liquids that were previously studied. An SRC wash solvent, having an intermediate boiling range between that of SRC light organic liquid and recycle solvent, was hydroprocessed over a wide range of conditions to obtain data for kinetic modeling. An additional study was made using water addition during the hydroprocessing of SRC recycle solvent to determine the extent of its benefit. A short aging study made while charging an H-Coal (syncrude) distillate at varying severity levels provided a series of fuels of increasing quality levels for use in turbine combustion tests performed by Westinghouse. Combustion of these liquids, and of samples of raw and hydroprocessed recycle solvent, provided information relating the quality of coal liquids to their performance in a turbine combustor and the emissions of NO_x, CO, CO_2, smoke, and unburned hydrocarbons (UHC).

3.1 SRC WASH SOLVENT

The SRC wash solvent, as with all the coal liquids used in this program, was obtained through EPRI. It was produced at the Solvent Refined Coal (SRC) pilot plant at Wilsonville, Alabama using Wyodak (Amax) coal. This is the fourth distillate coal liquid hydroprocessed under this project. Hydroprocessing studies on H-Coal distillate and SRC recycle solvent and light organic liquid were reported in the October 1977 Annual Report (3-1).

Table 3-1 contains source information and the elemental analyses of the SRC wash solvent. This solvent generally has properties intermediate between those reported for the SRC light organic liquid and recycle solvent. Having been derived from a low sulfur coal (Wyodak), however, it does have a lower sulfur content than the other SRC distillates obtained for a high sulfur Illinois No. 6 coal. The wash solvent has the narrowest boiling range (~150°F) of the distillate coal liquids investigated.

The Wyodak wash solvent was charged as received to a standard hydroprocessing unit (CT-199). This process variable study was made using a small pore $CoMo/Al_2O_3$ commercial catalyst (American Cyanamid HDS-1441A). A total of 16 material balances were obtained during a 13-day period. A wide range of operating conditions -- temperatures from 670-780°F, pressures from 500-2000 psig, and 0.5-2.1 LHSV -- were studied to provide data sufficient for kinetic modeling.

Table 3-1

SOURCE AND ANALYSES OF SRC WASH SOLVENT

Feed Coal	Wyodak (Amax)
Wilsonville Sample No.	22227
Wilsonville Sample Location	T-102 (Tray 3)
Date	January 6, 1977
Mobil Identification	77D-895
Elemental Analysis (as received)	
Gravity, °API	14.5
Hydrogen, wt %	8.68
Sulfur, wt %	0.158
Oxygen, wt %	5.9
Nitrogen, wt %	0.36
Aromatic Carbon, %	69
Simulated Distillation, °F @ %	
IBP	263
5	327
10	349
30	389
50	409
70	440
90	461
95	474
FBP	676

Table 3-2

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: WYODAK WASH SOLVENT (77D-895)
CATALYST: HDS-1441 (J-7322)
PROGRAM: 2569   UNIT: CT-199

	CHARGE	*MB-715*	*MB-716*	*MB-717*	*MB-718*	*MB-719*	*MB-720*	*MB-721*
OPERATING CONDITIONS								
TEMPERATURE, °F	-	720	670	767	763	727	677	676
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	1000
LHSV	-	0.58	0.52	0.51	0.94	1.01	1.00	0.95
DAYS ON STREAM	-	1.8	2.8	3.8	4.6	5.1	5.6	6.4
LIQUID PRODUCT PROPERTIES								
GRAVITY, °API	14.5	36.2	34.4	38.3	35.6	34.6	33.1	30.2
HYDROGEN, WT °/°	8.68	12.58	12.14	12.86	12.13	12.44	12.08	11.32
SULFUR, WT °/°	0.158	0.042	0.016	0.010	0.023	0.013	0.019	0.025
NITROGEN, WT °/°	0.360	0.008	0.011	0.008	0.009	0.010	0.028	0.120
OXYGEN, WT °/°	5.9	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.6
K.V. AT 40°C	-	1.66	0.99	1.03	1.21	1.15	1.16	1.18
AROMATIC CARBON, °/°	69	21	-	-	26	-	30	41
HETEROATOM REMOVAL,PCT								
SULFUR	-	74.4	90.2	93.9	86.0	92.1	88.4	84.7
OXYGEN	-	98.4	98.4	98.4	98.4	98.4	98.4	90.2
NITROGEN	-	97.9	97.0	97.8	97.6	97.3	92.5	67.9
HYDROGEN UTILIZATION,SCF/B								
H2 CONSUMED BY C1-C5	-	119	30	139	98	81	29	29
H2 CONSUMED BY C6+	-	2186	1937	2368	1882	2108	1898	1416
H2 CONSUMED BY S	-	5	6	6	5	6	6	5
H2 CONSUMED BY N	-	49	48	49	48	48	46	34
H2 CONSUMED BY O	-	468	468	468	468	468	468	429
H2 CONSUMED TOTAL	-	2826	2488	3029	2501	2711	2446	1912

Table 3-2 (Continued)

	MB-722	MB-723	MB-724	MB-725	MB-726	MB-727	MB-728	MB-729	MB-730
OPERATING CONDITIONS									
TEMPERATURE, •F	681	726	726	776	727	673	677	724	780
PRESSURE, PSIG	500	500	500	500	500	500	500	500	500
LHSV	1.02	0.99	1.10	0.53	0.52	0.52	1.96	2.08	2.03
DAYS ON STREAM	7.1	7.6	8.1	9.0	10.0	11.0	11.6	12.1	12.4
LIQUID PRODUCT PROPERTIES									
GRAVITY, •API	25.7	28.7	28.3	28.3	29.3	27.3	19.8	23.1	26.9
HYDROGEN, WT •/•	10.23	10.67	10.30	10.31	10.63	10.25	9.39	9.52	9.89
SULFUR, WT •/•	0.035	0.023	0.021	0.016	0.007	0.013	0.042	0.039	0.012
NITROGEN, WT •/•	0.260	0.120	0.057	0.018	0.051	0.190	0.306	0.301	0.199
OXYGEN, WT •/•	1.3	0.4	0.1	0.1	0.3	0.9	4.1	2.4	0.8
K.V. AT 40•C	1.33	1.12	1.04	0.99	0.97	1.11	0.92	0.92	0.99
AROMATIC CARBON, •/•	-	-	53	-	48	-	63	-	-
HETEROATOM REMOVAL,PCT									
SULFUR	78.7	86.1	87.5	90.5	95.8	95.2	73.9	76.1	92.8
OXYGEN	78.8	93.5	98.4	98.4	95.2	85.5	31.7	60.7	87.2
NITROGEN	30.6	68.3	85.1	95.3	86.6	49.8	16.4	19.2	47.6
HYDROGEN UTILIZATION,SCF/B									
H2 CONSUMED BY C1-C5	27	66	119	178	107	70	36	23	102
H2 CONSUMED BY C6+	729	927	620	586	862	673	343	324	413
H2 CONSUMED BY S	5	5	6	6	6	6	5	5	6
H2 CONSUMED BY N	15	34	42	47	43	25	8	10	24
H2 CONSUMED BY O	375	445	468	468	452	406	151	288	414
H2 CONSUMED TOTAL	1151	1478	1254	1286	1471	1180	543	649	959

The operating conditions, liquid product properties, and hydrogen consumption data are given in Table 3-2. The complete material balances and distillations of the liquid products are included in Table B-1 in the Appendix.

The products obtained showed a wide range of charge stock upgrading. The hydrogen content in the wash solvent (8.7 wt %) was increased to up to 12.9 wt %. The nitrogen content (0.36 wt % in charge) decreased from 0.3 to <0.01 wt % depending upon hydroprocessing severity. Sulfur removal was greater than 73 wt % at all the severity levels studied. Oxygen levels decreased to levels of <0.1 wt % at all high pressure conditions. Hydrogen consumption ranged from 500-3000 scf/B. A hydrogen consumption of approximately 2400 scf/B will produce a fuel containing 12 wt % hydrogen and <0.02 wt % nitrogen. Table 3-3 shows the effect of pressure on hydroprocessing the wash solvent at 675°F and 1.0 LHSV.

Table 3-3

HYDROPROCESSING OF WYODAK WASH SOLVENT:
EFFECT OF HYDROGEN PRESSURE

Run 2569-	Charge	MB-722	MB-721	MB-720
Hydrogen Pressure, psig	-	500	1000	2000
Liquid Product Properties				
Gravity, °API	14.5	25.7	30.2	33.1
Hydrogen, wt %	8.68	10.23	11.32	12.08
Sulfur, wt %	.158	.035	.025	.019
Nitrogen, wt %	.36	.26	.12	.028
Oxygen, wt %	5.9	1.3	.6	<.1
Heteroatom Removal, %				
Sulfur	-	79	85	88
Oxygen	-	79	90	>98
Nitrogen	-	31	68	92
Hydrogen Utilization				
H_2 Consumed by C_1-C_5	-	27	29	29
H_2 Consumed by C_6^+	-	729	1416	1898
H_2 Consumed by Heteroatoms	-	395	468	520
Total H_2 Consumed	-	1151	1912	2446

Denitrogenation activity has the greatest sensitivity to changes in processing severity while desulfurization is least affected. Nitrogen levels can be significantly reduced by increasing temperature or pressure or by decreasing space velocity. The first 75 wt % of the sulfur is removed immediately under the mildest of conditions tested here; however, the remainder presents considerable difficulty. Increasing temperature or residence time is more effective in removing the remaining sulfur than is increasing pressure. Oxygen is the easiest heteroatom to remove at moderate and high severity levels.

Hydrogen utilization and consumption data for the 16 material balances are included in Figures 3-1 and 3-2. Figure 3-1, showing the breakdown of total hydrogen consumption by C_1-C_5 gas make, total heteroatom removal, and C_6^+ liquid, is characteristic of coal liquid distillates -- relatively fast consumption of hydrogen for heteroatom removal at low severities with a leveling off above 1000 scf/B total hydrogen consumption as essentially all the heteroatoms have been removed; a relatively low, constant gas make regardless of severity; and a steady increase in aromatic saturation with more severe processing conditions. Examination of Figure 3-2, in which the hydrogen consumption by oxygen, nitrogen, and sulfur removal is plotted as a function of severity, shows an apparent delay in the removal of heteroatoms at the onset of hydrogenation. As has been found previously with recycle solvent (3-1, Tables 5-3 and 5-4), an initial saturation of some of the distillate appears necessary before significant heteroatom removal is observed. Again, a leveling off of the hydrogen consumption for specific heteroatom removal is seen with increasing severity. Oxygen removal accounts for about 95% of the hydrogen required for total heteroatom removal.

Several instances occurred during this study where the product hydrogen content decreased upon going to higher temperature at a given space velocity and pressure. This is an indication of aromatic saturation being limited by equilibrium constraints. Figure 3-3 dramatically shows the effect of hydrogen vs. nitrogen selectivity under aromatic saturation equilibrium conditions. The three constant temperature-constant space velocity points (dark symbols) show a decreasing nitrogen content with increasing hydrogen content as the pressure is increased from 500 to 2000 psig. However, if the severity is increased by either increasing the temperature or decreasing the space velocity while operating at a low hydrogen pressure (i.e., 500 psig), the nitrogen is decreased but the hydrogen content of the product remains unchanged (Figure 3-3, dashed line). This means that if fuel

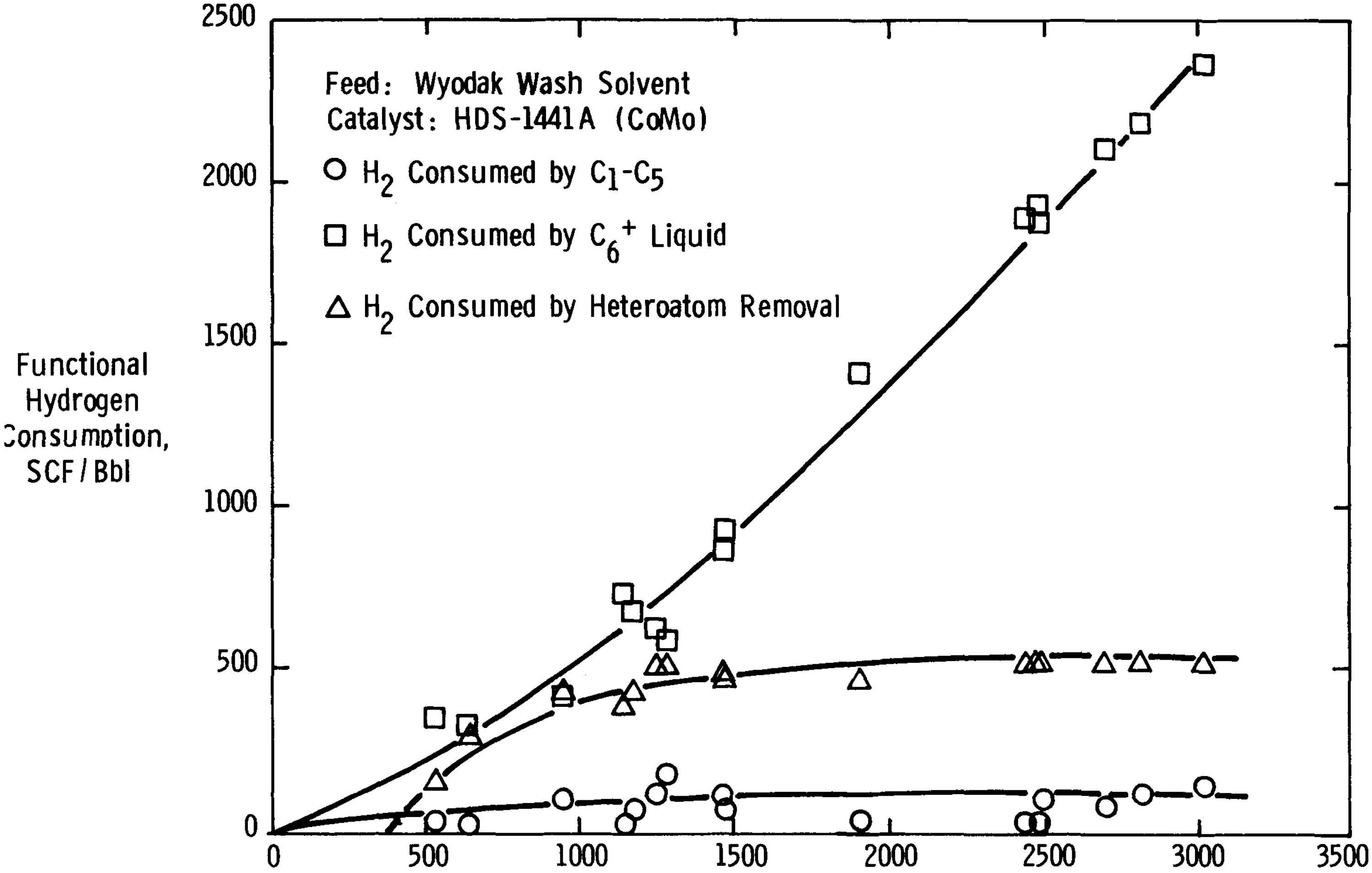

Figure 3-1. Hydrogen Utilization in Hydroprocessing of Wyodak WASH Solvent

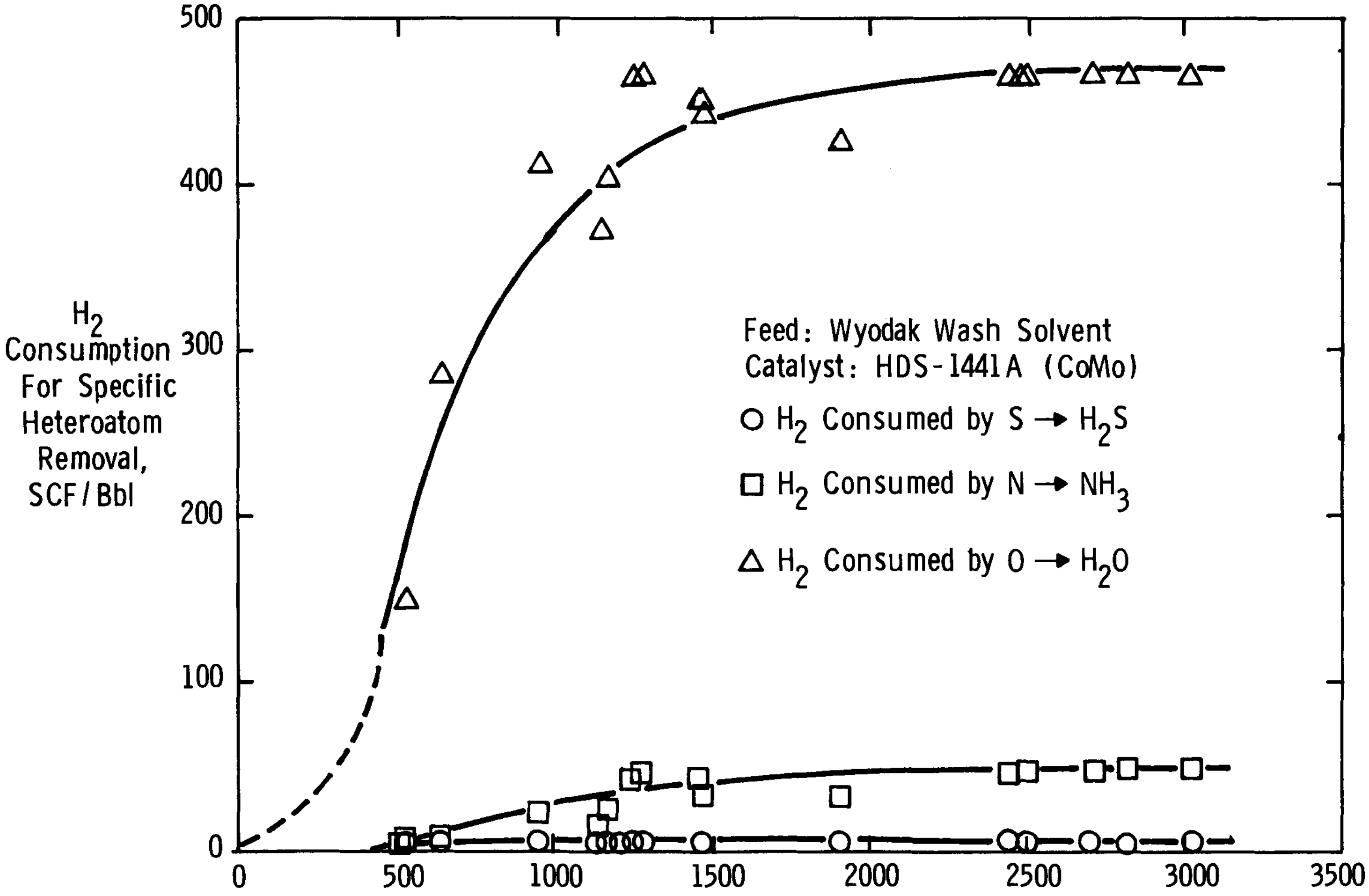

Figure 3-2. Hydrogen Consumption for Heteroatom Removal From Wyodak WASH Solvent

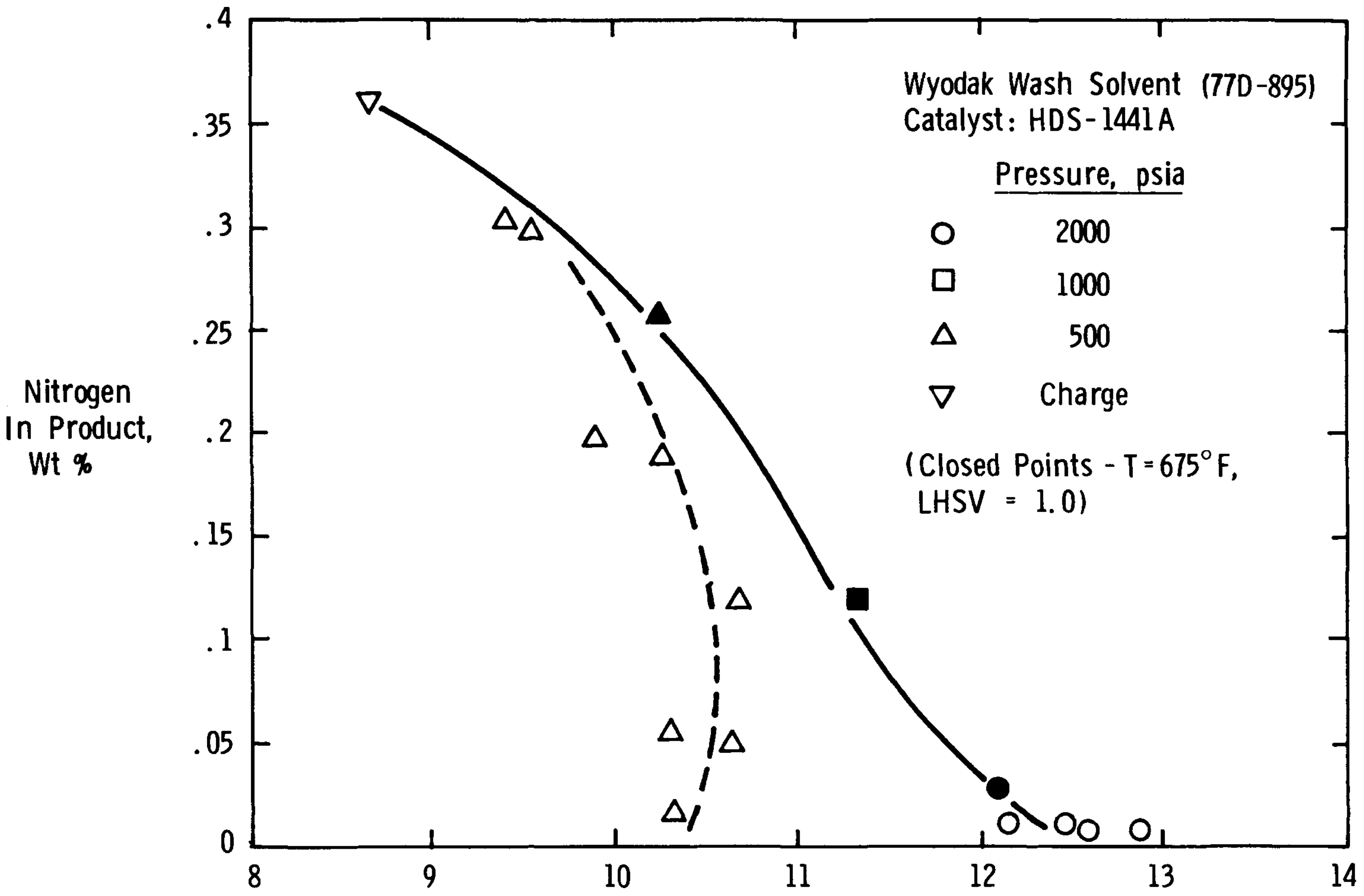

Figure 3-3. Nitrogen Content of Hydroprocessed Wyodak WASH Solvent as a Function of Product Hydrogen Content

bound nitrogen content and not hydrogen is the limiting specification for turbine fuels, a considerable savings in both capital and operating cost is possible by operating at a lower reactor pressure resulting in a significant saving in net chemical hydrogen consumption.

Kinetics for Aromatic Saturation

The wide range of conditions run and the actual observation of hydrogenation equilibrium makes this data ideally suited to fit to the aromatic saturation model developed previously for the H-Coal distillate and SRC recycle solvent. Details of the development of the model are found in October 1977 Annual Report (3-1).

Seven of the samples from the study charging the wash solvent over HDS-1441A catalyst were analyzed for % C_A by C^{13} NMR and are plotted as a function of hydrogen content in Figure 3-4. A second order polynomial fit shows an excellent correlation between % C_A and hydrogen content for this data. This plot was used to predict the % C_A of the remaining hydroprocessed products based on their hydrogen content. This gives a range of % C_A values from 18 to 69 and provides a total of 16 % C_A values obtained at pressures from 500-2000 psig, temperatures from 670-780°F, and space velocities from 0.51-2.08 hr^{-1}.

Comparing with similar % C_A versus hydrogen plots for other hydroprocessed coal liquids, the SRC wash solvent tends to lie parallel between the curves for light organic liquid and recycle solvent H-Coal distillate. SRC recycle solvent and H-Coal distillate, having a similar boiling range, follow a common curve on such a plot. SRC light organic liquid, containing only low boiling, low molecular weight material, has a slightly different curve showing less condensed aromatics with higher total product hydrogen content. The Wyodak SRC wash solvent, boiling between the other two SRC distillates, is found to lie between the two curves and parallels the recycle solvent/H-Coal distillate curve.

As with the fit of the previous data to the saturation model (3-1), the difference between the activation energies of the forward and reverse reactions (i.e., the heat of reaction, ΔH) was set at a value of 27,920, the approximate Btu/lb moles of hydrogen consumed for 2-4 ring aromatics. This leaves four unknown constants in the model to be determined -- the rate constants k_f and k_r, the activation energy E_f, and the Nth order pressure term. The integrated rate equation was then fit to the data using the modified Marquardt routine to determine the constants which minimize the average error. The fit of the data to the model is given in

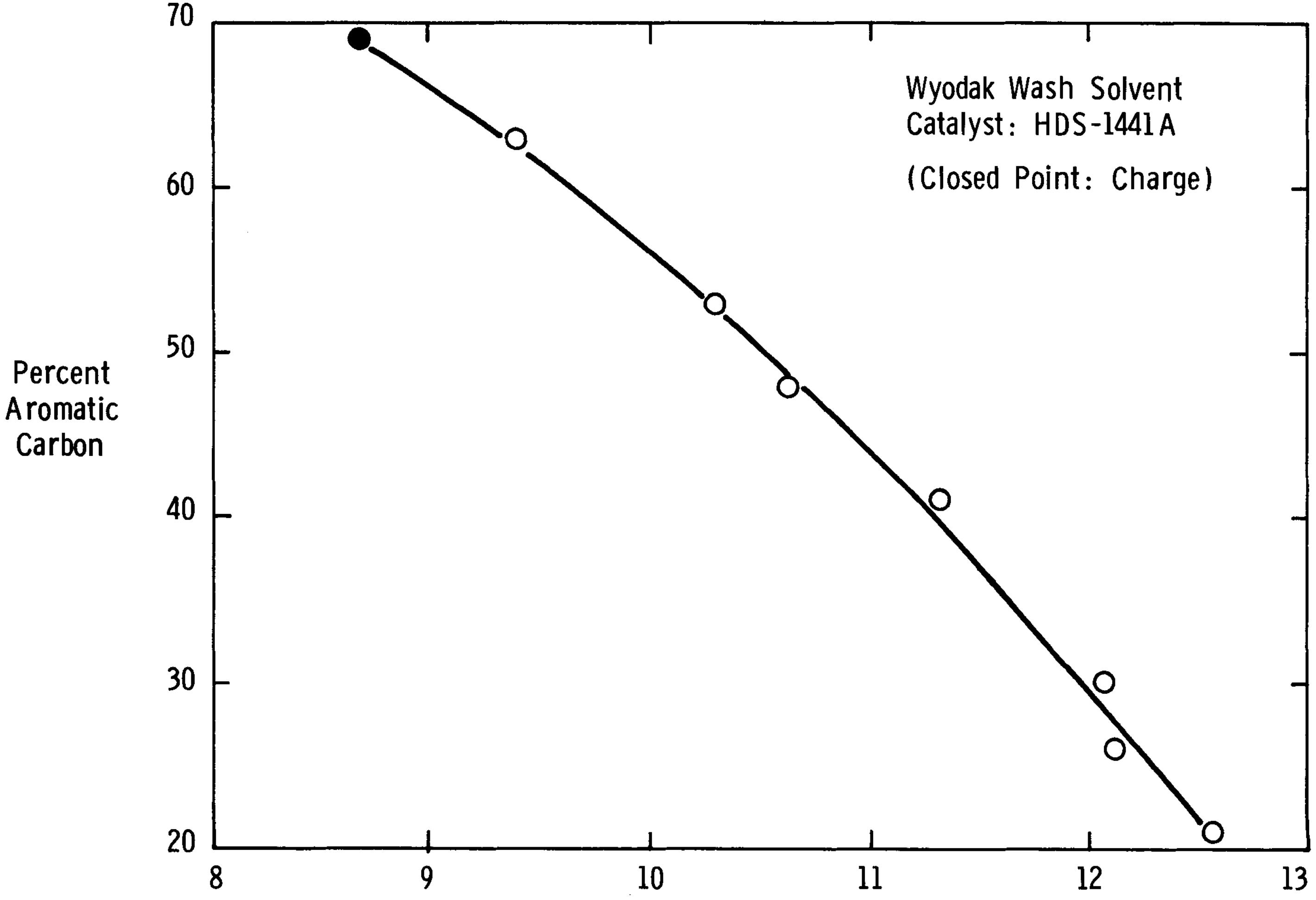

Figure 3-4. Aromatic Carbon Content as a Function of Hydrogen Content for Wyodak WASH Solvent

Figure 3-5. The numerical values of the constants determined are given in Table 3-4, along with the previously reported values for the SRC recycle solvent and H-Coal distillate data. Considering the limited number of data points, the fit to the model is good. The major difference between the two distillate range materials is that the rates for the reactions and the ratio of the forward and reverse reactions are greater for the wash solvent, indicating that saturation is easier for the wash solvent than for the higher boiling distillates.

Figure 3-6 shows the predicted dependence of aromatic content on temperature for three different pressures at 1.0 LHSV for the wash solvent. The figure shows minima in the pressure curves at approximately 750°F, indicating that increased temperatures can lead to less saturation at these conditions. It is also obvious that, at this space velocity, high pressure operation is necessary to achieve a significant decrease in the % C_A. This model is consistent with the observations from the data in Figure 3-3.

Figure 3-7 shows the predicted effect of saturation on temperature for several space velocities at 500 psig. As space velocity is decreased, the minimum in the saturation curves asymptotically approach an equilibrium aromatic saturation curve. At space velocities below about 0.1 LHSV, saturation is equilibrium controlled for temperatures greater than 675°F. The pressure dependence of the aromatic saturation equilibrium is calculated by predicting the percent C_A at a very low space velocity for different pressures, as is shown in Figure 3-8. These curves represent the minimum % C_A (i.e., maximum hydrogen content) that can be achieved at a given temperature and pressure.

These curves confirm the observed data that show aromatic saturation problems occurring throughout our study, at both high and low pressures. In comparison to the other distillates, the wash solvent is more like the H-Coal distillate in that it encounters the dehydrogenation phenomenon much sooner with increasing temperature at a given space velocity and pressure. At low temperatures, aromatic saturation is much more sensitive to changes in pressure but less sensitive to space velocity changes for the wash solvent than for either the recycle solvent or the H-Coal distillate. The wash solvent is more temperature sensitive at aromatic saturation equilibrium conditions than either of the other two distillate materials. In other words, it is generally easier to reach a lower percent aromatic content at lower temperatures when hydroprocessing wash solvent than when processing higher molecular weight SRC recycle solvent or H-Coal distillate.

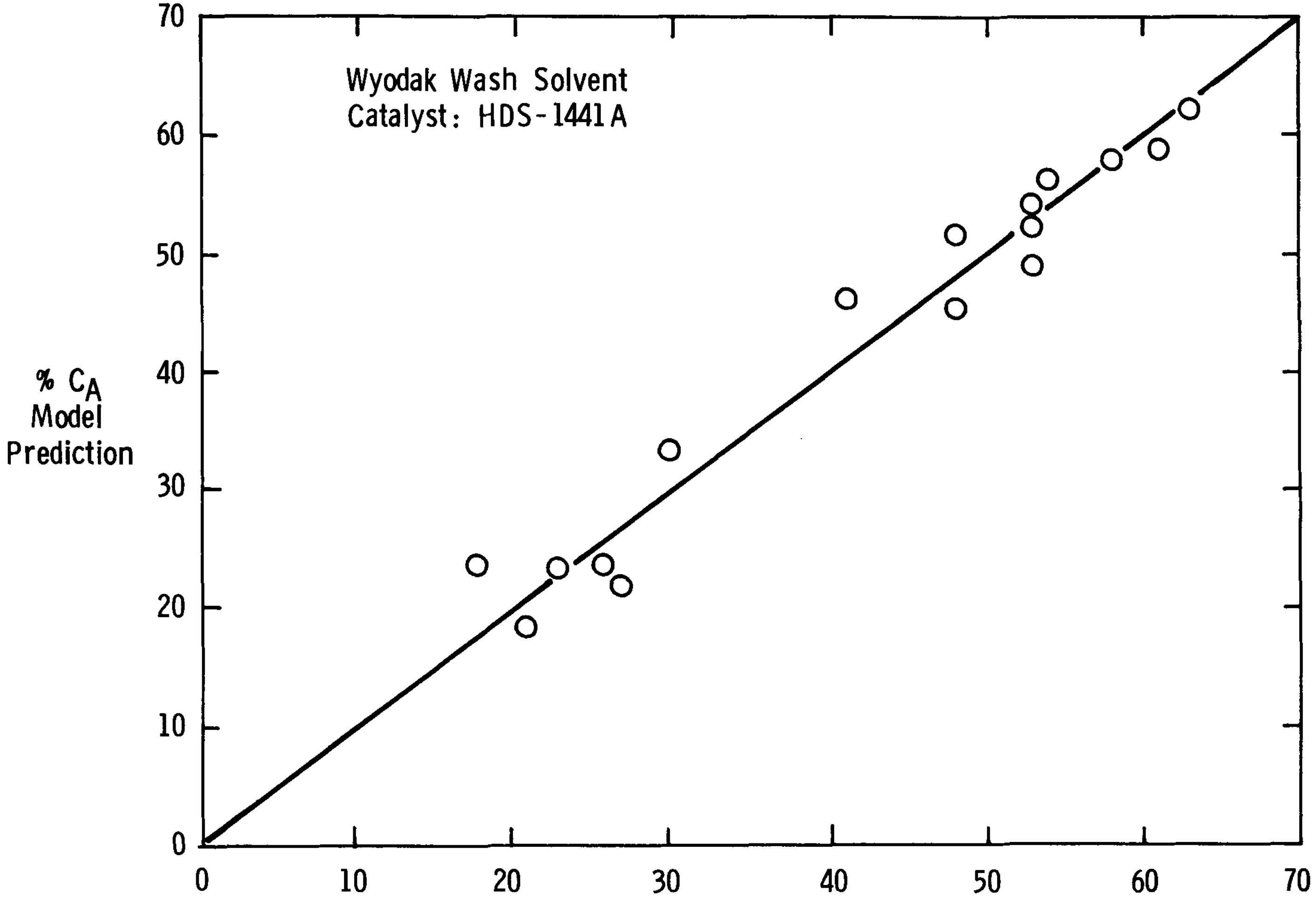

Figure 3-5. Fit of Aromatic Saturation Model to Measured % C_A Values

Table 3-4

KINETIC CONSTANTS FOR AROMATIC SATURATION MODEL(1)

	WYODAK WASH SOLVENT	RECYCLE SOLVENT/ H-COAL DISTILLATE(2)
k_{f_o} (725°F), hr^{-1} $psia^{-1}$	2.34×10^{-3}	0.23×10^{-3}
k_{r_o} (725°F), hr^{-1}	3.62×10^{-1}	0.93×10^{-1}
E_f, Btu/lb mole	40,240	49,320
E_r, Btu/lb mole	68,160	77,240
N	0.87	0.98
Standard Deviation (σ), % C_A	3.2	2.9

---

(1) $$f_A = \frac{k_r}{k_r+k_f\,P^N} + \left[f_o - \frac{k_r}{k_r+k_f\,P^N} \right] e^{-(k_f\,P^N+k_r)/(LHSV)}$$

where:

$$k_f = k_{f_o}\, e^{-E_f/RT}$$

$$k_r = k_{r_o}\, e^{-E_r/RT}$$

$E_r - E_f = 27,920$ Btu/lb mole

(2) EPRI AF-444, Project 361-2 Annual Report, October 1977.

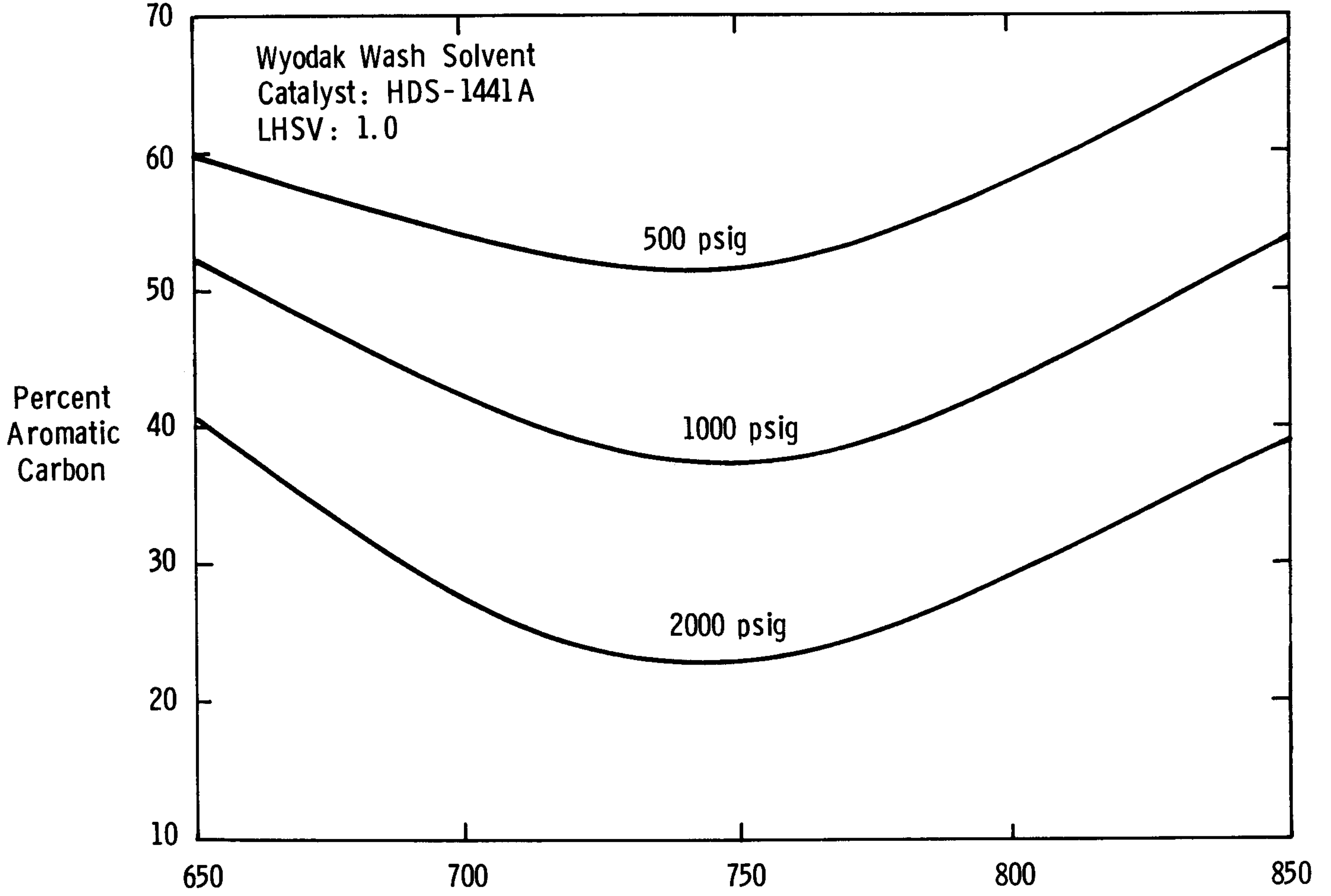

Figure 3-6. Predicted Effect of Pressure on Aromatic Saturation for Wyodak WASH Solvent

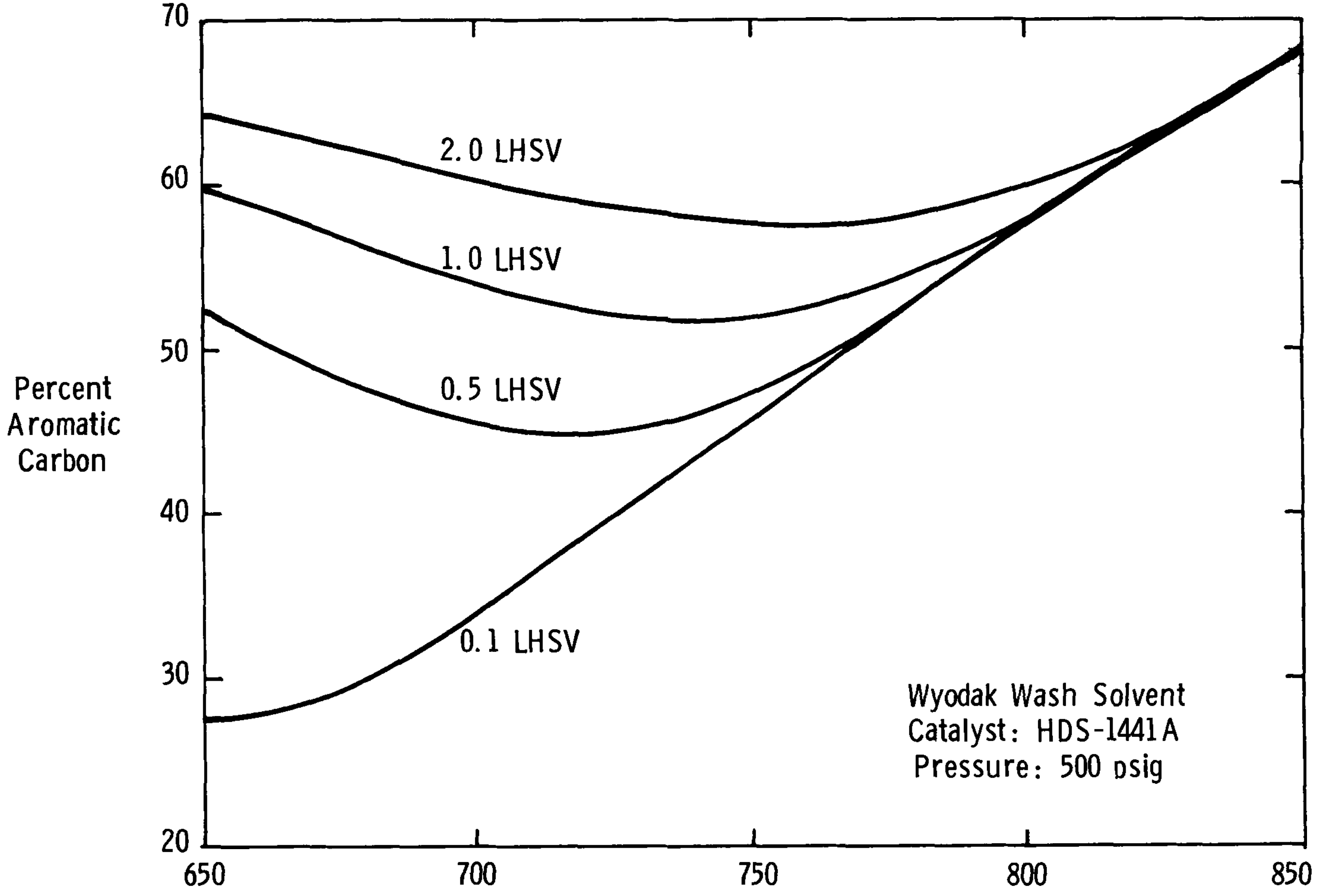

Figure 3-7. Predicted Effect of LHSV on Aromatic Saturation for Wyodak WASH Solvent

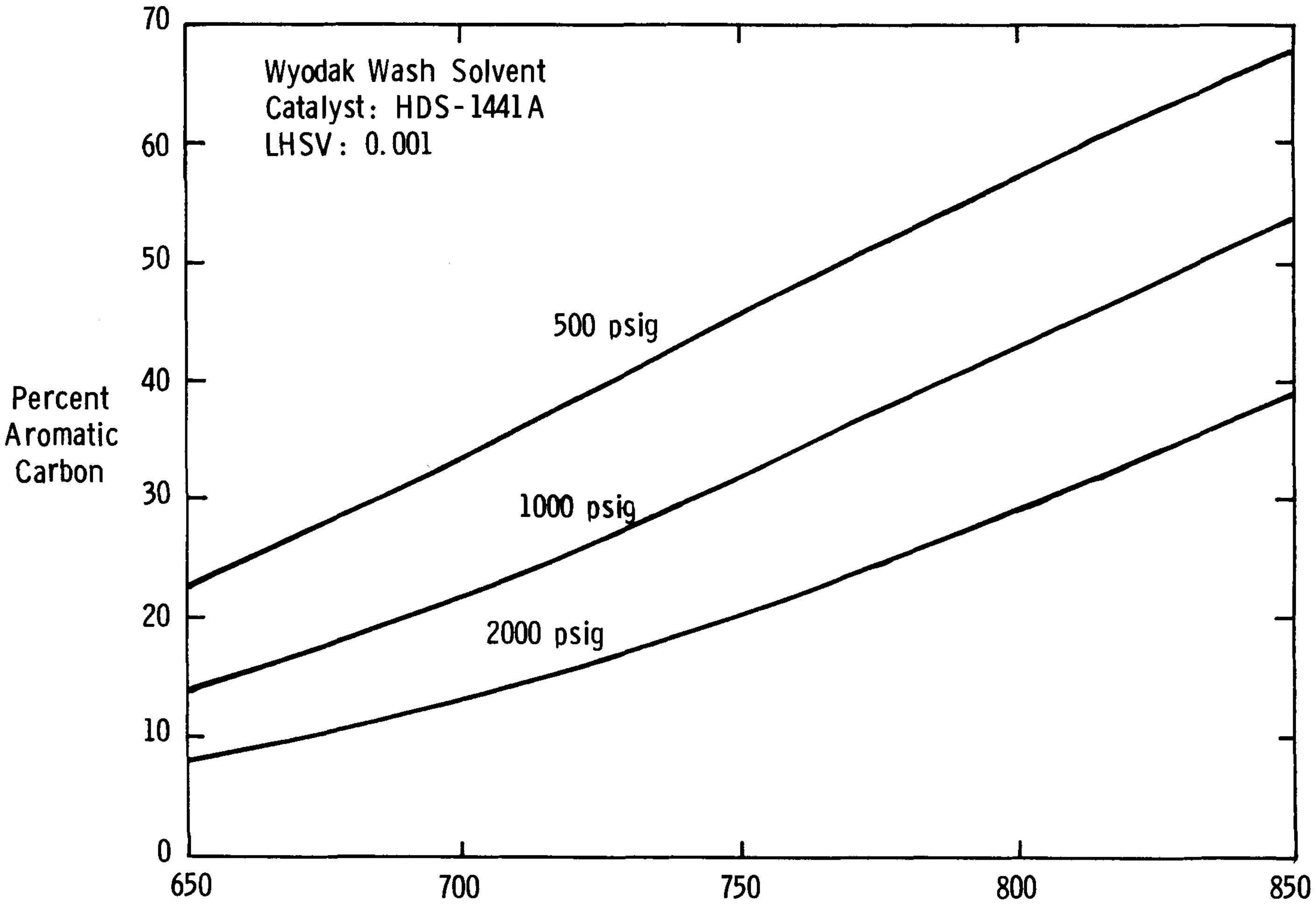

Figure 3-8. Effect of Pressure on Aromatic Saturation Equilibrium for Wyodak WASH Solvent

3.2 SRC RECYCLE SOLVENT

Results for the hydroprocessing of SRC recycle solvent were presented in the October 1977 Annual Report (3-1). Two process variable studies were made using NiMo/Al_2O_3 (American Cyanamid HDS-9A) and CoMo/Al_2O_3 (American Cyanamid HDS-1441A) catalysts to determine any differences in catalyst metals composition or support. The selectivity and activity of the two catalysts are quite similar, with the NiMo catalyst showing only a slight advantage with respect to nitrogen removal. Although existing turbine fuel specifications for nitrogen and hydrogen could be met within the range of process variables studied, considerable hydrogen is consumed. Some of this consumption results from the converting of the oxygen in the SRC recycle solvent to water. This reaction proceeds simultaneously with the removal of nitrogen and sulfur. However, there is no specification on the oxygen content of turbine fuels as there is on the sulfur and nitrogen levels. An attempt was made to minimize hydrogen consumption by inhibiting the deoxygenation reaction by injecting water with the feed to the catalyst.

Properties of the two recycle solvents used during these studies are shown in Table 3-5. The recycle solvent from Illinois No. 6 coal is the same one used during the previous process variable study. During the intervening time between the first and second water addition studies, this solvent was combined with several others and distilled to make a 400°F^+ solvent for use in preparing SRC blends and was therefore not available for the second study. A West Kentucky recycle solvent -- lower boiling material with fewer heteroatoms and a lower gravity and hydrogen content -- was used in the second study. The catalyst for both studies was a CoMo/Al_2O_3 (HDS-1441A).

The first study was made at moderate severity levels to obtain total hydrogen consumptions of about 1000 scf/B. Processing conditions were set at 675°F, 1.0 LHSV, and 2500 psig. The water and recycle solvent were charged to the unit via separate charge systems, which combined into a single feed line at the inlet of the reactor. After an initial line-out period, a material balance without water addition was made, followed by several balances with water to charge volume ratios of 0.16, 0.19, and 0.39, respectively. The final balance was again made without water addition. The operating conditions, liquid product properties, and hydrogen utilization for the balances are presented in Table 3-6. The product yields on charge and the distillations of the liquid products are included in Table B-2.

Table 3-5

ANALYSIS OF SRC RECYCLE SOLVENTS

Feed Coal	Ill. No. 6 Monterey	W. Kentucky 9 + 14
Wilsonville Sample No.	10853	10843(1)
Wilsonville Run No.	46A	-
Pressure, psig	1721	-
Temperature, °F	860	-
Feed Rate, lbs/hr	339	-
Mobil Identification	75D-3019	76D-3007
Elemental Analysis (as received)		
Gravity, °API	5.5	3.9
Hydrogen, wt %	7.71	7.14
Sulfur, wt %	0.40	0.30
Oxygen, wt %	3.77	2.60
Nitrogen, wt %	0.62	0.52
Simulated Distillation, °F @ %		
IBP	301	263
5	336	327
10	360	349
30	400	389
50	441	409
70	509	440
90	614	461
95	655	474
FBP	830	676

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(1) No other information available on this sample.

Table 3-6

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: SRC RECYCLE SOLVENT (75D-3019) W/H2O ADDITION
CATALYST: HDS-1441A (J-7278)
PROGRAM: 2335 UNIT: CT-146

	CHARGE	*MB-753*	*MB-754*	*MB-755*	*MB-756*	*MB-757*
OPERATING CONDITIONS						
TEMPERATURE, °F	-	676	675	675	672	675
PRESSURE, PSIG	-	2500	2500	2500	2500	2500
LHSV	-	1.04	1.02	1.00	1.00	0.98
DAYS ON STREAM	-	1.4	2.6	3.6	4.6	5.9
H2O/H2, MOLE RATIO	-	-	0.16	0.28	0.58	-
H2O/SOL., VOL. RATIO	-	-	0.11	0.19	0.39	-
LIQUID PRODUCT PROPERTIES						
GRAVITY, °API	5.5	13.5	13.3	13.3	12.5	13.2
HYDROGEN, WT °/°	7.71	9.12	9.05	9.04	8.87	8.99
SULFUR, WT °/°	0.400	0.043	0.037	0.037	0.044	0.046
NITROGEN, WT °/°	0.62	0.44	0.38	0.35	0.34	0.46
OXYGEN, WT °/°	3.77	1.70	2.20	2.20	2.10	2.10
HETEROATOM REMOVAL,PCT						
SULFUR	-	89.7	91.1	91.1	89.5	88.9
OXYGEN	-	59.2	47.1	47.1	49.7	49.5
NITROGEN	-	26.7	36.5	41.5	43.4	23.1
HYDROGEN UTILIZATION,SCF/B						
H2 CONSUMED BY C1-C5	-	16	25	22	26	17
H2 CONSUMED BY C6+	-	889	863	854	719	821
H2 CONSUMED BY S	-	16	16	16	16	16
H2 CONSUMED BY N	-	23	32	36	38	20
H2 CONSUMED BY O	-	209	166	166	175	174
H2 CONSUMED TOTAL	-	1153	1102	1094	974	1048

These data show that oxygen removal decreased slightly when water was added; however, there was a much greater effect on the nitrogen removal. The percent nitrogen removal increased from 27 wt % with no water to 36, 42, and 43 wt % with increasing water addition. When the water was removed, the nitrogen conversion again dropped to 23 wt %. This increased nitrogen removal with water addition apparently came without an increase in hydrogen consumption. These results appeared to offer a means of altering the selectivity between denitrogenation and hydrogenation, at moderate severity levels.

A second water addition study was made charging the West Kentucky recycle over HDS-1441A catalyst. The purpose of this study was to determine if the beneficial effect of water addition is observed at higher severities necessary to reduce the nitrogen content of the product to less than 0.1 wt %.

Conditions for achieving 0.1 wt % nitrogen in the feed were estimated from a previous run with recycle solvent (3-1, Table 5-4). Operating at the same space velocity (1.0 LHSV) and pressure (2500 psig) as the first water addition study, a temperature 50°F higher (i.e., 725 versus 675°F) would be sufficient to reduce the nitrogen content to the desired level while consuming ~ 2200 scf/B without water addition. Because the nitrogen content of the second recycle solvent was lower than the first, the resulting product nitrogen was less than 0.1 wt %. The sequence of balances were made similar to the first study. After running the first balance without water addition, three balances were made with water/recycle solvent volume ratios of 0.14, 0.22, and 0.39, respectively. After the fourth balance, the low temperature-high pressure separator sight glass gasket blew, resulting in loss of pressure causing an emergency shutdown of the unit. Although a recheck on the balance without water addition was not possible, the resultant balances provided some useful information.

Table 3-7 contains the operating conditions, liquid product properties, and hydrogen utilization data for the four balances completed during the second water addition study. Table B-3 presents the product yields on charge and the distillation data on the liquid product.

In contrast to the first study at lower severity (Table 3-6), adding water did not increase the percent nitrogen removal although it did inhibit the deoxygenation reaction. Total hydrogen consumption and hydrogen content of the liquid product

Table 3-7

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: SRC RECYCLE SOLVENT (76D-3007) W/H20 ADDITION
CATALYST: HDS-1441A (J-7278)
PROGRAM: 2562 UNIT: CT-146

	CHARGE	MB-860	MB-861	MB-862	MB-863
OPERATING CONDITIONS					
TEMPERATURE, °F	-	722	721	715	727
PRESSURE, PSIG	-	2500	2500	2500	2500
LHSV	-	1.04	1.00	1.00	0.97
DAYS ON STREAM	-	2.2	3.7	4.4	5.2
H2O/H2, MOLE RATIO	-	-	0.20	0.32	0.54
H2O/SOL., VOL. RATIO	-	-	0.14	0.22	0.39
LIQUID PRODUCT PROPERTIES					
GRAVITY, °API	3.9	16.8	14.3	13.5	13.7
HYDROGEN, WT °/°	7.14	9.89	9.35	9.24	9.28
SULFUR, WT °/°	0.300	0.013	0.020	0.034	0.038
NITROGEN, WT °/°	0.52	0.06	0.10	0.10	0.09
OXYGEN, WT °/°	2.60	0.50	0.70	0.80	1.10
CCR, WT °/°	-	0.03	0.03	0.01	0.01
K.V. AT 40°C	-	2.26	2.67	2.64	2.67
HETEROATOM REMOVAL, PCT					
SULFUR	-	95.7	93.4	88.7	87.4
OXYGEN	-	80.9	73.2	69.4	57.8
NITROGEN	-	88.5	80.9	80.9	82.7
HYDROGEN UTILIZATION, SCF/B					
H2 CONSUMED BY C1-C5	-	52	32	29	31
H2 CONSUMED BY C6+	-	1856	1477	1403	1456
H2 CONSUMED BY S	-	12	12	12	11
H2 CONSUMED BY N	-	68	63	63	64
H2 CONSUMED BY O	-	183	165	157	131
H2 CONSUMED TOTAL	-	2172	1749	1663	1693

are reduced in the presence of steam. The effect of the steam at this higher severity (i.e., higher temperature) appears to be an overall decrease in catalyst activity for hydrogenation, denitrogenation, and deoxygenation. Whether this effect is reversible when the water is removed could not be checked as the run was terminated when the sight glass ruptured.

3.3 H-COAL DISTILLATE

H-Coal distillates produced in both the fuel oil and syncrude modes of operation were prepared and evaluated during the first year of this contract. The H-Coal distillate from the fuel oil mode was processed over three commercial catalysts (HDS-1441A, Ketjen 153S, and HDS-20) while the syncrude mode distillate was processed using only the HDS-1441A catalyst. At the same operating severities, the Ketjen 153S ($NiMo/Al_2O_3$) gives 100-260 scf/B more hydrogen consumption and consistently lower nitrogen levels in the liquid product than HDS-1441A ($CoMo/Al_2O_3$). Over the same catalyst (HDS-1441A) both distillates are upgraded to approximately the same extent. The fuel oil mode distillate charge, which has slightly more hydrogen (~0.3 wt %) and less oxygen (~0.3 wt %), yields liquid products with more hydrogen and less heteroatoms at a given severity than the syncrude mode distillate. Hydrogen consumptions are approximately 100 scf/B greater when processing the lower quality syncrude distillate.

An additional study was made charging the H-Coal (Illinois No. 6 Burning Star) distillate from the syncrude mode of operation over the Ketjen 153S $NiMo/Al_2O_3$. The 39-day run, made in a standard hydroprocessing unit, was the only long-term aging study made with coal liquid distillate materials and presented no operational difficulties. The dual purpose of the study was to determine the catalyst deactivation rate and to produce sufficient quantities of hydroprocessed H-Coal distillate at several quality levels for turbine combustion tests. The Ketjen catalyst was used due to its better denitrogenation and hydrogenation activity. The properties of the H-Coal distillate used in this study are shown in Table 3-8.

This distillate was prepared by blending atmospheric overhead and bottoms products from the HRI PDU. Complete blend information was given in last year's report (3-1).

Thirteen material balances were completed during the one-month aging study in which two, three-gallon hydroprocessed samples were prepared for turbine combustion tests. Four hydroprocessing severities (i.e., space velocities of 0.5, 1.0, and 3.0 at 1500 psi and of 0.5 LHSV at 2500 psi) were run at a constant temperature of 700°F. Three of the balances at an intermediate severity were made at increased

Table 3-8

SOURCE AND ANALYSIS OF H-COAL (SYNCRUDE) DISTILLATE

Feed Coal	Ill. No. 6 Burning Star
Source Information	(3.1, Section 4.1)
Mobil Identification	76D-3521
Elemental Analysis	
Gravity, °API	17.1
Hydrogen, wt %	9.8
Sulfur, wt %	.13
Oxygen, wt %	1.5
Nitrogen, wt %	.38
Trace Metals (ppm)	
Si	-
Mg	-
Pb	-
Fe	12.3
Al	-
Ni	-
Ti	-
Ca	-
Na	-
K	-
V	0.1
Carbon	-
KV (100°F), cs	23.44
KV (210°F), cs	.98
CCR, wt %	.17
Basic Nitrogen, wt %	.32
Ash, ppm	<100
Molecular Weight	174
Simulated Distillation, °F 0%	
IBP	270
5	328
10	346
30	396
50	433
70	489
90	590
95	665
FBP	942

- = (a) - Analysis not determined.

temperature levels during the latter part of the study to obtain some necessary aging data. The operating conditions, hydrogen utilization and liquid product properties are shown in Table 3-9. The product yields on charge and the distillations of the liquid products are included in Appendix Table B-3. A comparison of balances at 1500 psig made during the first week of the study and after a month of processing is shown below.

Aging of H-Coal (Syn) Distillate

	MB-641	MB-642	MB-649	MB-650
Operating Conditions				
Days On-Stream	3.3	5.9	30.4	30.9
Temperature, °F	701	704	705	717
LHSV	1.44	1.43	1.49	1.51
Liquid Product Properties				
Gravity, °API	23.4	23.9	23.2	23.5
Hydrogen, Wt %	10.88	10.83	10.80	10.98
Sulfur, Wt %	<.001	<.001	<.002	.004
Nitrogen, Wt %	.073	.062	.079	.065
Oxygen, Wt %	.20	.20	.20	.40
CCR, Wt %	.14	.05	.09	.03
H_2 Consumed by C_1-C_5	18	42	39	48
H_2 Consumed by C_6^+	613	567	545	677
H_2 Consumed Total	781	760	733	861

These data show that aging of the catalyst is slight (i.e., a maximum aging rate of about 5-10°F for the first month).

Figure 3-9 shows a breakdown of total hydrogen consumption by C_1-C_5 gas make, total heteroatom removal, and C_6^+ liquid as a function of severity (i.e., total hydrogen consumption) for the data from both syncrude distillate studies. The hydrogen consumption to produce NH_3, H_2S, and H_2O from the nitrogen sulfur and oxygen is shown in Figure 3-10. The results from the HDS-1441A study are very similar to those presented previously for the regular H-Coal distillate (3-1, Figures 5-7 and 5-8). The one noted difference is the more gradual increase in conversion of nitrogen to NH_3 as a function of severity (Figure 3-10), indicating a more uniform removal of nitrogen (i.e., lower order reaction) with increasing severity.

Table 3-9

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: H-COAL DISTILLATE (HYNCRUDE) (76D-3521)
CATALYST: KETJEN 153-S (J-7709)
PROGRAM: 2502 UNIT: CT-199

	CHARGE	MB-641	MB-642	MB-643	MB-644	MB-645	MB-646
OPERATING CONDITIONS							
TEMPERATURE, °F	-	701	704	698	700	698	702
PRESSURE, PSIG	-	1500	1500	1500	1500	1500	2500
LHSV	-	1.44	1.43	0.53	0.51	0.48	0.47
DAYS ON STREAM	-	3.3	5.9	7.9	10.9	17.4	19.4
LIQUID PRODUCT PROPERTIES							
GRAVITY, °API	17.1	23.4	23.9	25.8	25.8	25.7	28.5
HYDROGEN, WT °/°	9.80	10.88	10.83	11.41	11.40	11.50	12.25
SULFUR, WT °/°	0.130	<0.001	<0.001	<0.001	0.002	<0.002	0.007
NITROGEN, WT °/°	0.380	0.073	0.062	0.006	0.005	0.004	0.002
OXYGEN, WT °/°	1.5	0.2	0.2	<0.1	<0.1	0.2	0.1
CCR, WT °/°	0.11	0.14	0.05	0.10	0.03	0.07	0.02
K.V. AT 40°C	23.44	1.99	1.81	1.76	1.87	1.75	-
AROMATIC CARBON, °/°	55	-	38	-	33	-	-
HETEROATOM REMOVAL, PCT							
SULFUR	-	99.2	99.3	99.2	98.5	98.5	94.7
OXYGEN	-	86.8	86.8	93.4	93.4	86.7	93.3
NITROGEN	-	80.9	83.8	98.3	98.8	99.0	99.5
HYDROGEN UTILIZATION, SCF/B							
H2 CONSUMED BY C1-C5	-	18	42	45	46	45	57
H2 CONSUMED BY C6+	-	613	567	956	947	1027	1546
H2 CONSUMED BY S	-	5	5	5	5	5	5
H2 CONSUMED BY N	-	42	43	51	51	51	51
H2 CONSUMED BY O	-	103	103	111	111	103	111
H2 CONSUMED TOTAL	-	781	760	1168	1160	1232	1770

Table 3-9 (Continued)

	MB-647	MB-648	MB-649	MB-650	MB-651	MB-652	MB-653
OPERATING CONDITIONS							
TEMPERATURE, °F	702	703	705	717	732	697	695
PRESSURE, PSIG	2500	2500	1500	1500	1500	1500	1500
LHSV	0.50	0.54	1.49	1.51	1.45	2.93	2.93
DAYS ON STREAM	22.4	29.5	30.4	30.9	31.4	32.1	33.6
LIQUID PRODUCT PROPERTIES							
GRAVITY, °API	28.0	27.5	23.2	23.5	23.9	21.3	20.3
HYDROGEN, WT °/°	12.20	12.01	10.80	10.98	11.12	10.60	10.36
SULFUR, WT °/°	<0.002	<0.002	<0.002	0.004	0.003	0.007	<0.002
NITROGEN, WT °/°	0.002	0.005	0.079	0.065	0.044	0.230	0.260
OXYGEN, WT °/°	<0.1	<0.1	0.2	0.4	0.3	0.7	0.8
CCR, WT °/°	0.02	0.01	0.09	0.03	<0.01	0.02	0.01
K.V. AT 40°C	1.77	2.01	1.90	1.89	1.76	2.15	2.06
AROMATIC CARBON, °/°	20	-	-	-	-	43	-
HETEROATOM REMOVAL, PCT							
SULFUR	98.5	98.5	98.5	97.0	97.7	94.7	98.5
OXYGEN	93.3	93.3	86.8	73.5	80.1	53.5	46.9
NITROGEN	99.3	98.6	79.4	83.0	88.5	39.7	31.9
HYDROGEN UTILIZATION, SCF/B							
H2 CONSUMED BY C1-C5	42	39	39	48	48	9	4
H2 CONSUMED BY C6+	1520	1384	545	677	768	474	319
H2 CONSUMED BY S	5	5	5	5	5	5	5
H2 CONSUMED BY N	51	51	41	43	46	20	16
H2 CONSUMED BY O	111	111	103	87	95	64	56
H2 CONSUMED TOTAL	1729	1590	733	861	962	572	400

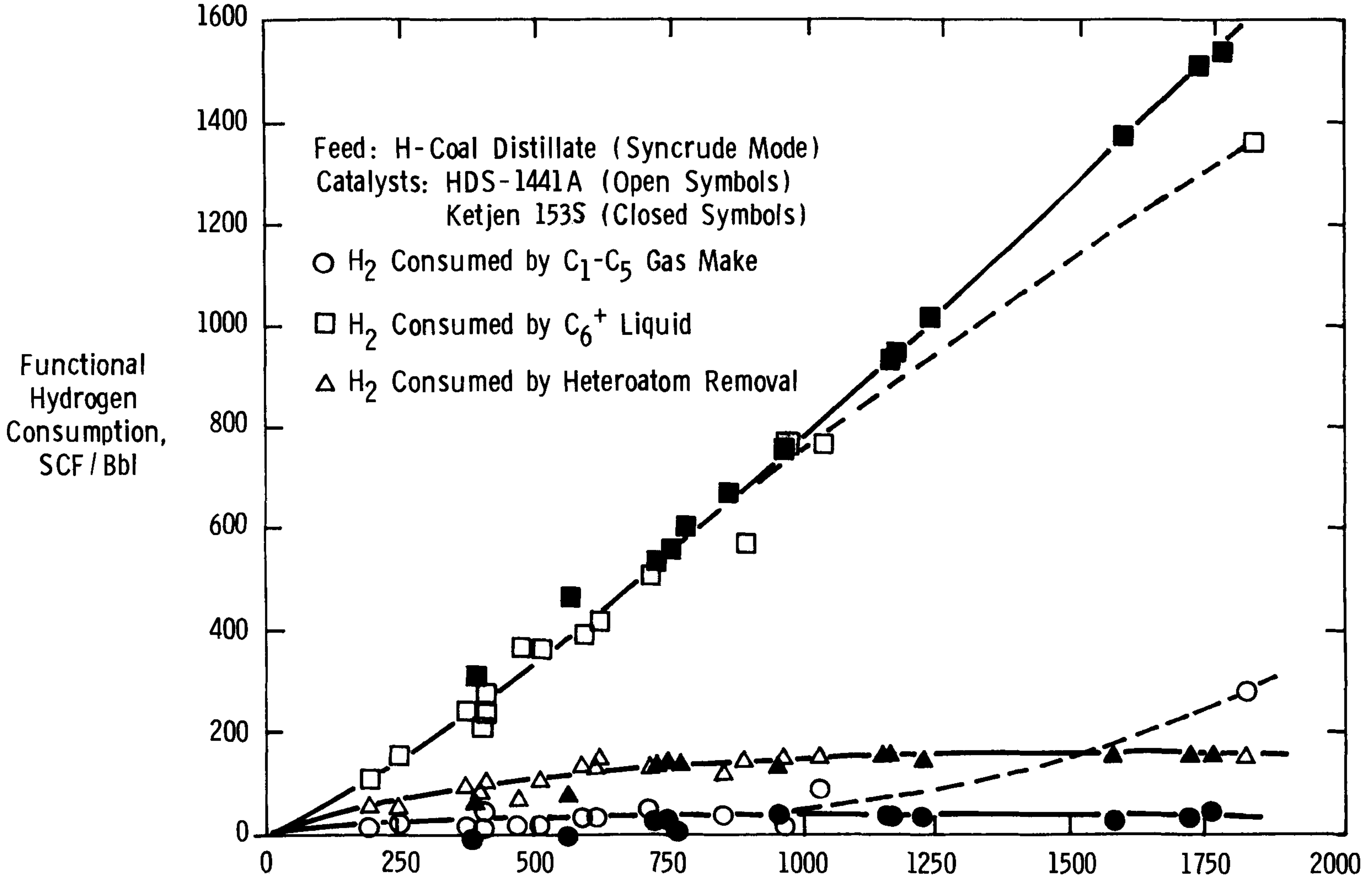

Figure 3-9. Hydrogen Utilization in Hydroprocessing of H-Coal (Syncrude Mode) Distillate

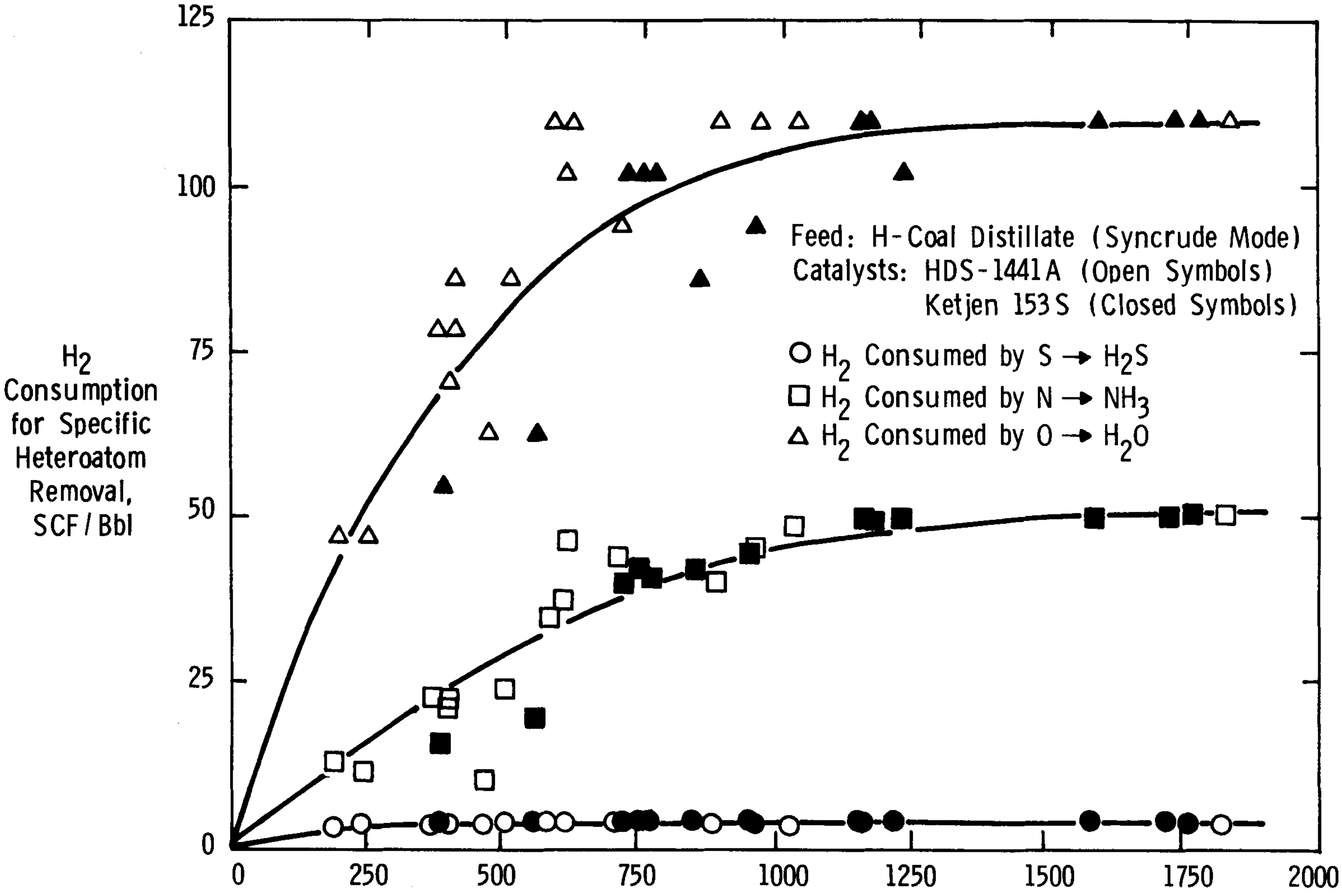

Figure 3-10. Hydrogen Consumption for Heteroatom Removal From H-Coal (Syncrude Mode) Distillate

Comparing the effect of catalyst on hydrogen utilization for the H-Coal syncrude distillate runs, the trends are generally identical at low processing severities. The major difference in the hydrogen utilization for the two catalysts is the lower hydrogen consumption by C_1-C_5 gas make with Ketjen 153S at the highest severity levels. The high C_1-C_5 gas make with HDS-1441A at high severities has consistently been observed with this catalyst when charging H-Coal distillates and SRC recycle solvents. At similar processing conditions, the Ketjen 153S shows slightly higher C_6^+ hydrogen consumption than obtained with HDS-1441A. This is as expected since the Ketjen 153S catalyst has been previously shown to have a higher hydrogenation activity.

Kinetic Model: Heteroatom Removal

Analysis of the data from the two H-Coal syncrude distillate catalyst studies in terms of simple kinetic reaction models was performed. The second order rate expression described in the first report (3-1) was applied to the oxygen, nitrogen, and sulfur removal data.

The fit of the second order model to the data is marginally acceptable for oxygen and nitrogen. The sulfur data gave a very good fit considering the decreased accuracy of the sulfur analysis at the low concentrations obtained (<0.03 wt %). The sulfur data for the Ketjen 153S study were not fit to the model since all balances had <0.01 wt % sulfur remaining. The fits for the aging study were generally not as good due to the fewer data available and the lack of a wide variety of operating severities.

The kinetic constants and average deviations determined by the modified Marquardt fitting routine are given in Table 3-10. The kinetic constants for the syncrude distillate run are somewhat different than those for the fuel oil mode distillate runs with the same catalyst (HDS-1441A) (3-1, Table 6-1). Activation energies and pre-exponential factors are less for the syncrude run, resulting in overall reaction rates that tend to be slightly lower. Use of the Ketjen 153S for hydroprocessing the H-Coal syncrude distillate gives larger rate constants for nitrogen indicating its better activity for nitrogen removal.

The nitrogen and oxygen data from the H-Coal syncrude distillate runs were also modeled using a first order rate equation. The kinetic constants and average deviations for the first order reaction model are included in Table 3-11. This model predicts the nitrogen and oxygen data significantly better than the second

Table 3-10

CONSTANTS FOR KINETIC MODEL FOR HETEROATOM REMOVAL

Second Order Rate Equation*

Feed: H-Coal Distillate (Syncrude)

Catalyst: HDS-1441A

	K	E, Btu/ # MOLE	n	AVG. DEVIATION WT %
Nitrogen	1.00×10^6	50,930	1.33	0.029
Oxygen	1.16×10^7	54,780	1.15	0.092
Sulfur	1.13×10^6	36,640	0.90	0.008

Catalyst: Ketjen 153S

	K	E, Btu/ # MOLE	n	AVG. DEVIATION WT %
Nitrogen	9.00×10^9	72,840	1.5	0.038
Oxygen	3.58×10^6	46,025	0.82	0.101

$$* \quad \frac{dC_i}{dt} = - k\, e^{-E/RT}\, C_i^{\,2}\, P^n$$

Table 3-11

CONSTANTS FOR KINETIC MODEL FOR HETEROATOM REMOVAL

First Order Rate Equation**

	K	E, Btu/ # MOLE	n	AVG. DEVIATION WT %
Nitrogen	1.56×10^4	40,320	1.08	0.012
Oxygen	7.29×10^4	39,890	0.93	0.088

Catalyst: Ketjen 153S

	K	E, Btu/ # MOLE	n	AVG. DEVIATION WT %
Nitrogen	9.00×10^9	66,980	0.92	0.019
Oxygen	1.00×10^4	33,300	0.82	0.110

$$** \quad \frac{dC_i}{dt} = - k\, e^{-E/RT}\, C_i\, P^n$$

model. This suggests a difference in the type of nitrogen and oxygen compounds present in the syncrude distillate (e.g., prepared at high severities in the H-Coal liquefaction step) compared to the fuel oil distillate (prepared at lower severities) which gave a better fit to second order kinetics for nitrogen and oxygen removal.

The relative activity of the two catalysts and the two types of H-Coal distillate for heteroatom removal can be compared by evaluating the fraction of nitrogen, oxygen, and sulfur converted at given reactor conditions. Table 3-12 below shows these comparisons for a relatively low severity of operation (3.0 LHSV, 700°F, 1500 psig) and for a higher reaction severity (1.0 LHSV, 725°F, 2000 psig).

Table 3-12

FRACTION OF SULFUR, NITROGEN, AND OXYGEN CONVERTED FROM H-COAL DISTILLATE USING BEST FIT CONSTANTS*

Low Severity: 3.0 LHSV, 700°F, 1500 psig

H-Coal Distillate	Fuel Oil Mode		Syncrude Mode			
Catalyst	HDS-1441A		HDS-1441A		Ketjen 153S	
Fraction Converted		wt %		wt %		wt%
Nitrogen*	.40	.23	.30	.27	.45	.21
Oxygen*	.53	.6	.49	.8	.51	.7
Sulfur	.79	.023	.82	.028	-	-

High Severity: 1.0 LHSV, 725°F, 2000 psig

H-Coal Distillate	Fuel Oil Mode		Syncrude Mode			
Catalyst	HDS-1441A		HDS-1441A		Ketjen 153S	
Fraction Converted		wt %		wt %		wt %
Nitrogen*	.83	.06	.88	.05	.98	.01
Oxygen*	<.9	<.1	<.9	<.1	<.9	<.1
Sulfur	.96	.004	.96	.005	-	-

---

*First order kinetic constants used for nitrogen and oxygen for the syncrude mode runs.

No significant differences in sulfur and oxygen removal are seen between both charge stocks, regardless of severity. At low severities, a lower percent conversion of nitrogen is predicted for the syncrude distillate, while the difference at high severities is within the accuracy of the data. On comparing the different catalysts, both show essentially the same deoxygenation activity while the Ketjen 153S shows a higher denitrogenation activity at all severity levels. The Ketjen catalyst also shows a lower gas make than the HDS-1441A. This is consistent with what has been observed previously with these two catalysts.

Preparation of Combustor Fuels

In addition to the catalyst aging data obtained, the hydroprocessing study charging H-Coal syncrude distillate over Ketjen 153S served to produce two upgraded H-Coal distillate samples at different severity levels for evaluation in Westinghouse combustor tests. These two samples were prepared by combining the products from the two lowest severity conditions and the products from the two highest severity conditions. Both the combined products and the raw charge were distilled to remove material boiling below 365°F. The 365°F$^+$ coal liquids more closely match the front-end volatility of the petroleum No. 2 fuel oil used as the base fuel in the combustion tests.

A list of the chemical and physical properties of the H-Coal test fuels is shown in Table 3-13. In general, the H-Coal test fuels are less aromatic (55-27% C_A) than the SRC recycle solvent samples (74-34% C_A) used in the combustion tests. The fuel bound nitrogen ranges from 0.04-0.39 wt %. Compatibility of the raw and hydroprocessed H-Coal products with a petroleum derived No. 6 fuel oil was determined by the method outlined in the first report (3-1, Section 3-3). The results are given below:

COMPATIBILITY OF RAW AND HYDROPROCESSED H-COAL DISTILLATE
WITH NO. 6 PETROLEUM FUEL

50/50 WT BLEND OF NO. 6 F.O. WITH:	INCOMPATIBILITY SEDIMENT, VOL %
Raw H-Coal Dist. (76D-3521)	0.15
Mild HDP H-Coal Dist. (J8828)	Trace
Severe HDP H-Coal Dist. (J8830)	Trace

Table 3-13

PROPERTIES OF RAW AND TWO HYDROPROCESSED SAMPLES OF H-COAL DISTILLATE (SYNCRUDE) FOR COMBUSTION TESTING (EPRI RP 361-2)

Sample Identification (365^+ Fractions)	76D-3521	J-8828	J-8830
Liquid Product Properties			
API Gravity	14.7	18.8	23.2
Hydrogen, wt %	9.14	10.46	11.66
Sulfur, wt %	0.102	<0.002	<0.002
Nitrogen, wt %	0.39	0.12	0.04
Oxygen, wt %	1.5	0.3	0.2
KV, cs (at 40°C)	2.988	2.445	2.171
KV, cs (at 100°C)	-	-	1.06
Aromatic Carbon, %	55	42	27
Titanium, ppm	<100	<100	<100
Sodium, ppm	0.59	0.36	0.36
Potassium, ppm	0.08	0.04	0.04
Calcium, ppm	0.14	0.09	0.09
Vanadium, ppm	0.1	0.1	0.1
Lead, ppm	<1.0	<1.0	<1.0
Iron, ppm	10.3	5.7	3.5
Flash Point, °F	195	190	175
Heat of Combustion, Btu/lb	18,081	18,627	19,045
Distillation, °F (D-2887)			
IBP	338	328	325
5%	366	357	352
10	388	379	366
30	406	402	396
50	450	444	441
70	503	493	485
90	595	580	563
95	659	632	609
EP	774	766	733

3.4 COMBUSTION CHARACTERISTICS

The primary objective of this work is to produce fuels derived from coal liquids that can be satisfactorily burned in turbine combustors. Raw coal liquids contain unacceptably high levels of nitrogen and trace metals and are deficient in hydrogen. In addition, they can be incompatible with conventional petroleum fuels. Since the ultimate acceptability of any new fuel can only be determined in actual performance, Westinghouse Electric Corporation was subcontracted to conduct small scale laboratory combustion tests on coal liquid derived fuels. Details of the combustion test program, preparation of the test fuels, and complete results of the tests are found in last year's report (3-1, Section 7) and the final report submitted by Westinghouse, which is included in this report as Appendix A. A paper based on their work was given at the American Chemical Society meeting in Anaheim, California, March 12-17, 1968 (3-2).

During the course of the two-phase combustion program, a series of seven coal-derived fuels were evaluated in a small scale combustor rig located at the Westinghouse Research Laboratory in Pittsburgh. Four Solvent Refined Coal derived liquid fuels were tested during Phase I and three H-Coal derived liquid fuels were evaluated during Phase II. The preparation of the SRC recycle solvent fuels is outlined in last year's report (3-1, Section 7.3). These fuels include a sample of the raw process solvent along with samples of this solvent hydroprocessed to three different severity levels. Details of the preparation of the H-Coal syncrude distillate raw charge and the two hydroprocessed samples of the distillate are found in Section 3.3 of this report.

Specific topics which were addressed during the Phase I program included fuel forwarding and atomization; combustion and emissions; and corrosion, erosion, and deposition. On the basis of these results, the H-Coal distillate testing during Phase II was limited to a determination of only the combustion and emission characteristics. A summary of the results and their interpretation follows.

Results from Combustion Tests

The coal liquid fuels tested, without exception, were: readily forwarded and atomized with no evidence of incompatibility with the #2 oil used for start-up purposes; had no problem with deposits in fuel lines and; had excellent atomizing characteristics. It is suggested, however, that the lighter fractions contained in the fuels may vaporize more readily than the bulk of the fuel creating fuel rich pockets of gas in the vicinity of the nozzle thus contributing to the coking problem.

The test results demonstrated that coke formation ranged from massive for the raw recycle solvent to nothing for the case of the most severely hydroprocessed fuels with more modest coke formation for the various intermediate hydroprocessed fuels. Coke formation is a serious consideration in the utilization of all but the most severely hydroprocessed of the coal liquids. This tendency should be considered in both the selection of combustor modifications and the degree of fuel upgrading required.

Radiant heat transfer from the primary zone to the combustor wall increased the wall temperature for all but one of the fuels, with the raw recycle solvent creating the biggest increase and the most severely hydroprocessed H-Coal fuel producing no perceptible increase. An increase in heat transfer to the combustor wall will require operation at derated conditions or redesign of the combustor. Alternatively, severe hydroprocessing of the coal liquids alleviates this problem. Although qualitatively correct, the actual magnitude of the increased wall temperature is dependent upon the combustor size and design, and larger scale tests are needed to better quantify this effect.

The emission characteristics of the combustor operating on coal-derived liquids were generally as expected. Hydroprocessed coal liquids with lower fuel bound nitrogen resulted in NO_x emissions equivalent to base #2 fuel.

The tendency of the combustor to smoke decreased with decreasing aromaticity of the fuels and a very pronounced decrease in coking was observed with increasing hydroprocessing severity. The recycle solvent produced massive amounts of coke and would require upgrading or major combustor modifications before it could be considered as a gas turbine fuel.

The level of CO observed in each of the tests was sufficiently low and consistent to conclude that CO would not be a concern. The unburned hydrocarbons, however, did increase with percent hydrogen in the fuel. This suggests that the improved wall cooling which will be required in a can type combustor for coal liquids may also return UHC levels to more typical levels. Combustor designs incorporating hot ceramic walls should be capable of particularly low levels of UHC's.

Insufficient operating time was available during these tests to establish the corrosion, erosion or deposition characteristics of each of the fuels. It should be noted, however, that in the test of raw process solvent, incipient deposition of some of the inorganic material in the fuel was observed. Because of the relatively low levels of alkali metals present in the fuels longer periods of exposure are required before definitive data on the rate of attack or deposition can be obtained. In addition, the reduced firing rate (~1.6 gallons per hour) on the pressurized passage utilized in these experiments to conserve fuel also permits extraneous sources of contamination to obscure the results of material testing. Therefore, it is desirable to couple the extended period of operation (e.g., 50 to 300 hrs.) with operation at firing rates equivalent to 15 gallons of fuel per hour. Tests at these conditions will provide reliable turbine life projections.

Section 4

RESIDUAL COAL LIQUIDS

Residual coal liquids can broadly be classified as those containing substantial amounts (>10%) of high boiling 1000°F$^+$ material. They are hydrogen deficient, highly aromatic, viscous liquids or solids containing varying amounts of heteroatoms. They contain high molecular weight (>600 gm/mol) asphaltenes and significant amounts of ash and trace metals.

Residual coal liquids are lower in hydrogen content and higher in heteroatom content than distillate liquids from the same process. In addition, the heteroatoms are more difficult to remove, and the rate of hydrogenation is significantly slower than for the lighter liquids. As a result, the hydroprocessing of residual coal liquids requires more severe operating conditions and results in higher hydrogen consumptions.

Initial work with residual coal liquids was begun in the first year of Phase II of this contract. SRC from both Illinois Burning Star and Monterey coals was hydroprocessed in recycle solvent blends containing 33% SRC. Extensive pilot unit modifications were necessary to enable the charging of these and more concentrated SRC liquids (3-1, Section 3.2). It was found that commercially available CoMo hydrotreating catalysts are effective in significantly increasing the hydrogen content, removing up to 90 wt % of the sulfur, oxygen, and nitrogen, and converting large quantities of the SRC to lower boiling, solvent range material. The nitrogen content could be reduced to less than 0.1 wt % and the hydrogen increased from less than 7 wt % to 11 wt % in these blends. Hydrogen consumption was as high as 4300 scf/B.

A major objective of this work with residual coal liquids was to hydroprocess as high a concentration of residual material as is practical in our modified pilot unit. Hydroprocessing of blends containing from 33-100 wt % SRC was attempted. The use of different catalysts and SRC's produced from several different coal sources (i.e., Monterey, Burning Star, and Wyodak) were evaluated. Considerable work was done on a short contact time variety of SRC produced from Monterey coal. An additional hydroprocessing study was made charging an H-Coal fuel oil product. GEC analyses were used to characterize the residual coal liquid charge stocks and products for comparative and kinetic modeling purposes.

4.1 ANALYTICAL TECHNIQUES

The analytical techniques and procedures used to determine the physical properties and elemental and molecular compositions of coal liquids were described in the EPRI RP 361-1 and RP 361-2 final reports (2-1, 3-1). These analytical procedures will not be described further.

During the past year we used an automated gradient elution chromatographic (GEC) technique for the analysis of non-distillable coal liquids. This was necessary to efficiently handle the number of hydroprocessed samples that were generated. This method uses a motorized valve to change eluents, a solvent pump, and an automatic fraction collector. These changes allow unattended development of the GEC chromatogram which in turn significantly reduce analyst time required to complete the analysis. The manpower and equipment costs necessary to develop this automated GEC technique were not charged to this contract.

Comparative results on a representative sample run by both the manual and automated methods are shown in Table 4-1. No significant difference is seen between these results, and the precision appears to be a few percent (relative) better with the automated method. The automated GEC method will be used to characterize future samples of raw and hydroprocessed residual-containing coal liquids during the third year of the Phase II contract.

Table 4-1

COMPARISON OF MANUAL AND AUTOMATIC GEC METHODS

Identification		650°F$^+$ of Monterey SRC 2:1 Blend (77D1039)	
GEC Method	Manual	Automatic	Automatic
GEC Cuts			
1	0.67	0.67	0.67
2-4	20.34	20.53	20.48
5-7	31.55	31.72	31.04
8-12	37.13	36.84	37.67
Non-eluted + Loss	10.31	10.24	10.14
	100.00	100.00	100.00

4.2 HYDROPROCESSING UNIT MODIFICATIONS

Our standard hydroprocessing pilot unit was extensively modified to enable the processing of high viscosity coal liquids. Modifications which enabled us to charge blends containing 33% SRC in process derived recycle solvent were described in our first report (3-1, Section 3.2). In order to extend our work to higher concentration blends, two short studies were made.

Viscosity-Temperature Data

The viscosity-temperature relationship was determined by measuring the kinematic viscosity at several temperatures for a range of SRC concentrations. The blends were made using SRC from an Illinois No. 6 Monterey coal (76D2155). The solvent (77D391) was prepared by distilling a sample of process derived recycle solvent to remove the material boiling below 400°F. This served to minimize the solvent vaporization at the higher temperatures. This solvent was also used for preparing all the charge blends in this contract. Figure 4-1 shows the viscosity as a function of temperature and SRC concentration. The maximum viscosity we can handle in our pilot unit charge system is 50-100 cs. Figure 4-1 can be used to determine the charge system temperature needed for a particular blend composition.

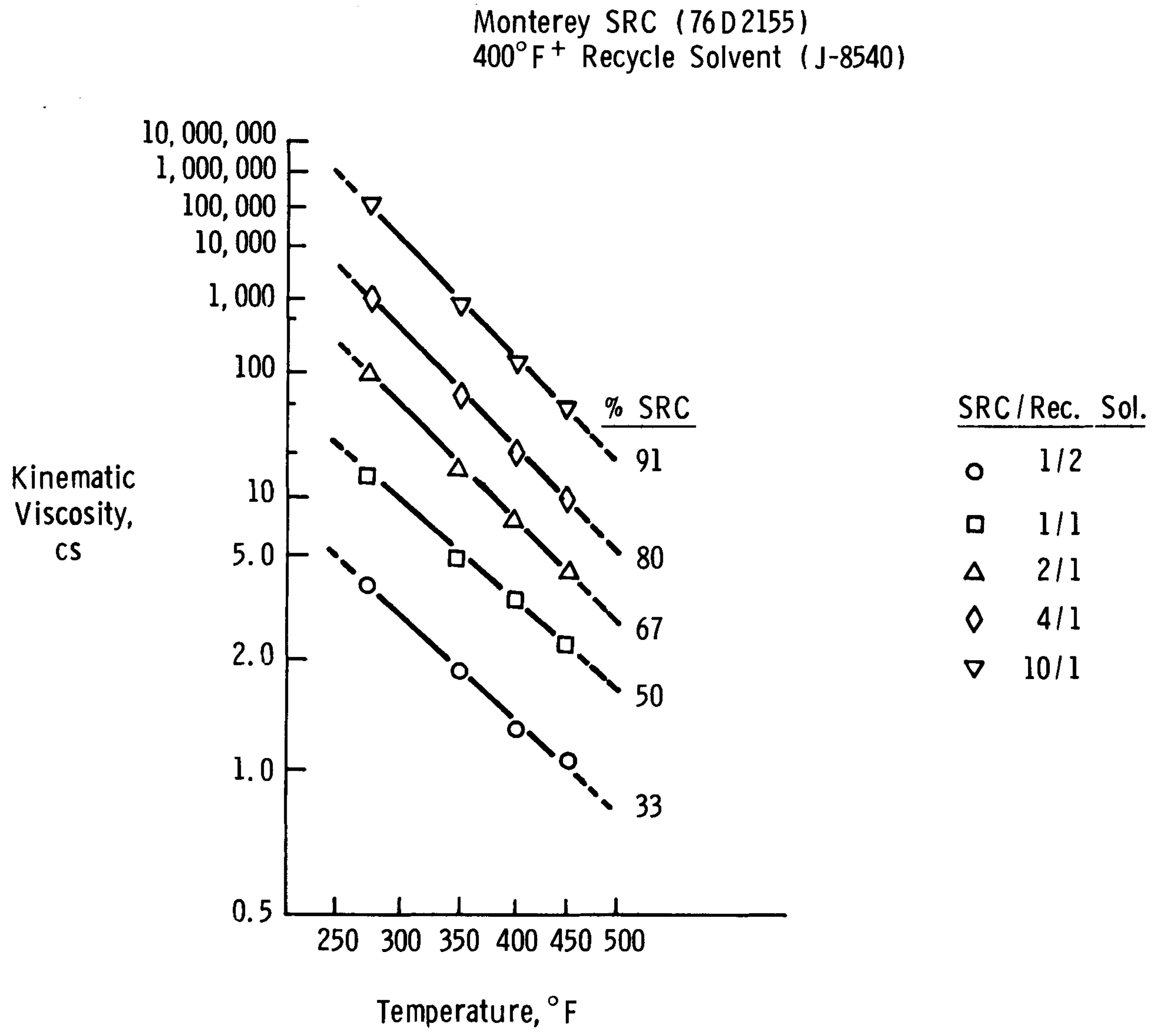

Figure 4-1. Viscosity of SRC/Recycle Solvent Blends

The data in Figure 4-1 were used to develop a simple model for predicting the viscosity as a function of temperature and SRC concentration. The form for the model equation was taken from the literature (4-1). The model equation is:

$$\ln KV = X_{SOL} \left(A_{SOL} + E_{SOL}/(T + B_{SOL})\right) + X_{SRC} \left(A_{SRC} + E_{SRC}/(T + B_{SRC})\right) \tag{4-1}$$

Where KV is the dimensionless kinematic viscosity of the blend relative to a basis of 1 centistoke; X is the mole fraction in the blend and T is the temperature in °F. A is a dimensionless constant, and B and E are constants with units of °F. In order to use this equation average molecular weights of 750 and 250 were assumed for the SRC and recycle solvent, respectively. The constants for Equation 4-1 determined by a least squares fit of the data are:

Constants for Viscosity Model

	A	B, °F	E, °F
Recycle Solvent	-0.733	-322.5	-2.95
SRC	-7.33	-12.4	5320

Figure 4-2 compares the data with the model predictions. The model fits the data very well with only the highest viscosity, highest SRC concentration data point deviating by any significant amount.

Thermal Degradation Experiment

The viscosity-temperature data show that temperatures greater than 400°F are necessary to pump high concentration blends of SRC. Since the feed must remain at these temperatures for several days in the charge reservoir, there was some concern about thermal degradation. A simple experiment was performed. A sample of Monterey SRC (76D2155) was held at 400°F under a nitrogen atmosphere for 11 days. Samples withdrawn after one day and 11 days were analyzed by our GEC technique. The results (Table 4-2) show the polar asphaltenes increase while the lower GEC fractions decrease with time. This degradation is not severe; however, any degradation is undesirable, and in our pilot plant runs efforts were made to avoid subjecting the charge to temperatures greater than 400°F for extended periods.

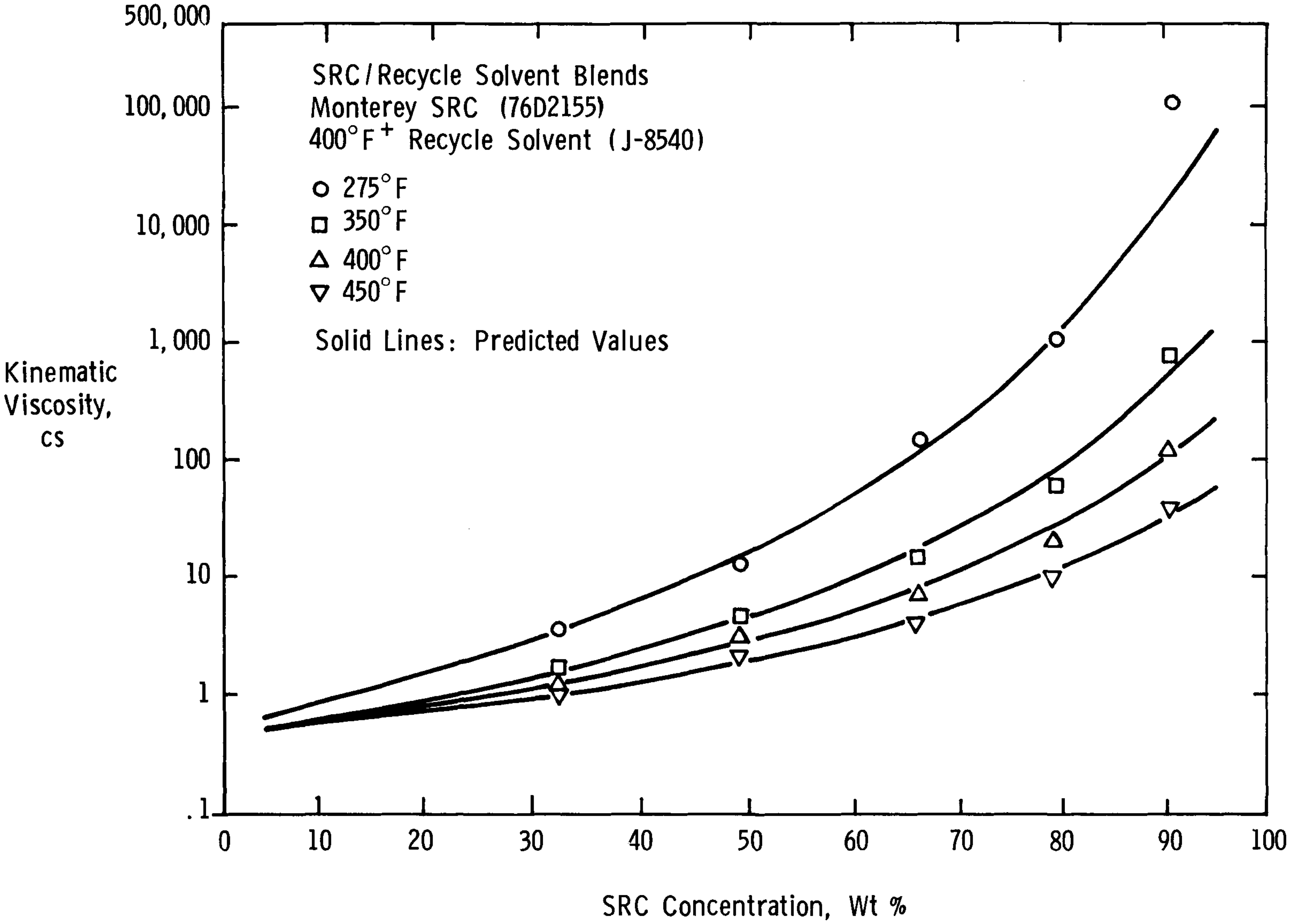

Figure 4-2. Kinematic Viscosity as a Function of SRC Concentration

Table 4-2

THERMAL STABILITY OF MONTEREY SRC (76D2155)

CUT NO.	GEC ANALYSIS, WT %	CHARGE	1 DAY AT 400°F	11 DAYS AT 400°F
1	Saturates	0.15	0.07	0.06
2-4	Aromatic Oils	11.08	9.49	9.56
5-7	Resins/Asphaltenes	36.58	35.45	33.01
8-13	Polar Asphaltenes	52.19	54.99	57.37
	TOTAL	100.00	100.00	100.00

To insure that we would detect any serious degradation of our feed, we always sampled the charge stock for analysis at a point immediately upstream of our charge pump. Only one charge stock, a 70% blend of Monterey SRC, showed signs of substantial degradation. This sample had an unexpected high concentration of oxygen and polar asphaltenes. It is suspected that the nitrogen bleed in the charge reservoir was interrupted and the blend was oxidized. This degradation, however, was not apparent from inspections of the hydroprocessed products from this run. Apparently, this oxidation was easily reversed in the pilot unit.

Pilot Unit Modifications

Modifications to our pilot unit to process 33% SRC blends were described in our last report (3-1).

Additional unit modifications were necessary before higher concentration blends and blends with short contact time SRC could be processed. Preparation of our 33% SRC blends in a steam-heated hot box (~ 220°F) with air-driven stirrers generally presented no problems. Dissolution of a 33% short contact time SRC blend required heating to ~300°F in a modifed steam chest. Noxious vapors released at these temperatures and the handling and transferring of the hot material presented serious problems.

Changes were made to the unit to enable the preparation of SCT or high concentration blends SRC at the pilot unit. An existing gear pump was connected to an auxiliary-heated feed tank system for use in preparing blends. The blends were prepared in the auxiliary tank and mixing was accomplished using the gear pump to recirculate the blend. The blends were then pumped to the charge reservoir.

Problems in maintaining flow in the feed reservoir system required additional heat tracing, insulation, and piping changed. The heat traced high pressure valves developed leaks on heating and cooling of the unit. The graphite-asbestos packing apparently was either attacked by the liquid or could not withstand the temperature cycles. We found more success with valves with extended stems and with valves having inert teflon-glass packing. Although these valves have a lower temperature rating (≈550°F), their packing is apparently more flexible.

The Milton Roy charge pump ball checks frequently failed to seat and required cleaning. Vapor locks also created some problems and a special bleed valve was installed and found to be useful.

No reactor plugging problems occurred; however, problems with the post-reactor separators were encountered. The capacitance probe in the high temperature, high pressure separator periodically gave false level indications. This resulted in either a drop in unit pressure or an overflow of heavy liquid into the low pressure separator. After dismantling the high pressure separator after one upset, a solid ring was found deposited on the probe. This apparently resulted in an erroneous high liquid level indication and allowed gas to escape through the liquid let-down valve.

A major recurring problem was the failure of the high pressure, low temperature separator gasket. This resulted in loss of unit pressure and premature shutdown of several runs. The graphite-asbestos gaskets always failed at a point where they contacted the liquid product. Apparently this liquid was able to soften and weaken the gasket material. The sight glass was replaced with an all-metal separator and the problem was eliminated.

4.3 SOLVENT REFINED COAL

Four different SRC products were used in this work; one from Illinois No. 6 coal (Burning Star Mine), one from Wyodak coal (Amax), and two from Illinois No. 6 coal (Monterey Mine). Hydrogen contents ranged from 5.6-6.2 wt %, nitrogen contents from 1.7 to 2.06 wt %, and sulfur contents from 0.10 to 0.96 wt %. Complete physical and chemical analyses of these SRC products were reported in our 1977 EPRI Report (3-1, Section 4.3).

We also obtained and analyzed a sample of Wilsonville SRC filter feed which had been deashed in the Lummus antisolvent deashing unit in Bloomfield, New Jersey. The SRC was produced from a Burning Star coal. The sample as received contained a liquid and a solid phase. The liquid (top) phase was sampled and analyzed and then the drum was heated to 300°F for 1-2 days. The solid apparently dissolved and the drum was mixed well and sampled again. Table 4-3 compares the analyses of the initial liquid phase and the well-mixed sample to the Lummus analysis. The top phase is apparently a lighter fraction of the well-mixed sample. Although our well-mixed sample is much closer to the Lummus analyses, it still appears to be somewhat lighter.

A GEC analysis shows the Lummus deashed product to be very similar in composition to our blend of 33% Burning Star SRC in recycle solvent (Table 4-4). However, it appears that their process has removed some of the oxygen and nitrogen from the SRC. No further analysis or hydroprocessing of the Lummus sample was done.

Hydroprocessing of 33% Blends of SRC

Blends containing 33% SRC in recycle solvent were hydroprocessed over a commercial catalyst (Cyanamid HDS 1441A) in our fixed bed pilot unit. Results for the processing of the 33% blends of Illinois No. 6 Burning Star and Monterey SRC's along with properties of this CoMo/Al_2O_3 catalyst, were included in the previous annual report (3-1). Although the chemical and physical properties of the two blends were quite similar, the Monterey SRC was found to be more difficult to process than the Burning Star. To obtain the same upgrading, the Monterey would have to be hydroprocessed at about half the space velocity as the Burning Star. It was possible, however, to substantially upgrade both of these SRC products.

Table 4-3

ANALYSES OF LUMMUS ANTISOLVENT-FREE OVERFLOW PRODUCT FROM WILSONVILLE FILTER FEED BURNING STAR COAL

SAMPLE	LUMMUS*	MOBIL TOP PHASE SAMPLE	MOBIL MIXED SAMPLE
Elemental Analysis			
Hydrogen, Wt %	6.72	7.81	7.03
Sulfur, Wt %	0.47	0.35	0.53
Oxygen, Wt %	3.26	2.4	3.1
Nitrogen, Wt %	0.93	0.55	0.75
Ash, ppm	200	200	--
Benzene Insolubles (D-367), Wt %	11.79	--	13.14
Distillation, °F @ %	(VAC)	(Sim. Dist)	(Sim. Dist)
IBP	405	365	371
5	447	400	408
10	465	408	411
30	529	447	478
50	612	482	557
70	703	532	673
90	--	621	--

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* EPRI Report AF-234, Table 4.

Table 4-4

COMPARISON OF GEC AND ELEMENTAL ANALYSES OF LUMMUS SOLVENT DEASHED PRODUCT AND 33% BURNING STAR SRC BLEND

Cut No.	GEC Analysis	LUMMUS PRODUCT 76D3031 (Wt %)	2:1 RECYCLE SOLVENT-BURNING STAR SRC BLEND J8151 (Wt %)
1	Saturates	2.18	2.73
2-4	Aromatic Oils	46.87	43.71
5-7	Resins/Asphaltenes	15.22	17.84
8-12	Polar Asphaltenes	17.40	17.60
13	Non-Eluted + Loss	18.33	18.12
		100.00	100.00
	Elemental Analysis		
	Hydrogen, Wt %	7.03	6.84
	Sulfur, Wt %	0.53	0.41
	Nitrogen, Wt %	0.75	1.03
	Oxygen, Wt %	3.1	3.91

GEC analyses of the charge stocks and some of the products from each of these studies have been obtained during this past year. An additional hydroprocessing study using an SRC prepared from a Wyodak Amax coal has also been done and the products analyzed by GEC. The GEC composition (i.e., oils, resins, and asphaltenes) have been related to hydroprocessing severity, and a simple kinetic model for conversion has been developed . New data obtained relating to each of these three studies are presented in the following sections.

Burning Star SRC. Hydroprocessing results for the study charging a 33 wt % blend of Illinois No. 6 Burning Star SRC were reported in Section 5.4 of our last annual report (3-1). GEC analyses were since obtained on the charge and five selected products from this run. These liquids were first distilled to a 500°F cut point to minimize loss of the sample during solvent stripping in the GEC procedure. Table 4-5 presents the normalized GEC analyses of the liquids based on the total sample along with the elemental analyses. The yield of 500°F$^-$ material increased from 25.1 wt % in the charge to 54.3 wt % in the most severely hydroprocessed sample (CT 199-448). The asphaltenes fraction decreases rapidly with increasing hydroprocessing severity, while the 500°F$^-$ material and saturates increase. The resins fraction decreases slowly, but drops to very low levels at high severity. The oils increase with increasing severity initially, then decrease at high severity.

The elemental analyses of the distillation overhead and bottom fractions are shown in Table 4-6. Although the overhead cuts (500°F$^-$) contain more hydrogen and less heteroatoms than the bottoms fraction (500°F$^+$), considerable heteroatom removal and hydrogenation of the heavier SRC fraction has occurred.

The hydrocarbon type analysis of the GEC saturate (Cut 1) and oil (Cut 2-4) fractions of the 500°F$^+$ material from the raw and hydroprocessed material is shown in Table 4-7. As a result of hydroprocessing, the aromatic oils contain less polynuclear aromatic hydrocarbons and more mono-aromatics. The possibility that polynuclear aromatics are hydrogenated to form naphthenes is supported by the observed increase in naphthene content of the saturate fraction with increased hydroprocessing severity. The concentration of paraffins in the saturates fraction decreases with increasing hydroprocessing severity. However, the absolute amounts of both paraffins and saturates are increasing.

Table 4-5

GEC ANALYSES OF HYDROPROCESSED 33% BURNING STAR SRC BLEND

	J8151 CHARGE	MB-440	MB-446	MB-442	MB-445	MB-448
H_2 Consumption, scf/B	--	1339	1745	1788	2937	3332
Elemental Analysis						
Gravity, °API	-3.0	10.2	4.1	12.6	16.9	20.1
Hydrogen, Wt %	6.84	8.38	8.98	9.44	10.17	10.57
Sulfur, Wt %	0.41	0.20	0.18	0.07	0.05	0.05
Oxygen, Wt %	3.91	1.80	2.20	0.50	0.20	0.20
Nitrogen, Wt %	1.03	0.75	0.92	0.63	0.16	0.10
CCR, Wt %	16.49	10.47	11.29	6.02	3.63	3.99
Analysis Total Liquid Product, Wt %						
500°F$^-$	25.08	36.20	34.13	44.47	47.45	54.34
Cut 1 (Saturates)	1.68	3.13	2.24	3.20	5.35	6.31
Cut 2-4 (Oils)	30.75	31.83	33.61	38.86	37.64	32.54
Cut 5-7 (Resins)	17.71	15.82	17.24	9.12	5.36	3.74
Cut 8-13 (Asphaltenes)	24.78	13.02	12.78	4.35	4.19	3.07
Total	100.00	100.00	100.00	100.00	100.00	100.00

Table 4-6

ELEMENTAL ANALYSES OF DISTILLATION CUTS FROM HYDROPROCESSED 33% BURNING STAR SRC BLEND

	CHARGE (J8151)	MB-440	MB-446	MB-442	MB-445	MB-448
$500°F^-$ Yield, Wt %	25.08	36.20	34.13	44.47	47.45	54.34
Carbon, Wt %	85.2	87.6	86.9	88.7	88.8	88.4
Hydrogen, Wt %	8.10	10.14	10.12	11.44	11.63	11.74
Sulfur, Wt %	0.12	0.13	0.011	0.003	<0.001	--
Nitrogen, Wt %	0.23	0.20	0.35	0.09	<0.03	<0.03
Oxygen, Wt %	4.6	1.1	2.1	0.2	0.2	0.1
$500°F^+$ Yield, Wt %	74.92	63.80	65.87	55.53	52.55	45.66
Carbon, Wt %	82.1	89.6	88.9	88.6	89.2	89.1
Hydrogen, Wt %	5.97	7.30	7.18	7.85	8.80	8.83
Sulfur, Wt %	0.61	0.20	0.15	0.035	0.09	0.014
Nitrogen, Wt %	1.25	1.02	1.02	0.62	0.32	0.18
Oxygen, Wt %	3.5	2.1	2.1	0.9	0.5	0.3

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*Listed in order of increasing severity of hydroprocessing.

Table 4-7

HYDROCARBON TYPE ANALYSIS FOR SATURATE AND OIL GEC FRACTIONS OF HYDROPROCESSED 33% BURNING STAR SRC BLEND

	500°F Residue of:											
CT-199-	J8151 Charge		440		446		442		445		448	
H_2 Consumption, scf/B	-		1339		1745		1788		2937		3332	
	Concentration in GEC Fractions, wt %											
GEC Fraction	1	2-4	1	2-4	1	2-4	1	2-4	1	2-4	1	2-4
Paraffins	59.3	0	50.4	0	48.8	0	37.6	0	25.1	0	26.8	0
Naphthenes	38.2	0	48.1	0	48.9	0	59.8	0	67.6	0	71.6	0
Mono-Aromatics	2.5	12.9	1.6	27.2	2.4	29.3	2.6	46.2	7.4	47.4	1.7	59.2
Di-Aromatics	0	55.7	0	46.9	0	46.0	0	36.3	0	27.9	0	29.3
Tri-Aromatics	0	23.4	0	17.3	0	15.6	0	10.6	0	8.1	0	7.5
Tetra-Aromatics	0	4.6	0	3.3	0	2.6	0	1.9	0	7.8	0	0.8
Penta-Aromatics	0	0.5	0	1.8	0	2.1	0	2.2	0	5.1	0	1.6
Aromatic Sulfur Cpds.	0	2.9	0	3.3	0	3.9	0	2.8	0	3.0	0	1.7
Unidentified Aromatics	0	0	0	0.1	0	0.5	0	0	0	0.8	0	0
Total	100.0	100.0	100.1	100.0	100.1	100.0	100.0	100.0	100.1	100.1	100.1	100.0

The data from this SRC were limited and process modeling was not attempted. The observed trends, however, are similar to those with other SRC samples.

Monterey SRC. Results from the hydroprocessing of a blend containing 33 wt % Monterey SRC (75D 3018) are reported in our last annual report (3-1, Section 5.4). GEC and elemental analyses of several of the products from this run are shown in Table 4-8. The total liquid products were distilled to remove the 650°F$^-$ material. This cut point was chosen over the 500°F point used in the Burning Star samples since the 650°F$^+$ material more nearly represents the unconverted SRC in the blend.

With increasing severity of hydroprocessing (i.e., hydrogen consumption), there is a corresponding decrease of 650°F$^+$ material; and, within this residue, Cuts 5-13 became proportionately less as the saturates and aromatic oils increase. Sulfur and nitrogen are concentrated in the residua, but oxygen is rather evenly distributed between the two boiling ranges. At the most severe hydroprocessing condition (MB-584 at 3924 scf/B hydrogen consumption), 90% of the liquid product boils below 650°F compared to 51 wt % in the charge. The polar and non-eluted asphaltenes account for less than 1 wt % of the total liquid product compared to 26 wt % in the charge. Thus, greater than 95 wt % of the most refractory fraction was converted to more saturated, lower boiling material.

The GEC analysis is a preparative technique and the resulting samples can be analyzed to obtain additional information. Table 4-9 gives the elemental analyses of the GEC cuts from the raw and hydroprocessed Monterey SRC blend. The data show the saturates fraction (Cut 1) to be uniformly high in hydrogen concentrations (12.54--->13.75 wt %). The higher GEC fractions show decreasing amounts of hydrogen. The hydrogen contents of the oils (Cuts 2-4) and resins (Cuts 5-7) fractions are low in the charge (6.41-6.57 wt %), but increase with increasing hydroprocessing severity. This indicates that these fractions can be hydrogenated to some extent without being converted to a lower fraction. The asphaltenes (Cuts 8-12), however, have uniformly low hydrogen concentrations (5.91--->6.38 wt %) even after the most severe hydroprocessing, and it appears that hydrogenation of the asphaltenes is slow and/or any hydrogenation which does occur removes that molecule from the asphaltene cut. Oxygen content increases with increasing GEC cut number. However, the nitrogen content is highest in the resin fraction.

Table 4-8

GEC ANALYSES OF HYDROPROCESSED 33% MONTEREY SRC BLEND

Unit: CT-199	CHARGE	MB-574	MB-575	MB-576	MB-577	MB-578	MB-579	MB-580	MB-581	MB-582	MB-584
Operating Conditions											
Temperature, °F	-	672	723	780	672	729	726	775	675	727	777
Pressure, psig	-	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.99	0.96	0.82	0.57	0.99	0.46	0.48	0.24	0.22	0.10
Hydrogen Consumption, scf/B	-	726	1047	1183	1085	1377	1504	1946	1289	2490	3924
Total Liquid Product, Wt %											
IBP-650°F	51.21	62.38	59.32	59.74	62.82	55.85	60.97	72.59	55.34	67.78	89.78
650°F+											
Cut 1 (Sats.)	0.26	0.23	0.48	0.74	0.28	0.65	0.93	0.48	0.63	0.60	1.12
Cut 2-4 (Oils)	9.51	8.48	17.11	22.54	9.40	17.40	16.80	13.70	17.33	15.40	7.63
Cut 5-7 (Resins/Asph.)	12.93	15.60	14.57	12.75	14.77	15.01	13.77	9.35	14.78	10.63	0.81
Cut 8-13 (Polar Asph.)	26.09	13.31	8.52	4.23	12.73	11.09	7.53	3.88	11.92	5.59	0.66
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Elemental Analysis(a)											
IBP-650°F											
Hydrogen, Wt %	7.66	8.57	9.14	8.83	8.75	9.95	9.41	9.61	9.34	10.86	11.14
Oxygen, Wt %	3.8	2.6	2.2	1.2	1.9	1.0	2.3	0.3	2.0	0.9	0.3
Nitrogen, Wt %	0.63	0.42	0.35	0.26	0.36	0.27	0.17	0.15	0.22	0.09	0.07
Sulfur, Wt %	0.25	0.085	(b)	0.024	0.042	0.016	0.011	0.011	0.034	0.011	0.011
650°F+ Residue											
Hydrogen, Wt %	6.16	6.50	6.60	6.89	6.73	6.60	7.31	7.14	7.12	7.50	8.47
Oxygen, Wt %	4.4	4.0	2.0	1.2	2.7	2.4	1.6	1.4	1.8	1.2	0.6
Nitrogen, Wt %	1.39	1.65	1.38	1.05	1.57	1.33	1.24	0.98	1.21	0.87	0.20
Sulfur, Wt %	0.91	0.47	(b)	1.26	0.46	0.37	0.25	0.13	0.25	0.24	0.031

(a) For each sample the elements were determined directly for one distillate cut and calculated by difference for the other cut.

(b) Analysis not determined.

Table 4-9

ELEMENTAL ANALYSES OF GEC FRACTIONS FOR HYDROPROCESSED 33% MONTEREY SRC BLEND

	76D1669 CHARGE	MB-574	MB-576	MB-578	MB-579	MB-580	MB-581	MB-582
Process Conditions								
Temperature, °F	-	672	780	729	726	775	675	727
LHSV, V/Hr/V	-	0.99	0.82	0.99	0.46	0.48	0.24	0.22
Pressure, psig	-	2000	2000	2000	2000	2000	2000	2000
650°F$^+$ Residue, wt %	49.79	37.62	40.26	41.15	39.03	27.41	44.66	32.28
Hydrogen, wt %								
650°F Residue	6.16	6.50	6.89	6.60	7.31	7.14	7.12	7.50
Cut 1	(a)	(a)	13.75	12.54	12.77	13.55	13.45	12.91
Cut 2-4	6.57	7.83	7.80	8.02	8.18	8.02	8.03	8.74
Cut 5-7	6.41	6.96	6.51	6.97	7.12	6.74	7.40	7.14
Cut 8-12	5.91	6.00	5.58	5.83	5.76	5.74	6.38	6.20
Oxygen, wt %								
650°F Residue	4.4	4.0	1.2	2.4	1.6	1.4	1.8	1.2
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4		2.5	1.3	0.9	0.8	0.7		0.5
Cut 5-7	3.8	3.4	1.9	3.5	4.0	2.9	3.3	3.3
Cut 8-12	8.2	6.2	4.3	6.6	8.6	5.6	8.0	5.7
Nitrogen, wt %								
650°F Residue	1.39	1.65	1.05	1.33	1.24	0.98	1.21	0.87
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	0.35	0.62	0.23	0.31	0.32	0.20	0.33	0.34
Cut 5-7	2.56	2.16	2.41	2.19	2.00	1.84	1.95	1.47
Cut 8-12	1.70	1.03	1.50	1.15	1.82	1.81	1.55	0.96
Sulfur, wt %								
650°F Residue	0.91	0.47	0.126	0.367	0.252	0.128	0.251	0.244
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	0.68	0.25	0.08	0.15	0.25	0.03	0.11	<0.03
Cut 5-7	0.63	0.21	0.07	0.24	0.27	0.10	0.13	<0.03
Cut 8-12	0.98	0.50	(a)	0.59	0.61	(a)	0.33	(a)

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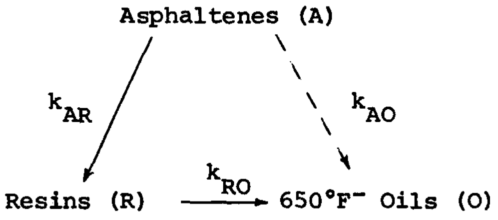
*(a) - Analysis not determined.

The conversion selectivity for the Monterey SRC is shown in Figures 4-3 and 4-4. The hydrogen content of the liquid product is used as an index of hydroprocessing severity. Figure 4-3 shows the asphaltenes decrease rapidly from the charge value of 26.09 wt % in the feed, to less than 1 wt % at the most severe conditions. The resins remain roughly constant at about 13-15 wt % until the asphaltenes are almost depleted, and then also decline to less than 1 wt % at high severity. The concentration of the oils, saturates and 650°F$^-$ material can be combined into one lump. The concentration of the lump increases almost linearly with the hydrogen content of the liquid product.

The observed selectivity suggests that the asphaltenes are converted to resins and then to lighter products. This results in the following reaction network.



The above reaction scheme was evaluated using first order rate equations for both steps and assuming an Arrhenius temperature dependence. It was found that the best fit to the data estimated too low an activity for the data at short times on stream (< 4 days) and too high an activity for the data at long times on-stream (> 4 days). Previously reported data had shown as much as 50°F aging in 7.5 days with a similar 2/1 blend over this small pore catalyst (HDS-1441A). These findings suggested that inclusion of an aging parameter into the rate equation may be necessary. The rate constants were assumed to decay according to the equation

$$k_{ij} = k^{o}_{ij} \exp^{-at} \qquad (4-2)$$

where: t = days on-stream

a = catalyst decay constant, days^{-1}

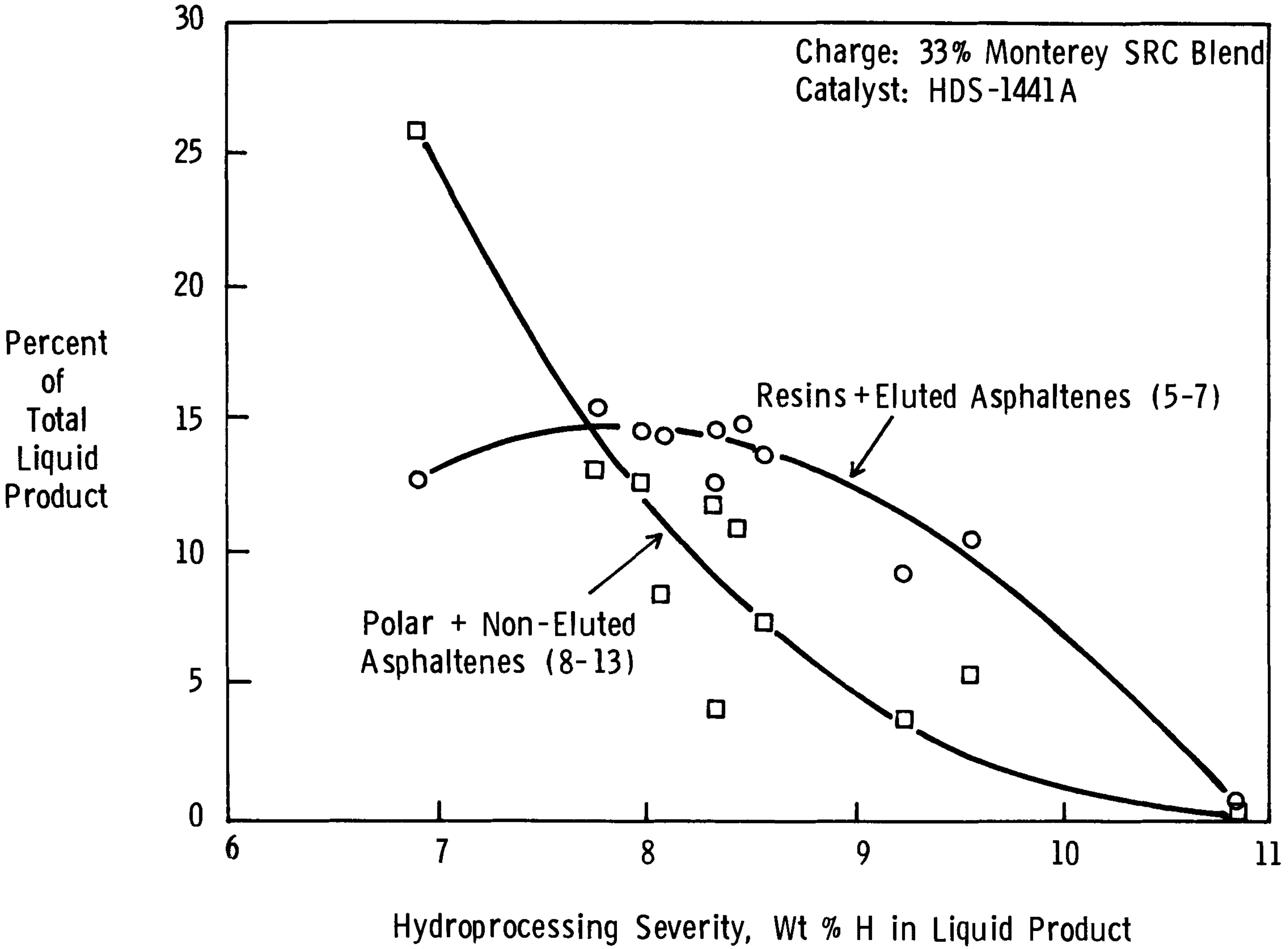


Figure 4-3. Resins and Asphaltenes Conversion for 33% Monterey SRC Blend

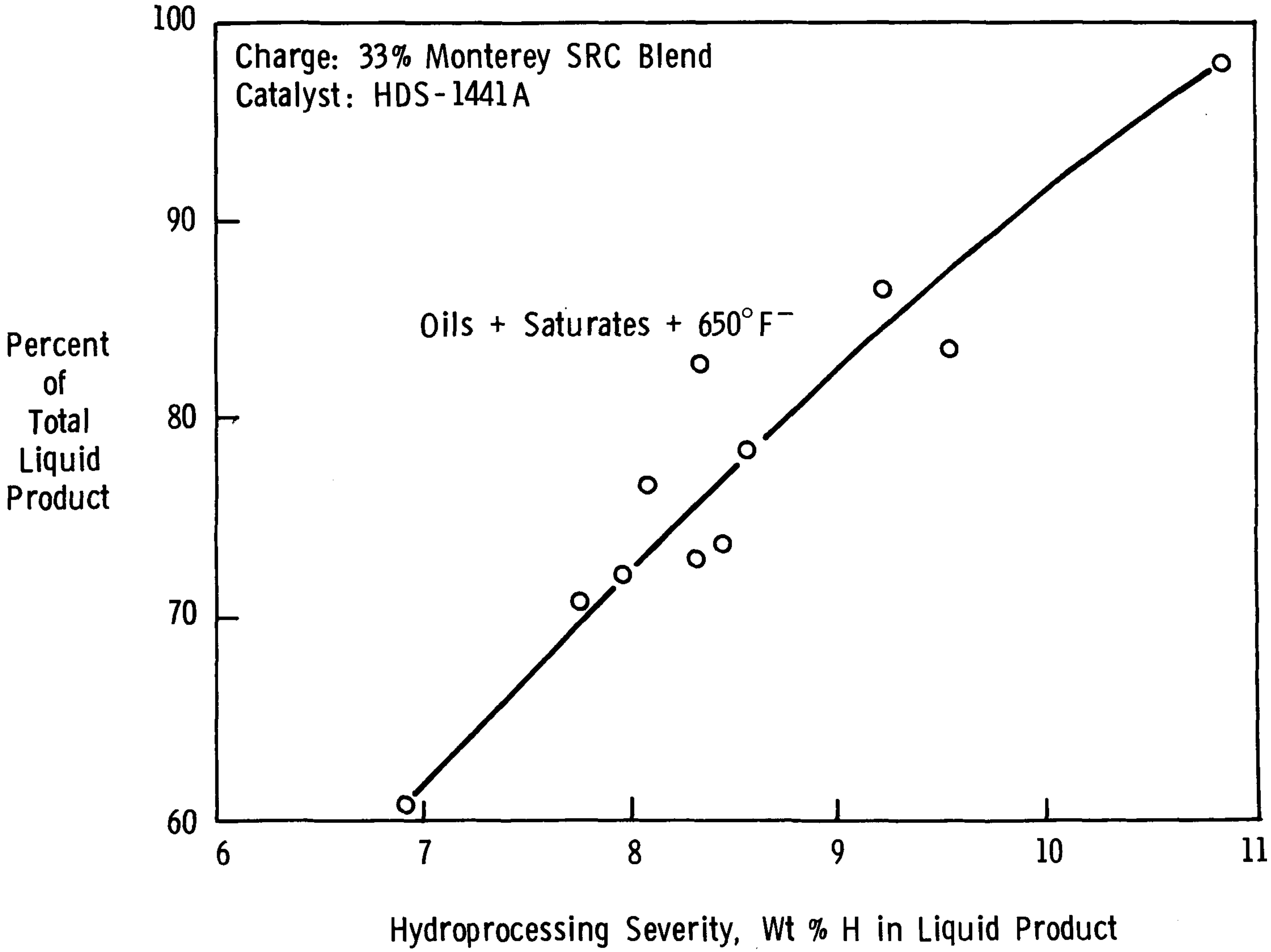


Figure 4-4. Oils + Saturates Converstion for 33% Monterey SRC Blend

Refitting the data with a time dependent rate constant yielded a value of k_{AO} very close to zero. This reaction path was eliminated, simplifying the reaction scheme to two consecutive first order reactions. Integration of the first order equations yields the following expressions for the asphaltene and resin concentrations

$$A = A_o \; e^{-k_{AR}/LHSV} \qquad (4\text{-}3)$$

$$R = \frac{k_{AR}A_o}{k_{RO}-k_{AR}} \; e^{-k_{AR}/LHSV} \; + \; R_o \; - \; \frac{k_{AR}A_o}{k_{RO}-k_{AR}} \; e^{-k_{RO}/LHSV} \qquad (4\text{-}4)$$

where k is a function of time as defined by equation (1).

The overall fit of the model to the data is given in Figure 4-5. The model fits the data well over the entire range of conversions. The constants obtained from this fit are given in Table 4-10 below:

Table 4-10

KINETIC CONSTANTS* FOR THE CONVERSION OF ASPHALTENES AND RESINS FROM A 33% BLEND OF MONTEREY SRC

$$A \xrightarrow{k_{AR}} R \qquad R \xrightarrow{k_{RO}} O$$

STEP	FREQUENCY FACTOR (k^o_{ij}), HR-1	ACTIVATION ENERGY (E_{ij}), BTU/LB MOLE	DECAY CONSTANT (a), DAY^{-1}
A ---> R	7.55×10^6	36,527	0.1633
R ---> O	9.69×10^5	31,493	

$$* \; k_{ij} = k^o_{ij} \; e^{-at} \; e^{-E_{ij}/RT}$$

The predicted effect of LHSV on the conversion of asphaltenes and resins is given in Figure 4-6. This prediction was made for four days on stream at 725°F and 2000 psia. The model predicts the characteristic maximum in the resin concentration and the relatively rapid decline is asphaltenes as a function of reaction time. Figure 4-7 shows the effect of catalyst aging on the resin and asphaltene

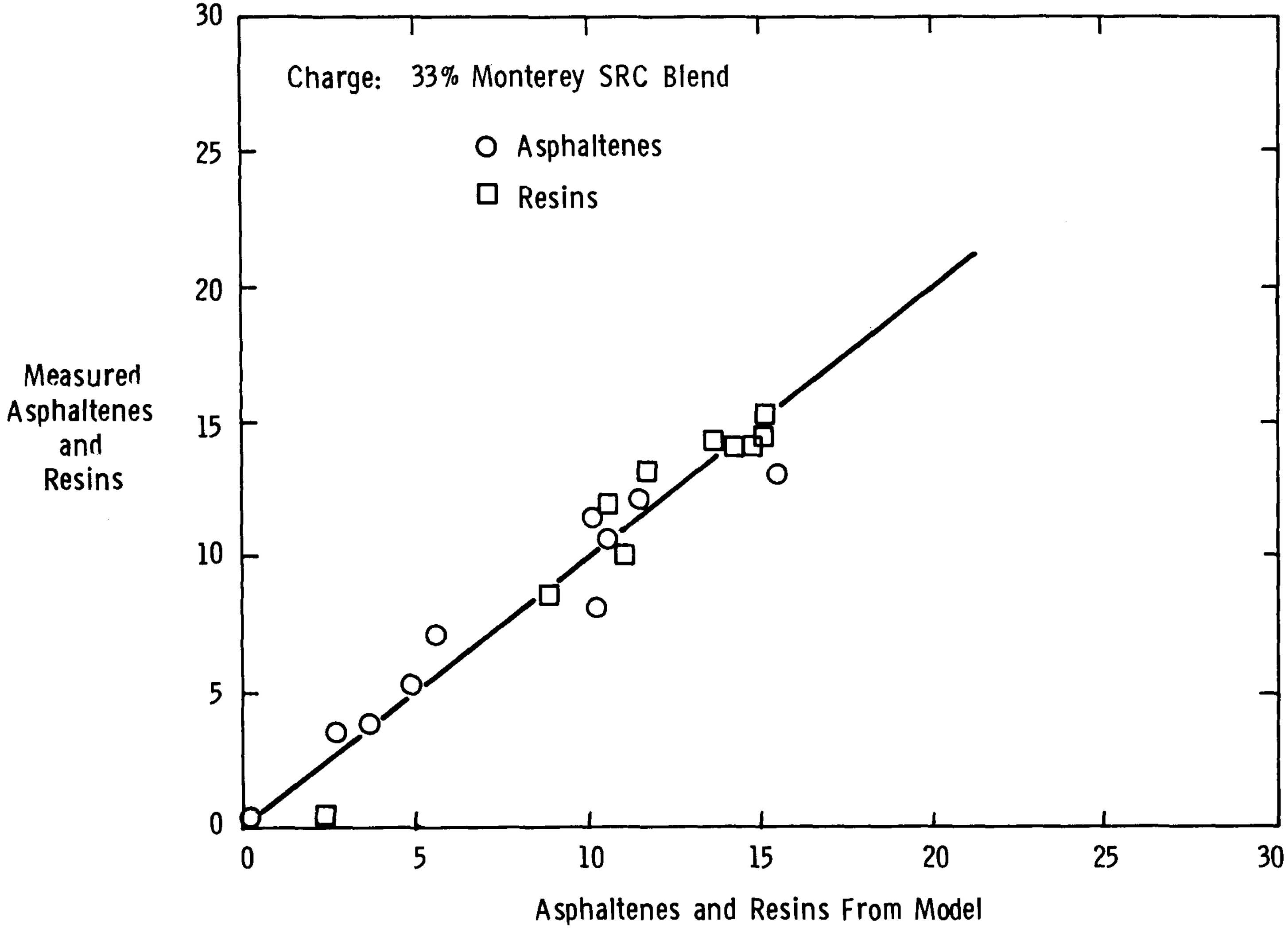


Figure 4-5. Fit of Model to Asphaltenes and Resins: 33% Monterey SRC Blend

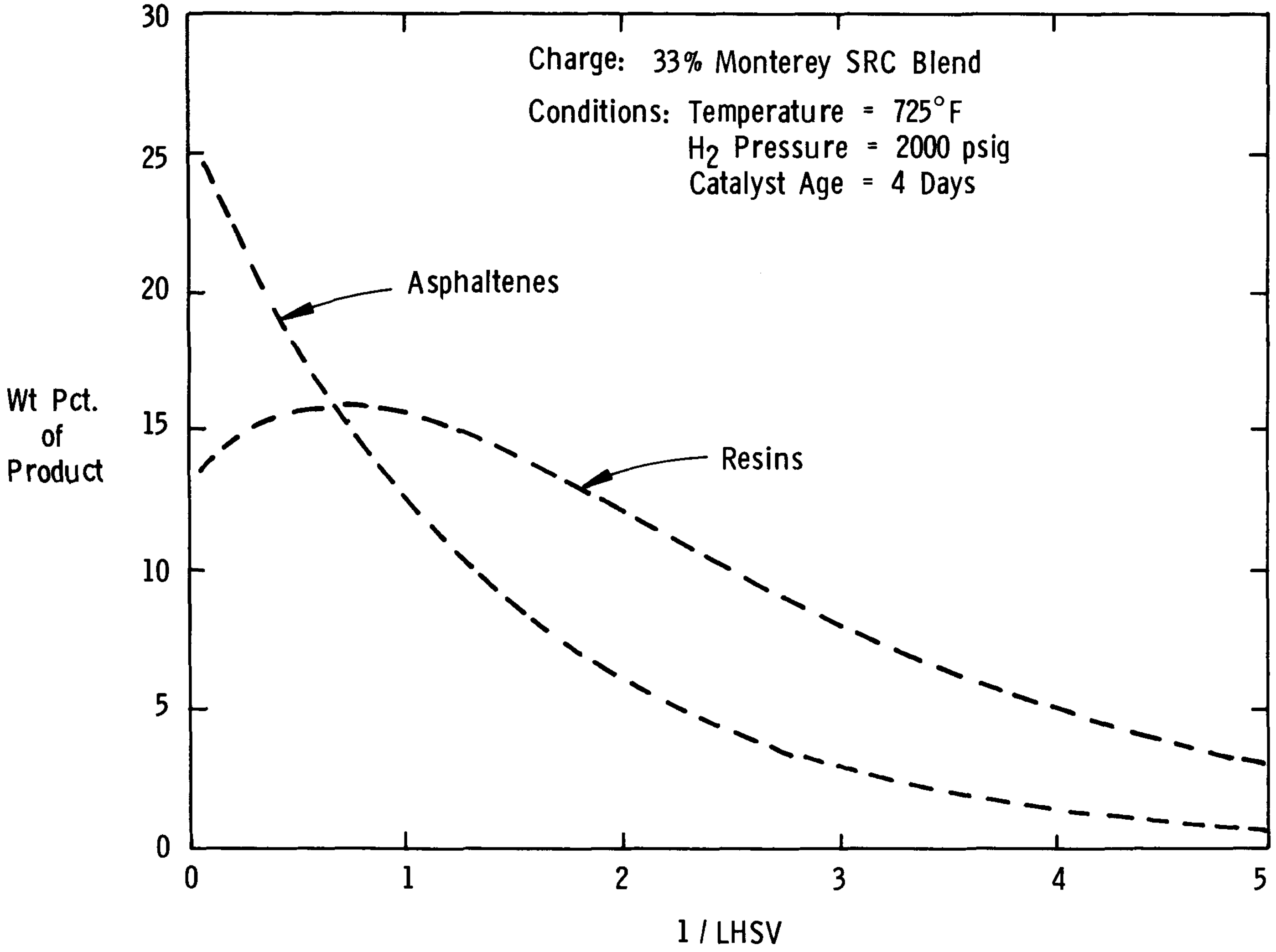


Figure 4-6. Predicted Effect of LHSV on the Converstion of Resins and Asphaltenes

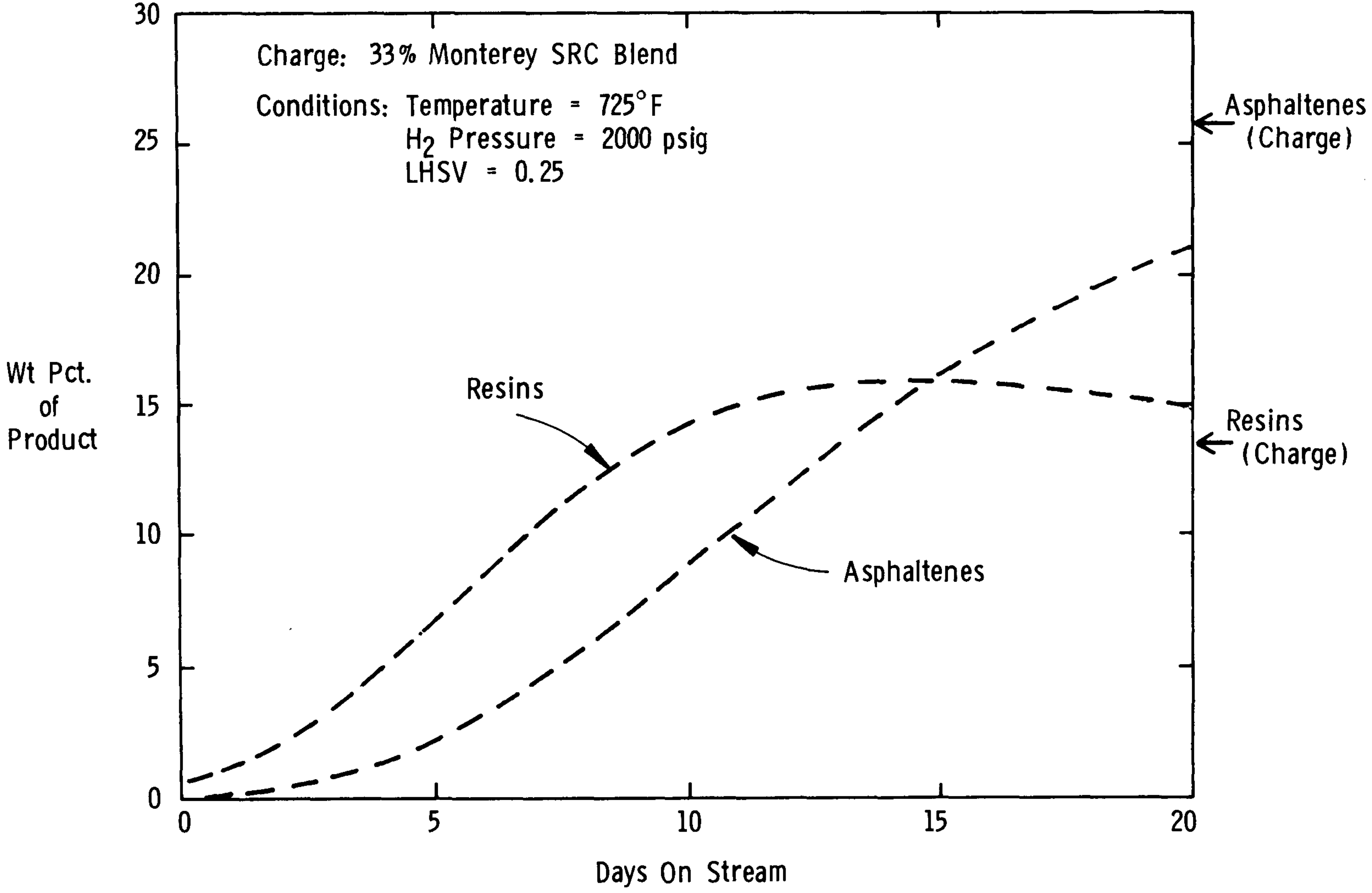


Figure 4-7. Prediction of Aging on Conversion of Resins and Asphaltenes

concentration at 725°F, 2000 psig H_2 pressure, and 0.25 LHSV. The model predicts that by twenty days on-stream this catalyst will have almost no activity for the conversion of asphaltenes and resins. The predicted activity loss for this small pore catalyst from day 1 to day 20 is equivalent to more than 250°F in reactor temperature. It should be noted that this was a ten day process variable study in which the conditions and aging rate varied widely. Any aging rate determined from these data is very qualitative; however, excessive aging may be a problem with these feeds.

Wyodak SRC. A 33 wt % blend of Wyodak SRC in recycle solvent was hydroprocessed in our pilot unit over HDS-1441A (CoMo) catalyst. After ten days on-stream, a slight pressure differential ($\sim$ 90 psi) across the reactor was observed, but this problem alleviated itself and the program was continued for fourteen days before a voluntary shutdown. The operating conditions, hydrogen consumption, and liquid product properties for the ten balances obtained are given in Table 4-11 together with the charge stock properties. The product yields on charge and the distillations of the liquid products are given in appendix Table B-4.

Figure 4-8 shows the fate of the functional hydrogen consumption in upgrading this SRC blend. The hydrogen consumed by heteroatoms is defined as the hydrogen which winds up in NH_3, H_2O, and H_2S. The hydrogen consumed by the C_1-C_5 production is the total hydrogen in these light products, and the hydrogen consumed by the C_6^+ is the remainder of the hydrogen consumed. About 75% of the hydrogen consumed goes toward increasing the hydrogen of the C_6^+ liquid product with smaller amounts going to the gaseous products. The production of light hydrocarbons (C_1-C_5) consumes increasingly more hydrogen as severity is increased. These results are similar to those reported last year for the Burning Star and Monterey SRC's (3-1).

Figure 4-9 shows that at least two-thirds of the hydrogen which winds up in heteroatom containing gases, is found in the water produced from the organically bound oxygen. The product ammonia accounts for most of the rest. This result is again similar to results reported last with other coal liquids (3-1).

Selected hydroprocessed products from this SRC were subjected to the fuels compatibility test described in last year's report (3-1, Section 3.3). The results are shown in Table 4-12.

Table 4-11

FIXED BED HYDRORPROCESSING OF COAL LIQUIDS
CHARGE: 33% BLEND WYODAK SRC/SOLVENT (76D-2917)
CATALYST: HDS-1441A (J-7278)
PROGRAM: 2418 UNIT: CT-146

	CHARGE	*MB-772*	*MB-773*	*MB-774*	*MB-775*	*MB-776*	*MB-777*	*MB-778*	*MB-779*	*MB-780*	*MB-781*
OPERATING CONDITIONS											
TEMPERATURE, °F	-	676	731	784	772	723	670	671	724	775	770
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.94	0.98	0.97	0.45	0.51	0.51	0.16	0.18	0.29	0.07
DAYS ON STREAM	-	1.6	2.6	3.6	4.8	6.0	7.2	8.8	10.4	11.9	13.4
LIQUID PRODUCT PROPERTIES											
GRAVITY, °API	-3.4	3.0	4.6	9.8	12.1	7.0	1.8	8.9	11.0	11.4	21.9
HYDROGEN, WT °/°	6.51	7.45	7.73	8.63	9.66	9.43	7.62	8.87	9.96	9.52	11.11
SULFUR, WT °/°	0.30	0.07	0.05	0.02	0.03	0.04	0.07	0.03	0.02	0.02	0.01
NITROGEN, WT °/°	0.99	0.78	0.66	0.42	0.32	0.49	0.79	0.51	0.27	0.35	0.03
OXYGEN, WT °/°	3.7	2.1	1.6	0.6	0.4	1.1	2.2	1.2	0.3	0.9	0.1
CCR, WT °/°	18.2	14.5	14.7	11.4	9.3	13.7	16.1	11.7	8.9	10.0	0.9
HETEROATOM REMOVAL,PCT											
SULFUR	-	77.4	83.4	93.1	89.6	87.5	77.5	90.2	94.9	94.5	95.4
OXYGEN	-	44.3	57.9	84.7	89.5	70.7	41.9	68.3	92.3	76.4	97.5
NITROGEN	-	22.7	35.1	60.0	68.6	51.2	22.0	49.6	74.1	65.7	97.2
HYDROGEN UTILIZATION,SCF/B											
H2 CONSUMED BY C1-C5	-	54	100	336	238	131	92	109	325	340	1061
H2 CONSUMED BY C6+	-	604	775	1318	2110	2026	744	1624	2309	1977	2782
H2 CONSUMED BY S	-	11	11	13	12	12	11	12	13	13	13
H2 CONSUMED BY N	-	35	55	93	107	80	34	77	115	102	151
H2 CONSUMED BY O	-	150	197	288	304	240	142	232	314	259	331
H2 CONSUMED TOTAL	-	855	1138	2047	2771	2488	1023	2055	3076	2692	4339

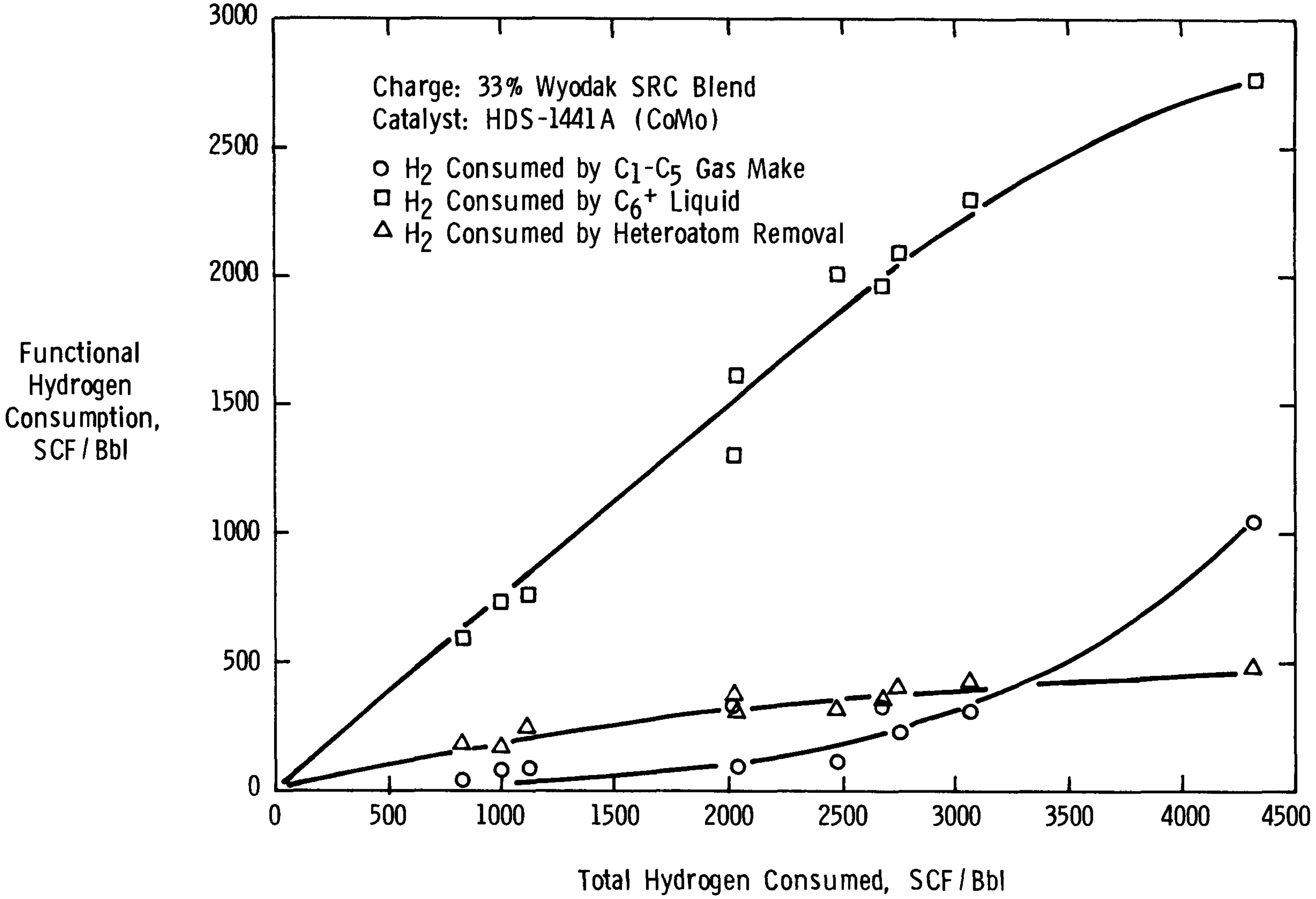


Figure 4-8. Hydrogen Utilization in Hydroprocessing of 33% Wyodak SRC Blend

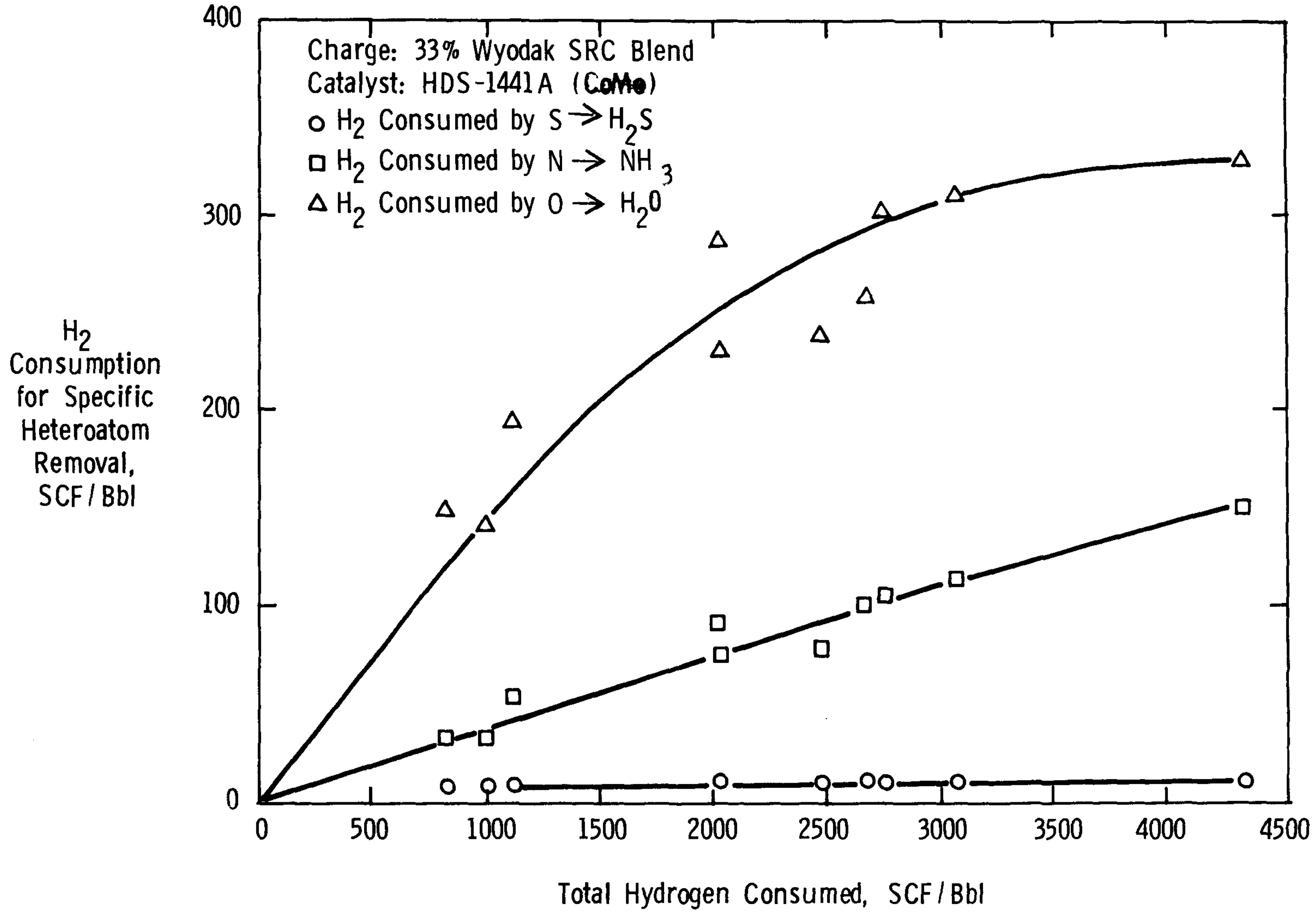


Figure 4-9. Hydrogen Consumption for Heteroatom Removal from 33% Wyodak SRC Blend

Table 4-12

COMPATIBILITY OF RAW AND HYDROPROCESSED 33% WYODAK SRC BLEND WITH PETROLEUM FUELS

	INCOMPATIBILITY SEDIMENT(1), VOL %	
PETROLEUM STOCK	NO. 2 DIESEL	EL PALITO NO. 6
Raw 2/1 Recycle Solv./SRC (76D2917)	12	14
HDT 2/1 Blend (CT-146-773)	5	2
HDT 2/1 Blend (CT-146-774)	3	0.3
HDT 2/1 Blend (CT-146-775)	0.3	1.0
HDT 2/1 Blend (CT-146-779)	13	11

---

(1) A 50/50 wt ratio of coal liquid/petroleum stock was agitated for six hours at 150°F followed by centrifuging for three hours at 150°F (Mobil Method 1006).

The raw 33% blend formed 12 and 14 vol % sediment with No. 2 diesel fuel and No. 6 fuel oil, respectively, Incompatibility sediment was reduced to 2-5 vol % in the mildest hydroprocessed sample (CT-146-773; 1138 scf/B H_2 consumption). Increasing severity to 2047 and 2771 scf/B H_2 consumption (CT-146-774 and CT-146-775) further reduced incompatibility sediment to 0.3-3 vol % with both No. 2 and No. 6 petroleum fuels. However, increasing the hydroprocessing severity to a hydrogen consumption of 3076 scf/B (CT-146-779) resulted in a product with the same level of incompatibility (11-13 vol % sediment) as the raw blend. This effect of a minimum in incompatibility sediment at an intermediate hydroprocessing severity was also observed for the 33 wt % blends of Burning Star SRC and Monterey SRC reported in the last annual report (3-1, Section 7.2).

The liquid products from the Wyodak study were also distilled to a 650°F cut point and the overhead and residual fractions analyzed for elemental composition. GEC analyses were performed on the 650°F$^+$ fractions (Table 4-13). At increasing severity of treatment, more hydrogen is added to the product and fewer heteroatoms remain. One result is an increase in the amount of distillate material. High-boiling saturates and aromatic oils (GEC Cuts 1-4) are also produced. Resins/asphaltenes (Cuts 5-7) occupy a somewhat more intermediate position and fluctuate along with changes in the balance between their conversion to distillate and GEC Cuts 1-4 and acquisition of products from Cuts 8-13. These trends are generally the same as those observed in the hydroprocessing studies of the two Illinois No. 6 SRC's.

These data have been fit to the simple kinetic scheme which was used for the Monterey SRC blend. The selectivity between the concentration of asphaltenes and resins as a function of hydrogen content of the liquid product is qualitatively the same as the data obtained with the Monterey SRC. This suggests the same reaction network should be applicable. A least squares fit of the data to equations 4-3 and 4-4 was used to determine the following kinetic constants.

Table 4-13

ELEMENTAL AND GEC ANALYSES OF HYDROPROCESSED 33% WYODAK SRC BLEND

	76D2917 CHARGE	MB-772	MB-773	MB-774	MB-775	MB-776	MB-777	MB-778	MB-779	MB-780
Operating Conditions										
Temperature, °F	-	676	731	784	772	723	670	671	724	775
LHSV, V/Hr/V	-	0.94	0.98	0.97	0.45	0.51	0.51	0.16	0.18	0.29
H_2 Pressure, psig	-	2000	2000	2000	2000	2000	2000	2000	2000	2000
Total Liquid Product										
Hydrogen, Wt %	6.51	7.45	7.73	8.63	9.66	9.43	7.62	8.87	9.96	9.52
Oxygen, Wt %	3.70	2.10	1.60	0.60	0.40	1.10	2.20	1.20	0.30	0.90
Nitrogen, Wt %	0.99	0.78	0.66	0.42	0.32	0.49	0.79	0.51	0.27	0.35
Sulfur, Wt %	0.30	0.07	0.05	0.02	0.03	0.04	0.07	0.03	0.02	0.02
650°F$^-$, Wt %	49.57	61.51	55.64	59.22	63.30	65.11	50.43	57.76	61.55	62.17
650°F$^+$, GEC Fractions										
Cut 1 (Saturates)	0.33	0.52	0.78	1.00	1.17	0.37	0.61	1.03	1.88	1.09
Cut 2-4 (Arom. Oils)	11.26	9.69	15.92	21.64	20.89	12.82	14.64	17.45	24.63	21.40
Cut 5-7 (Resin/Asph.)	12.72	13.13	13.80	10.44	8.78	11.81	15.84	12.77	8.21	9.33
Cut 8-12 (Polar Asph.)	26.12	15.15	13.86	8.10	5.86	9.89	18.48	10.99	3.73	6.01
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Elemental Analysis(a)										
IBP-650°F Cut										
Hydrogen, Wt %	7.84	8.67	9.06	9.72	10.53	9.80	8.85	9.91	10.60	10.38
Oxygen, Wt % (by diff.)	3.3	1.8	0.80	0.05	0.05	0.72	1.8	0.7	0.0	1.1
Nitrogen, Wt % (by diff.)	0.36	0.34	0.17	0.01	0.05	0.08	0.23	0.12	0.0	0.09
Sulfur, wt %	0.23	0.035	0.018	0.003	0.014	0.016	0.017	0.008	<0.002	0.005
650°F$^+$ Bottoms										
Hydrogen, Wt %	6.13	6.60	6.94	7.36	4.47	6.86	6.79	7.54	8.20	7.31
Oxygen, Wt %	4.1	2.6	2.6	1.4	1.0	1.8	2.6	2.0	0.8	0.6
Nitrogen, Wt %	1.61	1.49	1.28	1.01	0.78	1.25	1.36	1.04	0.72	0.78
Sulfur, Wt % (by diff.)	0.37	0.13	0.09	0.04	0.06	0.08	0.12	0.06	ca.0.05	0.04

(a) For each sample some elements were determined directly for one distillate cut and calculated by difference for the other cut.

Table 4-14

KINETIC CONSTANTS* FOR THE CONVERSION OF ASPHALTENES AND RESINS FROM A 33% WYODAK SRC BLEND

$$A \xrightarrow{k_{AR}} R \xrightarrow{k_{RO}} O$$

STEP	FREQUENCY FACTOR (k^o_{ij}), HR^{-1}	ACTIVATION ENERGY (E_{ij}), BTU/LB MOLE	DECAY CONSTANT (a), DAY^{-1}
A ⟶ R	8.06×10^5	33,052	0.0771
R ⟶ O	2.13×10^7	41,425	

$$* \; k_{ij} = k^o_{ij} \; e^{-at} \; e^{-E_{ij}/RT}$$

The fit of the model to the data is given in Figure 4-10.

The kinetic constants differ somewhat from those for the Monterey coal but are of the same order of magnitude. Wyodak SRC appears to age somewhat more slowly. This could be due to the high ash content (4600 ppm) of the Monterey SRC compared to the Wyodak (1000 ppm). Figure 4-11 compares the asphaltene conversion predicted using the kinetic constants in Table 4-10 and 4-14 for Monterey and Wyodak SRC, respectively. The small difference in conversion shown in this figure is within the accuracy of our data and suggests that the asphaltenes in these two samples are converted at an equivalent rate.

Comparison of SRC Hydroprocessing Results

The three SRC products discussed in this section are very similar in elemental and chemical (GEC) composition (Table 4-15).

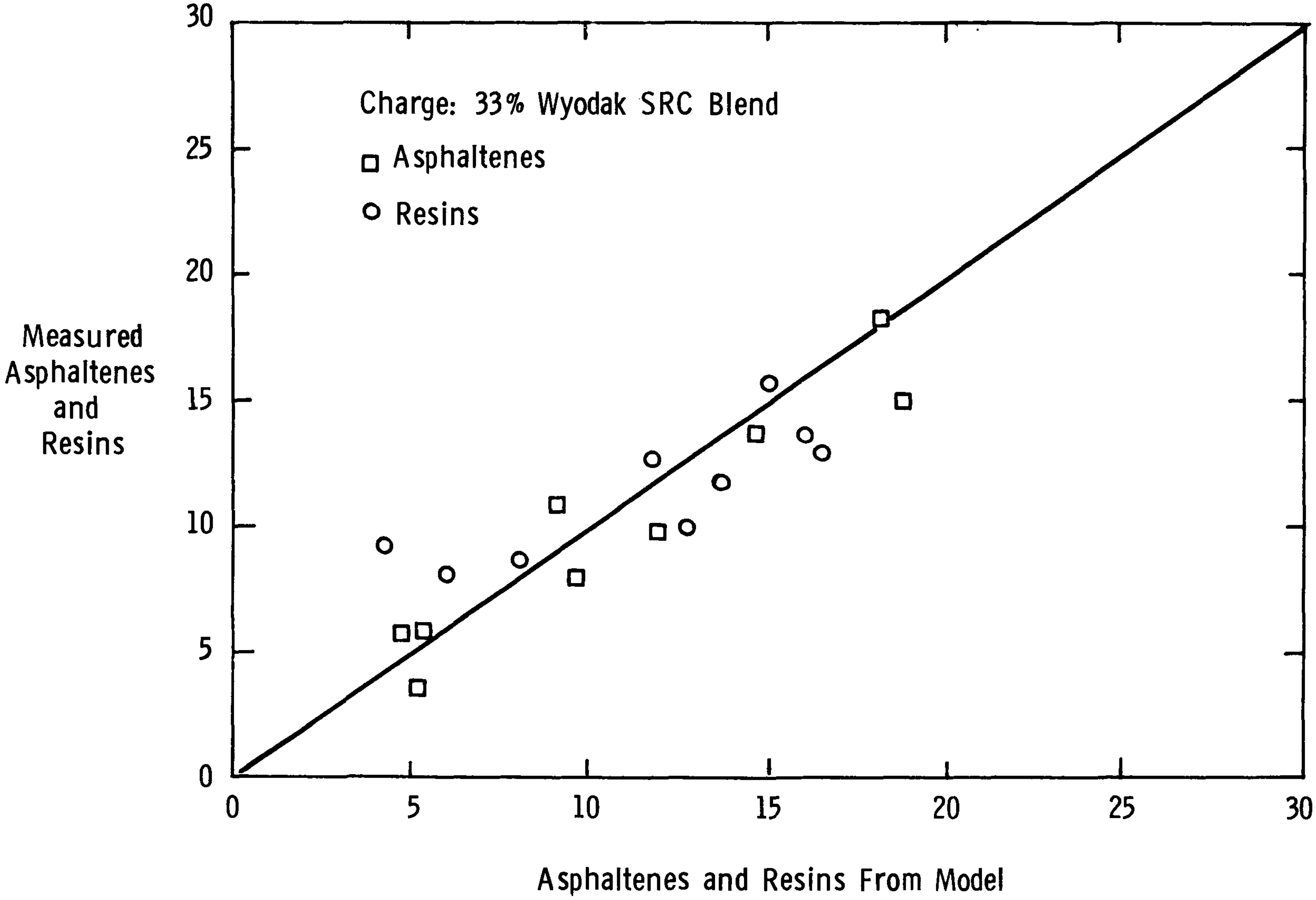


Figure 4-10. Fit of Model to Asphaltenes and Resins: 33% Wyodak SRC Blend

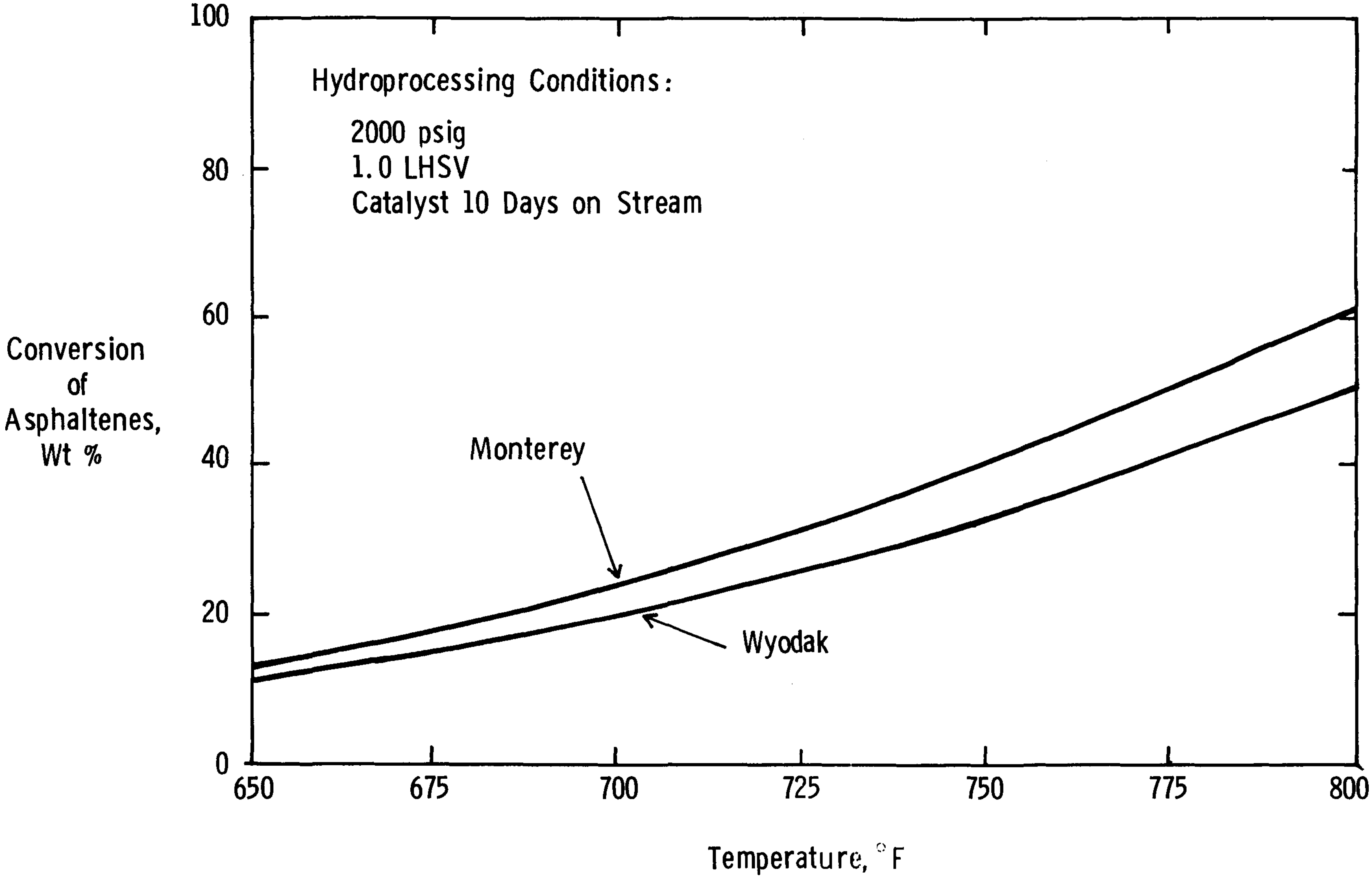


Figure 4-11. Comparison of Asphaltene Conversion for Monterey and Wyodak SRC

Table 4-15

COMPOSITION OF 33% SRC BLENDS

	BURNING STAR	MONTEREY	WYODAK
Properties			
Gravity, °API	-3.0	-2.7	-3.4
Hydrogen, wt %	6.84	6.93	6.51
Oxygen, wt %	3.91	4.1	3.70
Nitrogen, wt %	1.03	1.00	0.99
Sulfur, wt %	0.47	0.57	0.30
CCR, wt %	16.45	18.93	18.16
GEC Analysis, wt %			
Oils + Saturates + 650°F	57.51*	60.98	61.16
Resins (Cuts 5-7)	17.71	12.93	12.72
Asphaltenes (Cuts 8-12)	24.78	26.09	26.12

---

* Cut at 500°F instead of 650°F.

Table 4-16 gives a comparison of the liquid products from the hydroprocessing of these SRC blends over HDS-1441A catalyst. The results are given for two material balances at high and low severity for each blend. It can be seen that the products from the Burning Star sample are in general of higher quality than those from the Monterey or Wyodak blends. The Burning Star products are in general higher in hydrogen content and lower in nitrogen, oxygen and CCR content. It was noted in last year's report (3-1, Table 4-16) that the GEC fractions from the Wyodak and Monterey samples were 20-40% higher in molecular weight than those from the Burning Star sample. This fact may contribute significantly to the relative ease of processing Burning Star with this small pore catalyst.

Table 4-16

COMPARISON OF HYDROPROCESSED SRC BLENDS

2/1 BLEND	BURNING STAR		MONTEREY		WYODAK	
Material Balance	CT-199-441	-447	CT-199-578	-582	CT-146-773	-779
Operating Conditions						
Temperature, °F	731	729	729	727	731	729
Pressure, psig	2000	2000	2000	2000	2000	2000
LHSV, Hr^{-1}	0.98	0.21	0.99	0.22	0.98	0.18
H_2 Consumption, SCF/BBL	1986	2822	1047	2490	1138	3076
Liquid Product Properties						
API Gravity	9.6	15.5	6.1	13.1	4.6	11.0
Hydrogen, wt %	9.04	10.15	8.47	9.78	7.73	9.96
Nitrogen, wt %	0.58	0.22	0.74	0.34	0.66	0.27
Oxygen, wt %	0.70	0.2	1.6	1.0	1.6	0.30
Sulfur, wt %	0.11	0.06	0.17	0.09	0.05	0.02
CCR, wt %	8.64	4.30	12.88	6.29	14.69	8.88

The Conradson Carbon Residue (CCR) in the SRC blends can be attributed to the SRC fraction alone, and its reduction is a good indication of conversion of the SRC. Since CCR is a measure of the coke precursors, it is a useful parameter in hydroprocessing. Figure 4-12 shows that the CCR can be correlated with the asphaltenes and resins in the SRC blends for all three SRC's studies. The Wyodak samples appear to have a slightly higher CCR content at a given asphaltene plus resins content. Once again, the higher molecular weight of the Wyodak SRC may give it high coking tendencies.

The aromatic oils + saturates + 650°F$^-$ fractions of the SRC correspond roughly to the hexane soluble material. The concentrations of this lump can be correlated very well with the oxygen content of the liquid products, Figure 4-13. This correlation is independent of SRC source and stresses the importance of oxygen removal in the conversion of SRC.

Figure 4-14 shows the oxygen content of the resins and asphaltene GEC fractions for the hydroprocessed Burning Star and Monterey SRC's. The oxygen content of

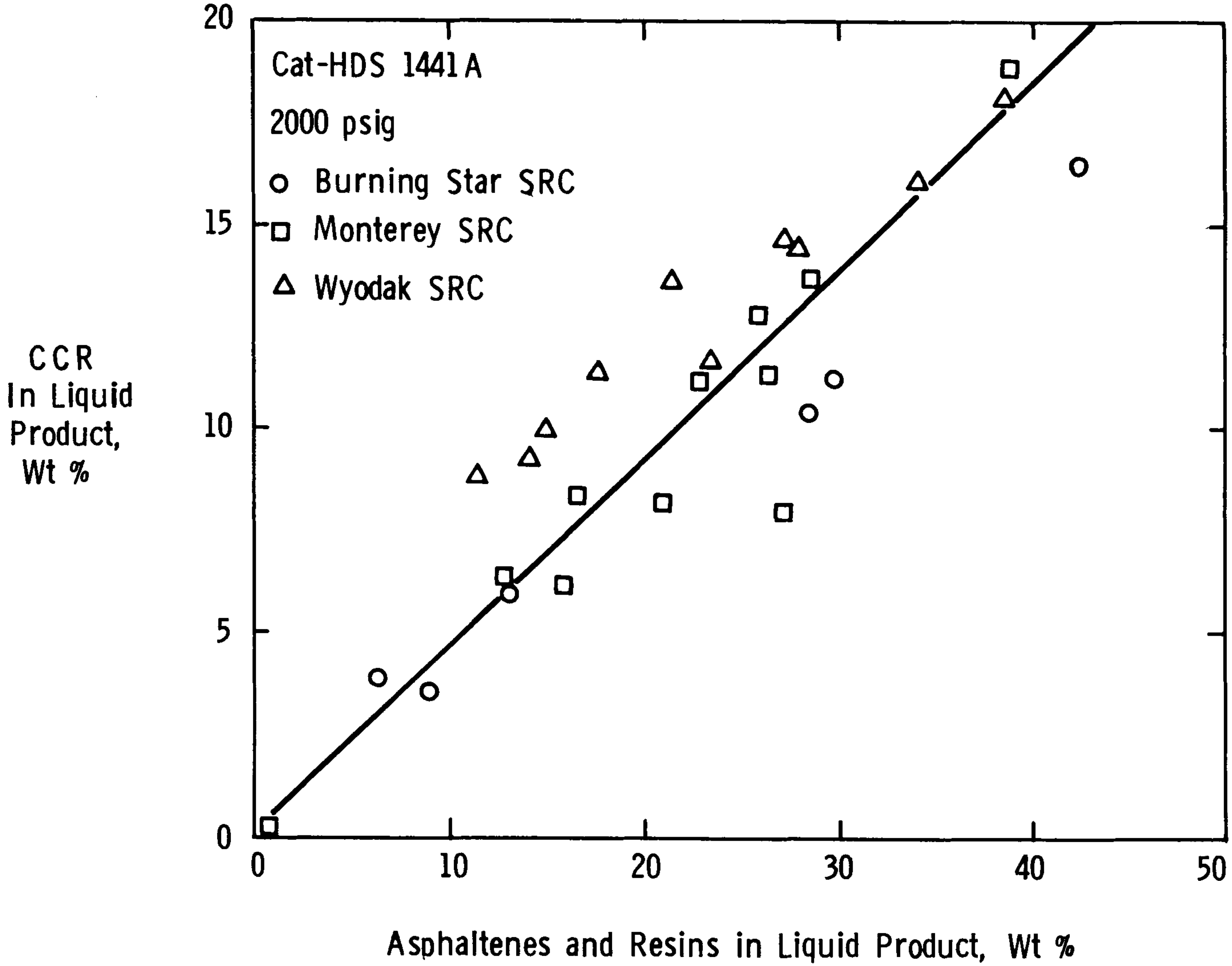


Figure 4-12. CCR as a Function of Asphaltenes Plus Resins for Hydroprocessed SRCs

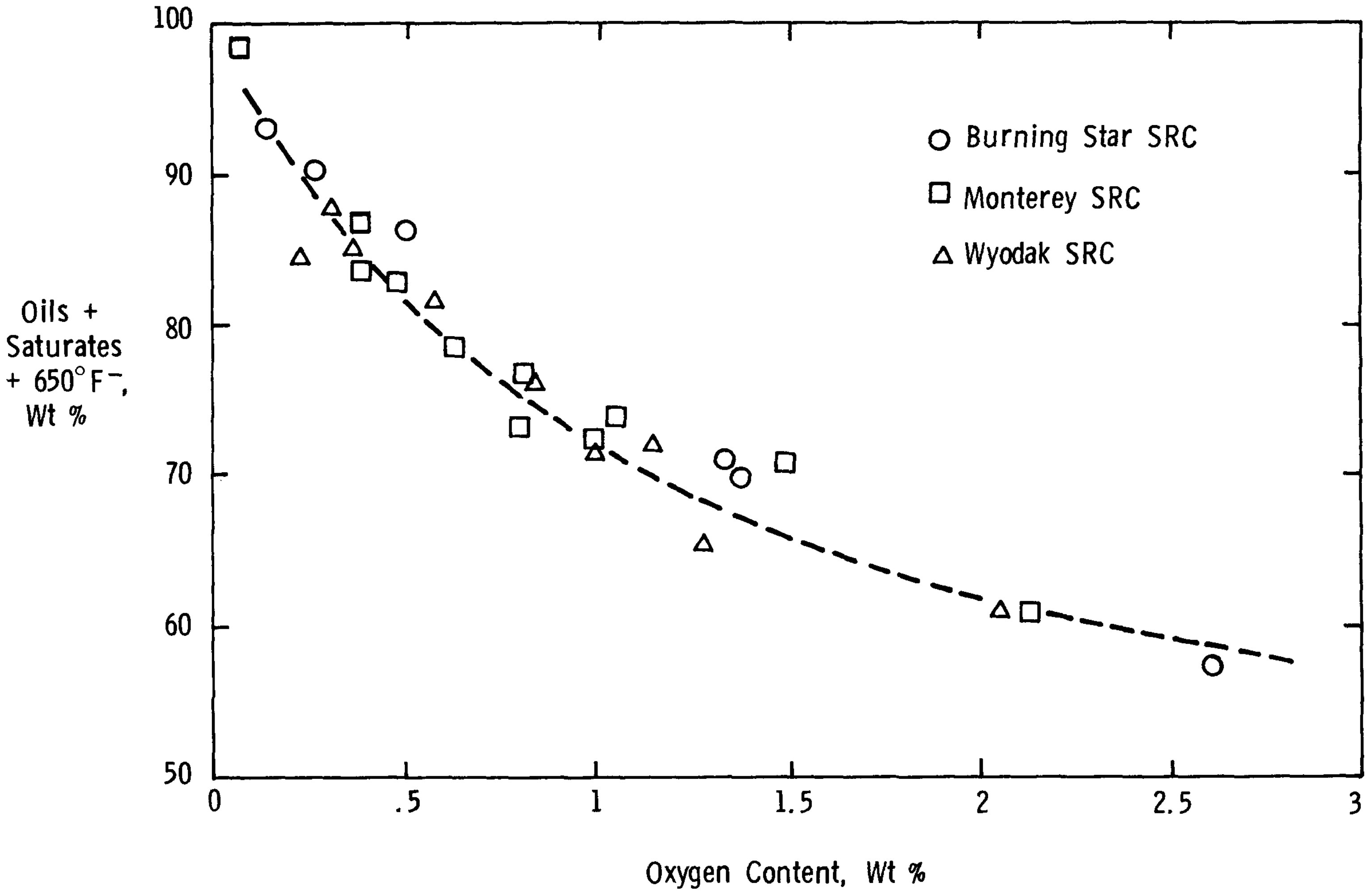


Figure 4-13. Oil Conversion as a Function of Oxygen Content for Hydroprocessed SRCs

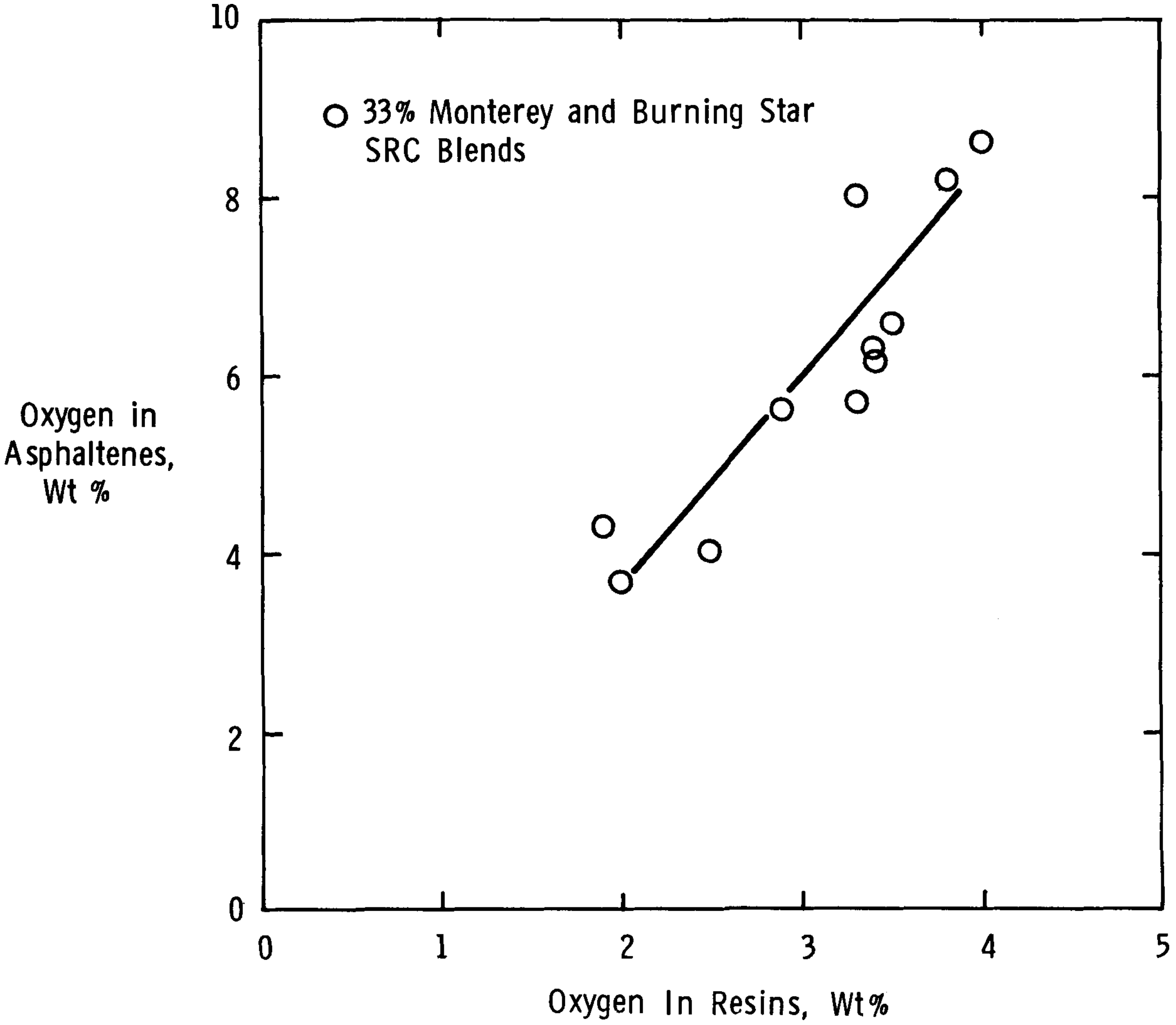


Figure 4-14. Oxygen Content of Resin and Asphaltenes in Hydroprocessed SRC

resins and asphaltenes in the charge blends are about 4.0 and 8.5 wt % respectively. Hydroprocessing reduces these concentrations. However, the oxygen content of the remaining asphaltenes and resins never goes below about 4 wt % and 2 wt % respectively. Apparently, removal of more oxygen drops these molecules to a lower GEC fraction.

Effect of Concentration on Hydroprocessing SRC

One of the goals of this work was to hydroprocess high concentration blends of SRC. Based on the viscosity-temperature studies, thermal stability experiments, and blend preparation work, we decided to increase the charge SRC concentration in stages. This would provide useful information on the effect of concentration on SRC hydroprocessing.

The SRC chosen for this study was the second of the Illinois No. 6 Monterey SRC's received from Wilsonville. This material, unlike the first batch received, met the low ash specifications (< 0.1 wt %) for SRC. Complete properties of this SRC are included in Table 4-17, along with the properties of the recycle solvent used in preparing all the blends used in this study. A 33 wt % blend of SRC (i.e., 1 part SRC per 2 parts solvent) was prepared in a 20-gallon quantity by heating in a hot box (200°F) with continuous stirring. The SRC was added slowly over a period of days to assure thorough dissolution. Each of the other higher concentration blends were prepared at the unit by mixing SRC and recycle solvent in the feed tank as described in Section 4.2. The temperature was increased at a rate of 50°F/hr to the desired blending temperature, and held there for at least 6-12 hours to allow complete melting of the SRC. After preparing the blend in the feed tank, the gear pump was used to recirculate the blend and achieve homogeneous mixing. After approximately 6 hours of mixing,the gear pump was used to transfer the blend to the charge reservoir in preparation for loading the charge weigh cell. Due to the limited capacity of the charge reservoir (3-4 gallons), several blends were often prepared for a given study. All of these runs were performed in our modified unit (CT-146) at a constant reactor pressure of 2000 psig.

Table 4-17

ANALYSES OF CHARGE STOCK BLEND COMPONENTS

Mobil ID	MONTEREY SRC 76D 2155	RECYCLE SOLVENT 77D 391
Gravity, °API	-15.3	- -
Hydrogen, wt %	6.22	7.42
Oxygen, wt %	4.0	3.3
Sulfur, wt %	0.70	0.32
Nitrogen, wt %	1.75	0.61
Basic Nitrogen, wt %	-	0.44
% Aromatic Carbon	93	71
CCR, wt %	-	1.08
Ash, wt %	.085	-
Trace Metals (ppm)		
Sodium	1200	-
Potassium	100	-
Calcium	<100	-
Vanadium	3.9	-
Iron	146	-
Nickel	3.4	-
Lead	200	-
Distillation, °F	D-1160-1	D-2887
IBP	681	354
5%	817	388
10%	898	397
30%	(1031 @ 22%)	441
50%	-	476
70%	-	527
90%	-	630
95%	-	673
FBP	-	917

The catalyst used in each of these studies was a large pore NiMo/Al_2O_3, Harshaw 618X. Physical properties for this catalyst are given in Table 4-18, along with the properties of other smaller pore catalysts used in this work. We had experienced post-reactor plugging problems and two phase products with the small pore CoM/Al_2O_3 catalyst (HDS-1441A) when processing high residual content blends (Section 4.4). These were overcome by going to this large pore catalyst.

Table 4-18

HYDROPROCESSING CATALYSTS

Catalyst	HARSHAW 618X	KETJEN 153S	CYANAMID HDS-1441A
Mobil ID	J-7423	J-7709	J-7278
Metals	NiMo	NiMo	CoMo
Avg. Pore Diameter, Å	172	81	71
Surface Area, m^2/g	140	255	323
Pore Volume, cc/g	0.602	0.52	0.57
Real Density, g/cc	3.603	3.55	3.67
Particle Density, g/cc	1.137	1.24	1.19
Pore Size Distribution, cc/g			
<50 Å	0.040	0.043	0.114
50-100 Å	0.062	0.426	0.441
100-200 Å	0.461	0.032	0.005
200-300 Å	0.024	0.006	0.000
>300 Å	0.013	0.015	0.010
Total	0.60	0.52	0.57

A brief history of the high concentration runs completed or attempted during this study is outlined below:

Wt % SRC	Result
33	Successful
70	Successful
100	Unsuccessful
70/80/90	Blown sight glass prevented 100% attempt.
90	Successful
100	Unsuccessful

The details and results from each of these runs are given in the following sections. A summary of the effects of concentration on hydroprocessing the Monterey SRC concludes this section.

33% Monterey SRC. This run with 33 wt % Monterey SRC served as the base case for both the concentration effect study and as a comparison with a short contact time Monterey SRC (Section 4.4). Nine material balances were completed while operating at conditions ranging from 622-788°F, 0.25-0.95 LHSV, and 2000 psig. The operating conditions, hydrogen utilization, and liquid product properties for these balances, along with the charge stock properties, are given in Table 4-19. Table B-6 contains additional information on the product yields on charge and the liquid product distillations. No operating problems were encountered during this run.

A comparison of the Monterey blend used in this study with the previous 33 wt % blend made with a high ash containing Monterey SRC is shown below.

	33% Blends of Monterey SRC	
	76D-1669	77D-1039
Properties:		
Gravity, °API	-2.7	-4.9
Hydrogen, wt %	6.93	7.00
Oxygen, wt %	4.1	3.70
Nitrogen, et %	1.00	1.12
Sulfur, wt %	0.57	.58
CCR, wt %	18.93	17.14
Ash, wt %	0.17	0.05

These blends are quite similar except for the high ash content of 76D-1669.

Comparing balances obtained from each study at equivalent processing severities, hydroprocessing with the large pore catalyst generally gives a product containing a similar amount of hydrogen with a lower heteroatom content. The small pore catalyst usually consumes more hydrogen in producing a higher gas make and less hydrogen in saturating the liquid product.

The first and last material balances (MB-827 & 835) in this study were made at similar conditions at 1.6 and 10.1 days on-stream respectively. The data from these balances show an increase in nitrogen and oxygen content and a decrease in hydrogen and API gravity over this 8.5 day period. This is indicative of the catalyst aging in this run.

Table 4-19

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33% BLEND MONTEREY SRC/SOLVENT (77-1039)
CATALYST: HARSHAW 618X (J-7242)
PROGRAM: 2501 UNIT: CR-146

	CHARGE	MB-827	MB-828	MB-829	MB-830	MB-831	MB-832	MB-833	MB-834	MB-835
OPERATING CONDITIONS										
TEMPERATURE, °F	-	724	777	622	623	724	776	788	775	730
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.47	0.53	0.53	0.25	0.29	0.28	0.95	1.04	0.49
DAYS ON STREAM	-	1.6	2.6	3.6	4.7	6.3	7.8	8.6	9.1	10.1
LIQUID PRODUCT PROPERTIES										
GRAVITY, °API	-4.9	8.7	12.2	-0.6	1.1	10.0	15.0	7.9	5.2	6.9
HYDROGEN, WT °/°	7.00	9.08	9.17	7.41	7.79	9.33	9.85	8.90	8.55	8.60
SULFUR, WT °/°	0.580	0.092	0.040	0.266	0.187	0.038	0.022	0.059	0.073	0.094
NITROGEN, WT °/°	1.120	0.480	0.320	0.900	0.590	0.320	0.170	0.450	0.570	0.550
OXYGEN, WT °/°	3.7	1.3	1.0	3.0	2.9	0.7	0.4	0.9	1.6	1.7
CCR, WT °/°	17.14	8.06	6.15	14.15	11.11	6.13	3.32	7.72	9.56	8.24
K.V. AT 40°C	13.41	2.51	1.85	7.86	5.95	2.29	-	-	3.32	3.09
AROMATIC CARBON, °/°	79	-	-	70	-	46	43	-	60	54
HETEROATOM REMOVAL, PCT										
SULFUR	-	84.7	93.4	54.7	68.2	93.7	96.4	90.3	87.9	84.2
OXYGEN	-	66.2	74.2	19.8	22.6	81.9	89.7	76.7	58.6	55.2
NITROGEN	-	58.8	72.7	20.6	48.0	72.7	85.5	61.6	51.3	52.1
HYDROGEN UTILIZATION, SCF/B										
H2 CONSUMED BY C1-C5	-	163	351	30	24	308	371	269	299	120
H2 CONSUMED BY C6+	-	1381	1323	239	512	1443	1780	1148	922	1024
H2 CONSUMED BY S	-	23	25	15	18	25	26	24	24	23
H2 CONSUMED BY N	-	105	130	37	85	129	152	110	91	93
H2 CONSUMED BY O	-	228	255	68	78	282	308	264	201	190
H2 CONSUMED TOTAL	-	1899	2084	388	717	2186	2637	1815	1537	1450

Catalyst aging and the limited amount and range of the data makes mathematical modeling difficult. However, the second order heteroatom removal model described in last year's annual report (3-1, Section 6.1) was applied to the data. The fit of this model to the data for the three heteroatoms was good. The kinetic constants and average deviations determined by the modified Marquardt fitting routine are given in Table 4-20 below. The activation energies are somewhat lower than those reported last year for the distillate coal liquids. This could be indicative of diffusional limitations with this higher molecular weight charge.

Table 4-20

CONSTANTS FOR KINETIC MODEL FOR HETEROATOM REMOVAL

Feed: 33 wt % Monterey SRC Blend

Catalyst: Harshaw 618X

SECOND ORDER RATE EQUATION *

	k, HR^{-1}	E, BTU/LB MOLE	$k_{750°F}$, HR^{-1}	AVG. DEVIATION WT %
Nitrogen	3.88×10^5	31,682	0.734	0.048
Oxygen	5.84×10^8	51,467	0.295	0.215
Sulfur	1.74×10^8	40,856	7.25	0.010

$$* \ \frac{dC_i}{dt} = - k e^{-E/RT} C_i^2$$

The charge blend and five products from this study were distilled into an IBP-650°F and a 650°F$^+$ fraction and analyzed separately for hydrogen and heteroatom contents (Table 4-21). The data show the hydrogen content of the 650°F$^-$ fraction. The oxygen content of the two fractions are roughly equivalent, whereas the nitrogen and sulfur content are significantly higher in the heavier fraction. Sulfur is uniformly low (<.2 wt %) in the 650°F$^+$ fraction of the hydroprocessed products.

Table 4-21

ANALYSES OF TOTAL LIQUID PRODUCT AND DISTILLATION CUTS FROM 33% MONTEREY SRC BLEND

	CHARGE 77D1039	MB-828	MB-831	MB-832	MB-834	MB-835
Process Conditions						
Temperature, °F	-	777	724	776	775	730
LHSV	-	0.53	0.29	0.28	1.04	0.49
H_2 Consumed, scf/B	-	2096	2212	2633	1554	1448
Days On-Stream	-	2.6	6.3	7.8	9.1	10.1
Material Balance, wt %	-	105.3	112.6	99.1	108.2	99.7
Total Liquid Product						
Hydrogen, wt %	7.00	9.17	9.33	9.85	8.82	8.53
Oxygen, wt %	3.7	1.0	0.7	0.4	1.6	1.7
Nitrogen, wt %	1.12	0.32	0.32	0.17	0.57	0.55
Sulfur, wt %	0.58	0.040	0.038	0.022	0.073	0.094
650°F$^-$, wt %	48.35	60.72	61.25	66.13	55.47	57.75
IBP-650°F Cut						
Hydrogen, wt %	7.55	9.74	9.97	10.18	9.07	9.24
Oxygen, wt %*	3.5	1.1	0.7	0.4	1.7	1.6
Nitrogen, wt %*	0.63	0.09	0.05	0.06	0.13	0.17
Sulfur, wt %	0.272	<0.002	<0.002	0.003	<0.002	0.008
650°F$^+$ Bottoms						
Hydrogen, wt %	6.11	7.68	8.31	8.10	7.37	7.54
Oxygen, wt %	3.9	0.9	0.7	0.4	1.5	1.9
Nitrogen, wt %	1.58	0.68	0.75	0.38	1.09	1.07
Sulfur, wt %	0.25	0.10	0.09	0.06	0.16	0.2

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* - Calculated from wt % hydrogen in total liquid product and 650°F$^+$ fraction.

Table 4-22

ELEMENTAL ANALYSES OF GEC FRACTIONS FROM HYDROPROCESSED 33% MONTEREY SRC BLEND

	77D1039 CHARGE	MB-828	MB-832
Temperature, °F	-	777	776
LHSV	-	0.53	0.28
Pressure, psig	-	2000	2000
650°F$^-$, wt %	48.35	60.72	61.25
650°F$^+$ Residue			
Cut 1 (sats.)	0.36	1.39	1.62
Cut 2-4 (arom. oils)	10.87	24.85	24.16
Cut 5-7 (resins/asph.)	15.92	9.91	10.37
Cut 8-13 (polar asph.)	24.50	3.13	2.60
Total	100.00	100.00	100.00
Elemental Analyses			
Hydrogen, wt %			
650°F Residue	6.11	7.68	8.31
Cut 1	(a)	12.84	13.02
Cut 2-4	6.84	8.53	8.79
Cut 5-7	6.34	6.62	7.09
Cut 8-12	5.67	5.54	6.28
Oxygen, wt %			
650°F Residue	3.9	0.9	0.7
Cut 1	(a)	(a)	(a)
Cut 2-4	1.0	0.9	0.8
Cut 5-7	3.8	2.0	2.5
Cut 8-12	6.3	3.7	4.0
Nitrogen, wt %			
650°F Residue	1.58	0.68	0.75
Cut 1	(a)	(a)	(a)
Cut 2-4	-	0.18	0.24
Cut 5-7	2.55	1.85	1.73
Cut 8-12	1.29	1.33	1.13
Sulfur, wt %			
650°F Residue	0.25	0.10	0.09
Cut 1	(a)	(a)	(a)
Cut 2-4	0.59	0.07	<0.03
Cut 5-7	0.75	<0.03	0.03
Cut 8-12	1.03	(a)	(a)

(a) - Analysis not determined.

Hydrogenation of the liquid product showed no preference in the two cuts obtained. Although the absolute percent hydrogen was higher, the IBP-650°F cut showed the same relative percent increase in hydrogen as the $650°F^+$ bottoms.

Table 4-22 presents the GEC compositional analyses of the $650°F^+$ bottoms for the Monterey charge and two of the products from this study. Detailed elemental analyses of these fractions are also included. As was shown in Section 4.3-1, the oxygen and sulfur content is highest in the highest GEC fractions, while the nitrogen content is highest in the resins (Cuts 5-7). The hydrogen content decreases with increasing fraction number.

Demetalation. Demetalation of coal liquids is important both with respect to meeting turbine fuel trace metals specifications and to aging of the catalyst. Five metals -- vanadium, sodium, calcium, potassium, and lead -- are of major concern to turbine users because they may cause deposition on or corrosion of the turbine blades. H-Coal and SRC coal liquids also tend to have high concentrations of both iron and titanium which may accelerate catalyst aging during hydroprocessing. Table 4-23 shows data obtained on trace metals removal during this study.

Table 4-23

TRACE METALS ANALYSIS IN HYDROPROCESSED 33% MONTEREY SRC BLEND

	H_2 CONSUMPTION (scf/B)	V	Fe	Ca	Na	K	TOTAL
CHG	-	1.5	98	4.2	3.4	1.4	108.5
MB-829	388	0.6	32	13	2.3	1.5	49.4
MB-835	1448	0.1*	10	0.5	0.8	0.2	1.6
MB-832	2633	<.1	1.5	1.8	1.0	0.2	4.5

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* Estimated value.

Although hydroprocessing removed most of the metals from this SRC blend, the sodium, potassium and calcium concentrations are still above the specifications of most turbine manufacturers (3.2, Section 4).

70% Monterey SRC. A blend of 70% Monterey SRC in recycle solvent was hydroprocessed over Harshaw 618X NiMo/Al_2O_3 catalyst in a 15 day run. Two separate blends were prepared in our 4 gallon blend tank to provide the charge stock for this study. The elemental analyses of the two blends and the SRC from which they were prepared, are given in Table 4-24. The blends are higher in oxygen content than the SRC. Even though they contain 30% recycle solvent containing 7.42 wt % hydrogen, they are no higher in hydrogen content than the SRC. Apparently, in the preparation of these blends, they were oxidized and light material was lost. A GEC analysis shown below also suggests degradation of these blends.

GEC Analysis Of 70% Monterey SRC Blend
Mobil ID-J-8864

GEC Cuts	
Saturates	0.99
Oils	19.99
Resins/Asphaltenes	14.33
Polar Asphaltenes	64.69
(Non-eluted polar asphaltenes)	(30.77)

This analysis was performed three times with essentially identical results. The non-eluted asphaltenes were at least twice as high as any sample of SRC we have yet analyzed, again indicating oxidation and degradation.

The operating conditions, hydrogen utilization and liquid product properties from this run are given in Table 4-25. Table B-7 in the Appendix B gives the product yields and product distillation data. Although the charge was apparently degraded, the products show no adverse effects. The total hydrogen consumption ranged from 1400 to 4039 scf/B. At the most severe conditions (MB-772) 86% removal of the nitrogen was achieved with even greater removal of the oxygen (91%) and sulfur (96%). This product contains almost 10 wt % hydrogen and its viscosity is 1.79 cs at 100°C. The feed contained only 6.15 wt % hydrogen and was solid at 100°C.

Table 4-24

CHARGE STOCK COMPARISONS OF 70% MONTEREY SRC BLENDS

SAMPLE	SRC	70% SRC BLEND 1	70% SRC BLEND 2	AVG. USED IN MATERIAL BALANCE CALC.
Mobil Identification	76D2155	J8864	J8864-1	
<u>Elemental Analyses</u>				
Gravity, °API	-15.3	-13.2	-13.4	-13.3
Hydrogen, wt %	6.22	6.07	6.29	6.15
Oxygen, wt %	4.0	5.2	4.97	5.1
Sulfur, wt %	0.70	0.89	0.94	0.92
Nitrogen, wt %	1.75	1.64	1.66	1.65
CCR, wt %	-	-	38.17	-
% Aromatic Carbon	-	82	-	-

Table 4-25

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 70% BLEND MONTEREY SRC/SOLVENT (J-8864)
CATALYST: HARSHAW 618X (J-7242)
PROGRAM: 2539 UNIT: CT-146

	CHARGE	MB-852	MB-853	MB-854	MB-855	MB-856	MB-857	MB-858	MB-859
OPERATING CONDITIONS									
TEMPERATURE, °F	-	726	768	772	723	720	772	781	779
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.44	1.17	0.20	0.30	0.14	0.13	0.41	0.42
DAYS ON STREAM	-	2.2	3.2	4.5	6.0	8.5	11.5	13.6	14.4
LIQUID PRODUCT PROPERTIES									
GRAVITY, °API	-13.3	1.9	2.3	9.4	3.9	6.0	14.2	5.2	6.1
HYDROGEN, WT °/°	6.15	8.18	8.16	9.62	8.89	9.54	9.98	8.67	8.77
SULFUR, WT °/°	0.920	0.340	0.253	0.050	0.151	0.101	0.041	0.121	0.120
NITROGEN, WT °/°	1.650	0.950	0.720	0.440	0.830	0.530	0.250	0.790	0.830
OXYGEN, WT °/°	5.1	2.2	1.7	0.7	1.9	1.1	0.5	1.6	1.5
CCR, WT °/°	38.17	18.36	21.32	8.34	15.59	11.34	4.43	16.86	14.22
K.V. AT 100°C	-	45.70	118.70	2.95	275.50	-	1.79	61.32	36.99
AROMATIC CARBON, °/°	82	57	60	44	51	44	42	48	53
HETEROATOM REMOVAL,PCT									
SULFUR	-	64.5	73.7	94.9	84.5	90.4	96.0	87.6	88.1
OXYGEN	-	59.1	67.9	86.9	65.1	80.9	90.9	71.1	73.1
NITROGEN	-	44.7	58.3	75.2	52.3	72.0	86.2	54.8	54.0
HYDROGEN UTILIZATION,SCF/B									
H2 CONSUMED BY C1-C5	-	216	171	553	312	1055	902	396	587
H2 CONSUMED BY C6+	-	1376	1317	2247	1896	2078	2351	1647	1687
H2 CONSUMED BY S	-	29	34	43	39	41	44	40	40
H2 CONSUMED BY N	-	126	164	211	147	202	242	154	152
H2 CONSUMED BY O	-	300	345	441	330	410	461	361	371
H2 CONSUMED TOTAL	-	2048	2030	3495	2724	3787	4001	2598	2836

The first and last material balances at 2.2 and 14.4 days on-stream respectively can give some indication of catalyst aging. These runs were made at approximately the same space velocity, but the last was made at 53°F higher temperature. The higher hydrogen consumption and heteroatom removal rate for the last balance indicate that the activity loss is somewhat less than 53°F over this 12.2 day period. The catalyst was removed from the reactor after 15 days and analyzed to determine the effects of this operation.

Harshaw 618X (J-7243)

	Fresh	Aged 15 Days
Surface Area, m^2/g	140	67
Real Density, g/cc	3.603	2.489
Particle Density, g/cc	1.137	1.649
Pore Volume, cc/g	602	.205
Avg. Pore Diameter, Å	172	122
Coke, wt %	-	17.6
Sulfur, wt %	-	4.08
Iron, ppm	-	4000
Calcium, ppm	-	1000
Sodium, ppm	-	900
Potassium, ppm	-	500

The results show a 52% decrease in surface area and a 66% decrease in pore volume over this period. A significant deposition of coke (17.6 wt %) and metals was also observed.

Properties of the IBP-650°F and the 650°F$^+$ fractions for three products obtained during this study are listed in Table 4-26, along with the GEC analyses of the bottom fractions. Considerable upgrading of the two fractions is shown with increasing severity. At the highest severity condition (i.e., 772°F, 0.12 LHSV, 2000 psig), 56 wt % of the product is contained in an IBP-650°F distillate, indicating an SRC conversion of over 40% to 650°F$^-$material. The 650°F$^-$ material which it produced is low in nitrogen and sulfur and except perhaps for its relatively low hydrogen, would probably be an acceptable turbine fuel.

Table 4-26

GEC AND ELEMENTAL ANALYSES OF OVERHEAD AND BOTTOM FRACTIONS FROM HYDROPROCESSED 70% MONTEREY SRC BLEND

	CHARGE STOCK**			
	J8864-1	MB-852	MB-854	MB-857
Temperature, °F	-	726	772	772
LHSV	-	0.44	0.20	0.13
Pressure, psig	-	2000	2000	2000
H_2 Consumed, scf/B	-	2048	3495	4001
<u>Total Liquid Product</u>				
H, wt %	6.15	8.18	9.62	9.98
O, "	5.10	2.17	0.72	0.51
N, "	1.65	0.95	0.44	0.25
S, "	0.92	0.34	0.05	0.04
650°F$^-$, wt %	-	38.75	51.34	56.20
<u>650°F$^+$ Residue</u>				
Cut 1 (saturates)	1.14	0.66	2.90	3.11
Cut 2-4 (arom. oils)	21.43	14.32	28.84	29.70
Cut 5-7 (resins/asph.)	15.69	20.27	12.74	7.69
Cut 8-13 (polar asph.)*	61.74	26.00	4.18	3.30
Total	100.00	100.00	100.00	100.00
<u>IBP-650°F Cut</u>				
H, wt %	-	9.32	9.37	10.64
O, " (by diff.)	-	1.2	0.8	0.6
N, "	-	0.21	0.12	0.08
S, "	-	0.034	0.015	0.014
<u>650°F$^+$ Bottoms</u>				
H, wt %	-	6.72	7.92	8.51
O, "	-	2.8	0.6	0.4
N, "	-	1.71	0.78	0.51
S, " (by diff.)	-	0.53	0.087	0.073

---

*Cut 13, wt %	26.77	5.80	0.59	1.30

**No distillation done on charge stock.

Elemental analyses of the GEC fractions (Table 4-27) show much the same trend observed with the 33% blend of this Monterey SRC (Table 4-22). The hydrogen content decreases and the oxygen increases with increasing GEC fraction number. The nitrogen is highest in the resins fraction (Cuts 5-7).

70-90% Monterey SRC. After several unsuccessful attempts at running 100% SRC, we dropped back to the 70% concentration and made a run in which we increased the SRC concentration in 10% increments. Three blends of 70, 80 and 90% SRC were prepared sequentially in our blend tank and charged to the units. Properties of these blends are given in Table 4-28. The hydrogen content of the blends decreases and the density increases with increasing SRC concentrations. The remaining properties do not reflect the increasing SRC concentration. A blend tank temperature of up to 450°F was necessary for blending and pumping of these liquids. Apparently some solvent was lost at these temperatures. A six day run with five material balances was completed before the unit was shut down due to failure of the high pressure, low temperature sight glass gasket. This problem is discussed in Section 4.2.

The operating conditions, hydrogen utilization, and liquid product properties are given in Table 4-29 together with the properties of the three charge stocks employed. Appendix Table B-8 includes the product yields on charge and the distillations of the liquid products. The following table shows a comparison of balances from the 70 and 80% SRC runs, and the 80 and 90% SRC runs at equivalent processing conditions:

Table 4-27

ELEMENTAL ANALYSES OF GEC FRACTIONS FROM HYDROPROCESSED 70% MONTEREY SRC BLEND

	CHARGE STOCK** J8864-1	MB-852	MB-854	MB-857
Temperature, °F	-	726	772	772
LHSV	-	0.44	0.20	0.13
Pressure, psig	-	2000	2000	2000
Elemental Analyses				
Hydrogen, wt %				
650°F Residue	-	6.72	7.92	8.51
Cut 1	13.63	13.71	13.02	13.66
Cut 2-4	6.81	6.38	8.28	8.70
Cut 5-7	6.46	6.81	6.26	5.76
Cut 8-12	5.29	5.60	5.06	(a)
Oxygen, wt %				
650°F Residue	-	2.8	0.6	0.4
Cut 1	0	0	0	0
Cut 2-4	1.3	0.6	0.6	0.2
Cut 5-7	4.7	2.6	1.8	1.3
Cut 8-12	7.9	5.1	4.1	3.5
Nitrogen, wt %				
650°F Residue	-	1.71	0.78	0.51
Cut 1	0	0	0	0
Cut 2-4	0.34	0.53	0.23	0.13
Cut 5-7	2.64	2.13	2.20	2.02
Cut 8-12	1.87	1.90	1.61	1.54
Sulfur, wt %				
650°F Residue	-	0.53	0.087	0.073
Cut 1	0	0	0	0
Cut 2-4	0.57	0.14	0.06	0.08
Cut 5-7	0.87	0.21	0.21	0.19
Cut 8-12	1.19	0.04	(a)	(a)

(a) - Analysis not determined.

**No distillation done on charge stock.

Table 4-28

CHARGE STOCK PROPERTIES OF
70, 80, AND 90% MONTEREY SRC BLENDS

Mobil Identification	J8977	J8978	J8979
% SRC	70	80	90
<u>Elemental Analyses</u>			
Gravity, °API	-15.8	-15.9	-16.3
Hydrogen, wt %	6.40	6.23	6.10
Sulfur, wt %	0.84	0.81	0.86
Nitrogen, wt %	2.01	1.91	1.72
Oxygen, wt %	3.7	4.3	3.8
CCR, wt %	39.53	38.02	36.35
Aromatic Carbon, wt %	93	90	91
<u>Trace Metals Analyses, ppm</u>			
Sodium	900	400	400
Potassium	200	300	<100
Calcium	<100	100	<100
Vanadium	3.3	2.9	2.8
Iron	119	132	122
<u>Distillation, °F (D-1160-1)</u>			
IBP	560	537	511
5%	619	594	610
10%	713	628	688
30%	1015	926	994
50%	-	-	-

Table 4-29

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 70, 80, AND 90% BLENDS MONTEREY SRC/SOLVENT
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2593 UNIT: CT-146

	J-8977	MB-864	MB-865	J-8978	MB-866	MB-867	MB-868	J-8979	MB-869
°/° SRC	70			80				90	
OPERATING CONDITIONS									
TEMPERATURE, °F	-	723	792	-	719	781	780	-	781
PRESSURE, PSIG	-	2000	2000	-	2000	2000	2000	-	2000
LHSV	-	0.21	0.32	-	0.28	0.22	0.41	-	0.38
DAYS ON STREAM	-	1.9	2.7	-	3.6	4.6	5.2	-	5.9
LIQUID PRODUCT PROPERTIES									
GRAVITY, °API	-15.8	-1.4	5.0	-15.9	-3.5	4.8	0.0	-16.3	0.4
HYDROGEN, WT °/°	6.40	8.26	8.83	6.23	7.79	9.06	7.93	6.10	8.42
SULFUR, WT °/°	0.840	0.187	0.084	0.810	0.166	0.032	0.105	0.860	0.089
NITROGEN, WT °/°	2.010	0.790	0.480	1.910	0.960	0.390	0.790	1.720	0.800
OXYGEN, WT °/°	3.7	1.7	0.8	4.3	1.8	0.4	1.2	3.8	1.0
CCR, WT °/°	39.53	21.20	14.16	38.02	20.59	11.96	19.30	36.35	18.82
K.V. AT 100°C	-	107.90	7.34	-	264.20	6.15	25.60	-	23.92
AROMATIC CARBON, °/°	93	54	51	90	59	50	58	91	53
HETEROATOM REMOVAL, PCT									
SULFUR	-	78.6	90.6	-	80.3	96.5	88.2	-	91.3
OXYGEN	-	55.8	79.6	-	59.7	91.8	74.6	-	78.0
NITROGEN	-	62.2	77.5	-	51.6	82.0	62.4	-	61.0
HYDROGEN UTILIZATION, SCF/B									
H2 CONSUMED BY C1-C5	-	250	409	-	137	1254	826	-	1462
H2 CONSUMED BY C6+	-	1241	1588	-	1026	1524	884	-	1266
H2 CONSUMED BY S	-	34	39	-	33	40	36	-	40
H2 CONSUMED BY N	-	218	271	-	172	273	207	-	183
H2 CONSUMED BY O	-	210	299	-	261	402	326	-	302
H2 CONSUMED TOTAL	-	1952	2606	-	1628	3493	2281	-	3255

	MB-864	MB-866	MB-868	MB-869
% SRC	70	80	80	90
Operating Conditions				
Pressure, psig	2000	2000	2000	2000
Temperature, °F	723	719	780	781
LHSV, V/Hr/V	0.21	0.28	0.41	0.38
Liquid Product Properties				
Gravity, °API	-1.4	-3.5	0.0	0.4
Hydrogen, wt %	8.58	7.79	7.93	8.42
Sulfur, wt %	0.187	0.166	0.105	0.089
Nitrogen, wt %	0.79	0.96	0.79	0.80
Oxygen, wt %	1.7	1.8	1.2	1.0
CCR, wt %	21.20	20.59	19.3	18.82
Heteroatom Removal, %				
Sulfur	79	80	88	91
Oxygen	56	60	75	78
Nitrogen	62	52	62	61
Total H_2 Consumption, scf/B	2224	1628	2281	3255

Taking into account slight differences in temperatures and space velocity, there is no dramatic difference between the various hydroprocessed blends at the severities used. No additional analyses were done on the products from this run.

90% Monterey SRC. After the premature shutdown of the run charging the 70-80-90% SRC blends, the catalyst was unloaded and another run charging 90% SRC was made over the Harshaw 618X. Problems with the charge pump check valves and capacitance probe high temperature separator were encountered. However, an 11 day run, including ten material balances was completed. At this point, an attempt was made to run 100% SRC through the unit. The charge pump and post-reactor product separation system could not handle this material, and the run was shut down.

The operating conditions, hydrogen utilization, and liquid product properties are given along with the charge stock properties in Table 4-30. Appendix Table B-9 includes the product yields and distillations of the liquid products. The hydrogen consumptions ranged from 1665 to 3125 scf/B with heteroatom removals

Table 4-30

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 90% BLEND MONTEREY SRC/SOLVENT (J-8991)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2628 UNIT: CT-146

	CHARGE	MB-871	MB-872	MB-873	MB-874	MB-875	MB-876	MB-877	MB-878	MB-879	MB-880
OPERATING CONDITIONS											
TEMPERATURE, °F	-	720	778	781	769	748	777	775	798	800	770
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.24	0.28	0.46	0.40	0.17	0.22	0.20	0.15	0.38	0.11
DAYS ON STREAM	-	2.2	3.7	4.6	5.1	6.0	7.0	8.0	9.0	9.7	10.5
LIQUID PRODUCT PROPERTIES											
GRAVITY, °API	-17.2	-3.5	0.6	-1.9	-6.2	-2.6	0.6	0.9	4.3	-4.1	1.0
HYDROGEN, WT °/°	6.11	7.78	8.60	8.28	7.75	8.36	8.73	8.72	8.86	8.08	8.71
SULFUR, WT °/°	1.00	0.11	0.05	0.20	0.23	0.05	0.05	0.05	0.02	0.13	0.04
NITROGEN, WT °/°	1.86	0.98	0.58	0.89	1.21	0.85	0.68	0.65	0.41	0.98	0.56
OXYGEN, WT °/°	4.3	1.9	0.5	1.3	1.5	1.1	0.7	0.7	0.3	1.3	0.5
CCR, WT °/°	44.08	22.19	16.35	21.32	26.67	20.38	16.45	10.12	12.57	25.53	13.82
K.V. AT 100°C	-	204.40	12.26	49.58	487.70	-	24.02	20.78	5.96	82.26	19.91
AROMATIC CARBON, °/°	77	54	56	63	62	56	54	56	55	61	53
HETEROATOM REMOVAL, PCT											
SULFUR	-	89.3	95.3	81.6	78.5	95.3	95.2	95.3	97.9	88.1	95.9
OXYGEN	-	57.2	89.1	72.2	67.3	75.9	84.4	84.8	93.6	72.1	89.4
NITROGEN	-	48.9	70.9	56.0	39.1	56.9	65.0	67.5	79.8	51.3	72.0
HYDROGEN UTILIZATION, SCF/B											
H2 CONSUMED BY C1-C5	-	38	977	633	529	381	120	593	777	803	507
H2 CONSUMED BY C6+	-	1168	1684	1384	939	1452	1839	1642	1622	1109	1636
H2 CONSUMED BY S	-	46	49	42	40	49	49	49	50	45	49
H2 CONSUMED BY N	-	160	232	183	128	186	213	221	261	168	236
H2 CONSUMED BY O	-	253	394	319	298	336	374	375	414	319	396
H2 CONSUMED TOTAL	-	1665	3337	2562	1934	2404	2594	2881	3125	2444	2824

as high as 98, 94, and 80 percent for the sulfur oxygen and nitrogen respectively. The products from MB-872 and 880 are very similar in spite of the fact that MB-880 was run at less than half the space velocity of MB-872. This is indicative of the catalyst aging experienced with these high residual content coal liquids.

The data in Table 4-30 were modeled using a second order rate equation. The kinetic constants and average deviation determined using our modified Marquardt fitting routine are given below. The activation energies are similar to those reported for the 33% blend (Table 4-21). However, the reaction rate constant at 750°F ($k_{750°F}$) is 20-50 percent of those reported for the 33% blend. The nitrogen $k_{750°F}$ is particularly low for this 90% blend, since most of the nitrogen present is derived from the SRC and not the recycle solvent portion of the blend. Heteratoms are, of course, more difficult to remove from the heavier fractions.

Table 4-31

CONSTANTS FOR KINETIC MODEL FOR HETEROATOM REMOVAL

Charge: 90 Wt % SRC Blend
Catalyst: Harshaw 618X
Pressure: 2000 psig

SECOND ORDER RATE EQUATION *

	k, HR^{-1} $(WT\ \%)^{-1}$	E, BTU/LB MOLE	$k_{750°F}$, HR^{-1} $(WT\ \%)^{-1}$	AVG. DEVIATION WT %
Nitrogen	1.36 E5	33,097	0.143	0.127
Oxygen	2.44 E9	56,808	0.162	0.208
Sulfur	1.00 E5	26,129	2.084	0.04

$$^{*}\frac{dC_i}{dt} = k\ e^{-E/RT} C_i^{\ 2}$$

The products were distilled to obtain a 650°F$^+$ and 650°F$^-$ fraction. The 650°F$^+$ fraction was then analyzed by our GEC technique. Table 4-32 gives the GEC analyses of the Monterey SRC, the 90% blend and four 650°F$^+$ hydroprocessed products. The 90% blend is almost identical in composition to the SRC, indicating some of the light material may have been lost in the preparation and charging of this blend. The charge contains very little 650°F material, and was too heavy to obtain a meaningful distillation. The products, however, contained up to 32-58 wt % 650°F$^-$ material. At this high severity, almost 90% of the polar asphaltenes have been converted to lighter materials. This reduction in asphaltenes is largely responsible for the big decrease in viscosity shown in Table 4-30.

High Concentration Burning Star and Wyodak SRC Studies

Although the bulk of the SRC hydroprocessing studies concentrated on Monterey SRC's several additional runs were done on high concentration blends of Illinois Burning Star and Wyodak (Amax) SRC's. Previous work was done on low concentration, 33 wt % SRC blends of these two SRC's. The present work involved only short process variable studies to determine if these other SRC's at higher concentrations presented any unusual problems in processing. No kinetic modeling or GEC analyses are presented for the data obtained. Two separate studies were made processing the Burning Star SRC at concentrations of 50% and 70-90% SRC, and one charging a 70% blend of Wyodak SRC.

50% Burning Star SRC. A 50 wt % blend of Illinois No. 6 Burning Star was hydroprocessed over Ketjen 153S, a small pore NiMo/Al_2O_3 catalyst. Catalyst properties are given in Table 4-18. Severe post reactor plugging caused a premature shutdown of this run. Although material balances were not completed, two different reactor products were obtained. These products contained both a liquid and a solid phase. The analyses of these phases (Table 4-33) show them to be substantially different in hydrogen and nitrogen content, although each has been upgraded relative to the charge blend. The weighted average properties of the two phases shows significant upgrading of the blend as a whole. This was our first run with a relatively high concentration SRC blend over a small pore catalyst. It was concluded that this catalyst upgraded the distillate fraction of the blend to the point where it was no longer compatible with the residual fraction and in-situ deasphalting occurred. This same phenomenon was observed when hydroprocessing a blend of the short contact time SRC (Section 4.4).

Table 4-32

GEC ANALYSES OF HYDROPROCESSED 90% MONTEREY SRC BLEND

	76D2155 MONTEREY SRC**	J8991 90% SRC CHARGE STOCK**	MB-871	MB-872	MB-877	MB-878
Temperature, °F	-	-	720	778	777	798
LHSV	-	-	0.24	0.28	0.20	0.15
Pressure, psig	-	-	2000	2000	2000	2000
H_2 Consumed, scf/B	-	-				
Days On Stream	-	-	2.2	3.7	8.0	10.5
<u>Total Liquid Product</u>						
Gravity, °API	-15.3	-17.2	-3.5	0.4	0.6	4.3
Hydrogen, wt %	6.22	6.11	7.78	8.60	8.73	8.86
Oxygen, "	4.0	4.3	1.9	0.5	0.7	0.3
Nitrogen, "	1.75	1.86	0.98	0.58	0.65	0.41
Sulfur, "	0.7	1.00	0.11	<0.05	<0.05	0.023
650°F^-, wt %	-	-	16.76	30.21	25.83	32.58
<u>650°F^+ Residue</u>						
Cut 1 (saturates)	0.13	0.15	1.15	1.34	2.31	1.23
Cut 2-4 (arom. oils)	11.88	12.47	32.10	39.77	42.71	42.22
Cut 5-7 (resins/asph.)	35.92	33.51	33.78	21.79	22.00	18.42
Cut 8-13 (polar asph.)*	52.07	53.87	16.21	6.89	7.15	5.55
Total	100.00	100.00	100.00	100.00	100.00	100.00

---

*Cut 13, wt % (Non-eluted + lost)	7.21	7.36	0.82	1.24	1.82	1.52

**No distillation done on charge stocks.

Table 4-33

ANALYSES OF TWO PHASE PRODUCTS FROM HYDROPROCESSED 50% BURNING STAR SRC BLEND

SEVERITY		LOW(1)			HIGH(2)		
	CHARGE	LIQUID	SOLID	TOTAL (CALC.)	LIQUID	SOLID	TOTAL (CALC.)
Wt % Product		87	13	(100)	75	25	(100)
Analysis, Wt %							
Hydrogen	6.34	9.54	8.06	(9.34)	10.61	9.04	(10.22)
Nitrogen	1.25	0.53	1.02	(0.59)	0.25	0.96	(0.43)

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(1) 2000 psig H_2 pressure, 725°F, 1.0 LHSV.

(2) 2000 psig H_2 pressure, 725°F, 0.25 LHSV.

Table 4-34

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 50% BLEND BURNING STAR SRC/SOLVENT (76D-2351)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2455 UNIT: CT-146

	CHARGE	MB-806	MB-807	MB-808	MB-809	MB-810	MB-811
OPERATING CONDITIONS							
TEMPERATURE, °F	-	672	729	780	673	725	777
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
LHSV	-	0.84	0.84	0.77	0.22	0.19	0.27
DAYS ON STREAM	-	1.0	1.5	1.8	3.0	4.6	6.0
LIQUID PRODUCT PROPERTIES							
GRAVITY, °API	-5.3	1.2	5.2	9.2	5.9	11.8	13.6
HYDROGEN, WT °/°	6.36	7.70	8.63	9.26	8.95	9.90	9.79
SULFUR, WT °/°	0.47	0.18	0.13	0.06	0.12	0.06	0.04
NITROGEN, WT °/°	1.25	0.90	0.74	0.25	0.50	0.32	0.28
OXYGEN, WT °/°	4.3	2.3	1.4	0.7	1.2	0.4	0.3
CCR, WT °/°	24.96	17.32	12.82	10.04	11.83	8.18	6.00
AROMATIC CARBON, °/°	94	67	54	47	-	-	46
HETEROATOM REMOVAL, PCT							
SULFUR	-	61.8	73.8	86.7	74.9	87.9	91.9
OXYGEN	-	47.5	68.2	84.4	72.8	91.0	93.4
NITROGEN	-	29.3	42.2	80.8	61.0	75.3	78.8
HYDROGEN UTILIZATION, SCF/B							
H2 CONSUMED BY C1-C5	-	44	89	260	90	228	510
H2 CONSUMED BY C6+	-	889	1545	1879	1763	2378	2147
H2 CONSUMED BY S	-	14	16	19	16	19	20
H2 CONSUMED BY N	-	58	84	161	122	150	157
H2 CONSUMED BY O	-	190	273	338	292	365	374
H2 CONSUMED TOTAL	-	1195	2007	2658	2283	3140	3209

This in-situ deasphalting problem was overcome by going to a large pore catalyst, Harshaw 618X (properties in Table 4-18). This same 50% blend of Burning Star SRC was run for six days over this catalyst with no problems. The operating conditions, liquid product properties, and hydrogen utilization for the six material balances in this run are given in Table 4-34. Appendix Table B-10 gives the yields and product distillation data. The data in Table 4-35 show hydrogen consumptions from 1195 to 3209 sct/B for this blend, with greater than 90% removal of the sulfur and oxygen and up to 88% removal of the nitrogen.

<u>70 and 90% Burning Star SRC</u>. A study was made hydroprocessing a 70% and 90% concentration blends of Burning Star SRC. Table 4-35 shows the elemental composition and CCR content of the Burning Star SRC, the recycle solvent, and three different concentration blends. The blends were sampled at the charge pump just prior to entering the unit.

Table 4-35

COMPOSITION OF BURNING STAR SRC AND CONCENTRATED FEED BLENDS

					(RECYCLE SOLVENT)	
SRC CONCENTRATION	100	90	70	50	0	
Gravity, °API	-12.2	-18.7	-16.4	-5.3	-3.4*	3.7
Hydrogen, Wt %	5.72	5.81	6.25	6.36	7.56	7.42
Sulfur, Wt %	0.57	0.56	0.53	0.47	0.32	0.32
Nitrogen, Wt %	1.71	1.95	1.66	1.25	0.59	0.61
Oxygen, Wt %	3.50	3.27	3.40	4.30	4.05	3.30
CCR, Wt %	48.0	51.2	45.0	25.0	0.1	1.1

---

[*] Used only in 50% blend.

From the properties of the blends and blend components, it appears that light materials were lost in our charge system. For the 70 and 90% blends this system was operated at up to 400°F with a continuous flow of nitrogen over the liquid. For the 50% blend, the charge system was operated below 250°F. It appears that at the high temperatures needed to mix and charge the high concentration blends, a considerable amount of light material was lost from the solvent, and perhaps from the SRC itself. In most respects, these blends are equivalent to undiluted Burning Star SRC. Modifications to our charge system should correct these problems in the future.

Operating conditions, product properties, and hydrogen utilization data for the 70 and 90% blends are given in Table 4-36. Yields and product distillation data are given in Appendix Table B-11. We had difficulty in maintaining low pump rates with these feeds, and as a result, the processing is relatively mild. Hydrogen consumptions in the range of 1700 scf/B were achieved. The table below compares the upgrading obtained with that obtained from the Monterey 70% SRC run. At almost equivalent conditions, the percent removal of heteroatoms and CCR is similar. The Burning Star blend contains more CCR and is more dense. This is reflected in the products from this run.

	70% Burning Star SRC		70% Monterey SRC	
	Charge	MB-882	Charge	MB-858
Operating Conditions				
Pressure, psig	-	2000	-	2000
Temperature, °F	-	773	-	781
LHSV, V/hr/V	-	0.43	-	0.41
Days On-Stream	-	3.1	-	13.6
Liquid Product Properties				
Gravity, °API	-16.4	-3.3	-13.4	5.2
Hydrogen, wt %	6.25	7.79	6.15	8.67
Sulfur, wt %	0.53	0.09	0.92	0.12
Nitrogen, wt %	1.66	0.94	1.65	0.79
Oxygen, wt %	3.4	0.9	5.1	1.6
CCR, wt %	45.0	23.0	38.2	16.9
K.V., cs @ 100°C	-	87.0	-	61.3
Heteroatom Removal, wt %				
Sulfur	-	83	-	88
Oxygen	-	75	-	71
Nitrogen	-	46	-	55
CCR	-	51	-	59
Total H_2 Consumption, scf/B	-	1750	-	2600

Table 4-36

FIXED BED HYDROPROCESSING OF COAL LIQUIDS

CHARGE: 70 AND 90% BLENDS BURNING STAR SRC/SOLVENT (J-9031/J9032)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2644 UNIT: CT-146

	CHARGE	MB-881	MB-882	CHARGE	MB-883	MB-884
OPERATING CONDITIONS						
TEMPERATURE, °F	-	724	773	-	722	773
PRESSURE, PSIG	-	2000	2000	-	2000	2000
LHSV	-	0.84	0.43	-	0.55	1.04
DAYS ON STREAM	-	2.6	3.1	-	7.2	8.2
LIQUID PRODUCT PROPERTIES						
GRAVITY, °API	-16.4	-8.8	-3.3	-18.7	-10.9	-6.7
HYDROGEN, WT °/°	6.25	7.08	7.79	5.81	7.01	7.14
SULFUR, WT °/°	0.53	0.26	0.09	0.56	0.28	0.30
NITROGEN, WT °/°	1.66	1.32	0.94	1.95	1.49	1.59
OXYGEN, WT °/°	3.4	1.8	0.9	3.3	3.0	1.8
CCR, WT °/°	44.98	29.95	23.02	51.19	33.87	37.58
K.V. AT 100°C	-	>99M	86.96	-	>99M	>99M
AROMATIC CARBON, °/°	-	59	58	-	-	-
HETEROATOM REMOVAL,PCT						
SULFUR	-	53.2	83.1	-	50.2	48.9
OXYGEN	-	48.7	74.8	-	10.2	48.6
NITROGEN	-	22.9	46.0	-	24.2	21.7
HYDROGEN UTILIZATION,SCF/B						
H2 CONSUMED BY C1-C5	-	123	332	-	123	473
H2 CONSUMED BY C6+	-	576	1000	-	976	921
H2 CONSUMED BY S	-	14	22	-	15	14
H2 CONSUMED BY N	-	66	133	-	84	75
H2 CONSUMED BY O	-	169	260	-	35	166
H2 CONSUMED TOTAL	-	950	1748	-	1232	1650

70% Wyodak SRC. A six day run with six material balances was made with a 70% blend of Wyodak SRC in recycle solvent over Harshaw 618X catalyst. The operating conditions and product properties are given in Table 4-37. Yields and product distillations are in Appendix Table B-12. Hydrogen consumptions as high as 3200 scf/B were obtained. The sulfur content of the products was very low (0.01 to 0.03 wt %), reflecting the low sulfur of this SRC blend (0.16 wt %). The removal of heteroatoms and reduction of CCR were similar to those obtained with the Burning Star and Monterey blends.

Effect of SRC Concentration in Hydroprocessing of SRC Blends. In this section we compare the results obtained in the runs charging varying concentrations of Monterey SRC. Figure 4-15 shows that about 65% of the total hydrogen consumed winds up in the liquid product, 20% winds up in the heteroatom containing gaseous products, and 15% winds up in the hydrocarbon (C_1-C_5) gases. The 90% blend appears to put somewhat more hydrogen into the gaseous products and less into the C_6^+ liquid. However, little difference is seen between the different blends.

Table 4-38 shows a comparison of balances taken at similar operating conditions from each of three SRC concentration runs. These data show higher hydrogen consumption for the higher concentration blends. However, as should be expected, the products from the higher concentration blends are lower quality with respect to heteroatom and CCR content. The percent heteroatom removal and CCR reduction is also lower for the higher concentration blends. The relative ease of heteroatom removal is sulfur >oxygen>nitrogen. Figure 4-16 shows the sulfur content as a function of hydrogen content for these blends. When the hydrogen content of the blend reaches 8 wt %, the sulfur is less than 0.2 wt % for all the blends. Figure 4-17 shows the aromatic carbon content as a function of the hydrogen content. At 10% hydrogen in the liquid product, the fraction of the carbon atoms which are aromatic, is reduced to less than 40% for all blends.

Using the kinetic constants determined from the data for the second order heteroatom removal rate expression, it is possible to calculate the percent heteroatom removal for recycle solvent and the Monterey SRC blends at the same operating conditions. (The kinetic constant for the recycle solvent over American Cyanamid HDS-9A were determined last year.) The table shown on page 4-75 shows the predicted heteroatom removal at 750°F, 1.0 LHSV and 2000 psig. These data show that the percent removal of all heteroatoms decreases with increasing SRC concentration. This is particularly true for the nitrogen which appears to be quite difficult to remove from SRC.

Table 4-37

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 70% BLEND WYODAK SRC/SOLVENT (J-9075)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2671 UNIT: 146

	CHARGE	MB-885	MB-886	MB-887	MB-888	MB-889	MB-890
OPERATING CONDITIONS							
TEMPERATURE, °F	-	739	739	775	727	799	723
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
LHSV	-	0.70	0.58	0.25	0.38	0.32	0.40
DAYS ON STREAM	-	1.2	1.7	2.5	4.0	5.0	5.9
LIQUID PRODUCT PROPERTIES							
GRAVITY, °API	-15.5	-5.6	-5.6	-2.1	-4.4	2.3	-4.4
HYDROGEN, WT °/°	6.30	7.60	7.78	8.05	8.18	8.58	7.65
SULFUR, WT °/°	0.16	0.04	0.04	0.01	0.03	0.01	0.03
NITROGEN, WT °/°	1.52	1.06	1.03	0.69	0.91	0.52	1.04
OXYGEN, WT °/°	3.8	1.6	1.4	0.5	1.2	0.5	1.4
CCR, WT °/°	41.26	27.77	26.38	22.94	27.06	18.79	27.09
K.V. AT 100°C	-	180.70	16.70	28.42	214.10	6.48	230.40
AROMATIC CARBON, °/°	-	-	-	60	-	58	-
HETEROATOM REMOVAL, PCT							
SULFUR	-	75.2	76.8	94.2	78.7	95.9	80.0
OXYGEN	-	60.1	64.1	88.9	69.3	87.7	64.3
NITROGEN	-	34.0	33.9	61.7	41.7	68.1	33.7
HYDROGEN UTILIZATION, SCF/B							
H2 CONSUMED BY C1-C5	-	442	113	2115	140	673	155
H2 CONSUMED BY C6+	-	855	1050	574	1363	1438	935
H2 CONSUMED BY S	-	6	6	8	6	8	6
H2 CONSUMED BY N	-	90	90	163	110	179	89
H2 CONSUMED BY O	-	232	247	343	267	338	248
H2 CONSUMED TOTAL	-	1624	1506	3202	1885	2637	1433

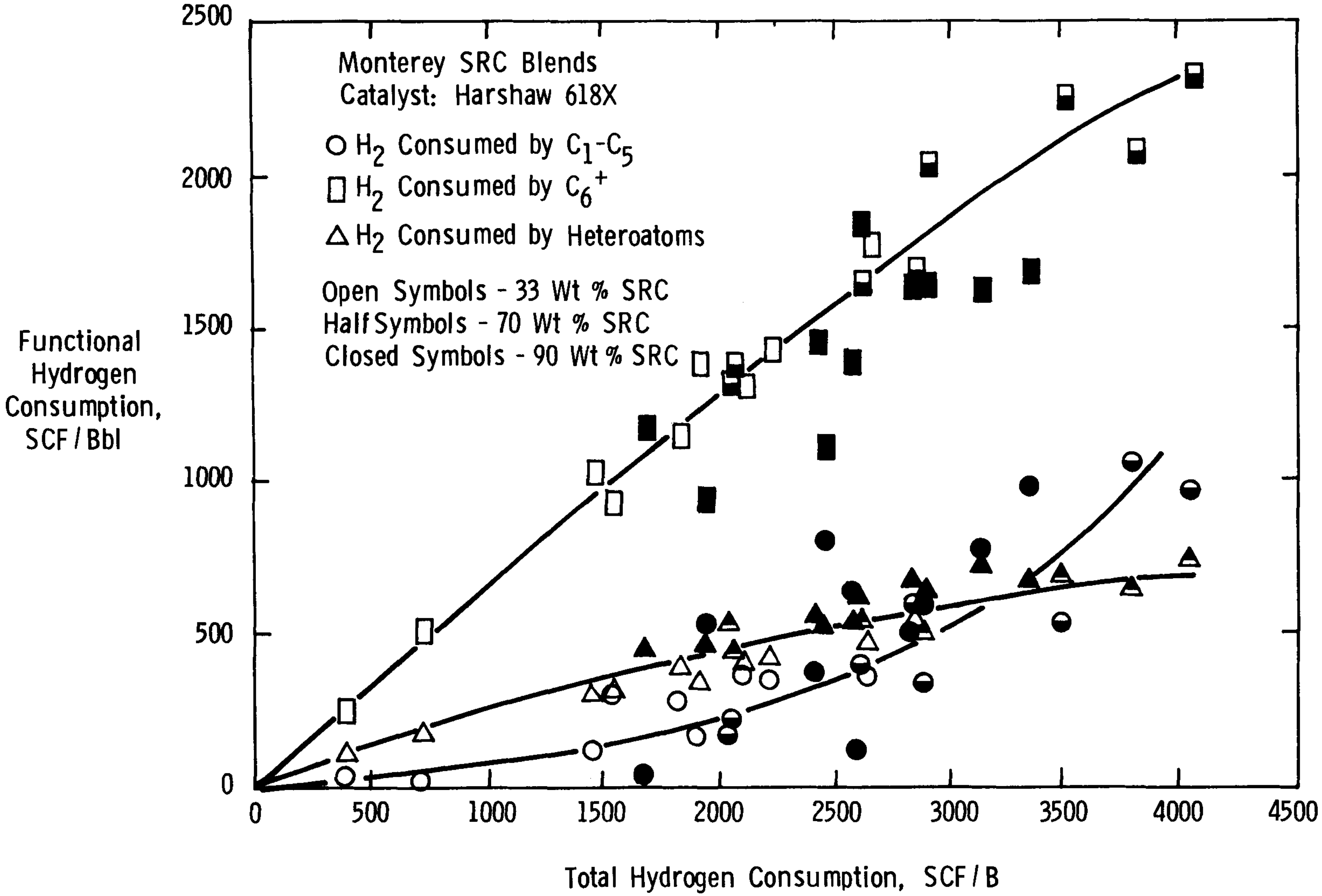

Figure 4-15. Hydrogen Utilization in Hydroprocessing of Monterey SRC

Table 4-38

EFFECT OF SRC CONCENTRATION ON HYDROPROCESSING MONTEREY SRC BLENDS

% SRC	33	70	90
MB-	828	858	873
Temperature, °F	777	781	781
LHSV	0.53	0.41	0.46
Pressure, psig	2000	2000	2000
Days On Stream	2.6	13.6	4.6
H_2 Consumption, scf/B	2084	2600	2562
Elemental Analyses			
Gravity, °API	12.2	5.2	-1.9
Hydrogen, wt %	9.17	8.67	8.28
Sulfur, wt %	0.04	0.12	0.20
Nitrogen, wt %	0.32	0.79	0.89
Oxygen, wt %	1.0	1.56	1.30
CCR, wt %	6.15	16.32	21.32
% C_A	-	48	63
Heteroatom Removal, wt %			
Sulfur	93.4	87.6	81.7
Oxygen	74.2	71.1	72.3
Nitrogen	72.7	54.8	56.1
CCR Removal, %	64	56	52

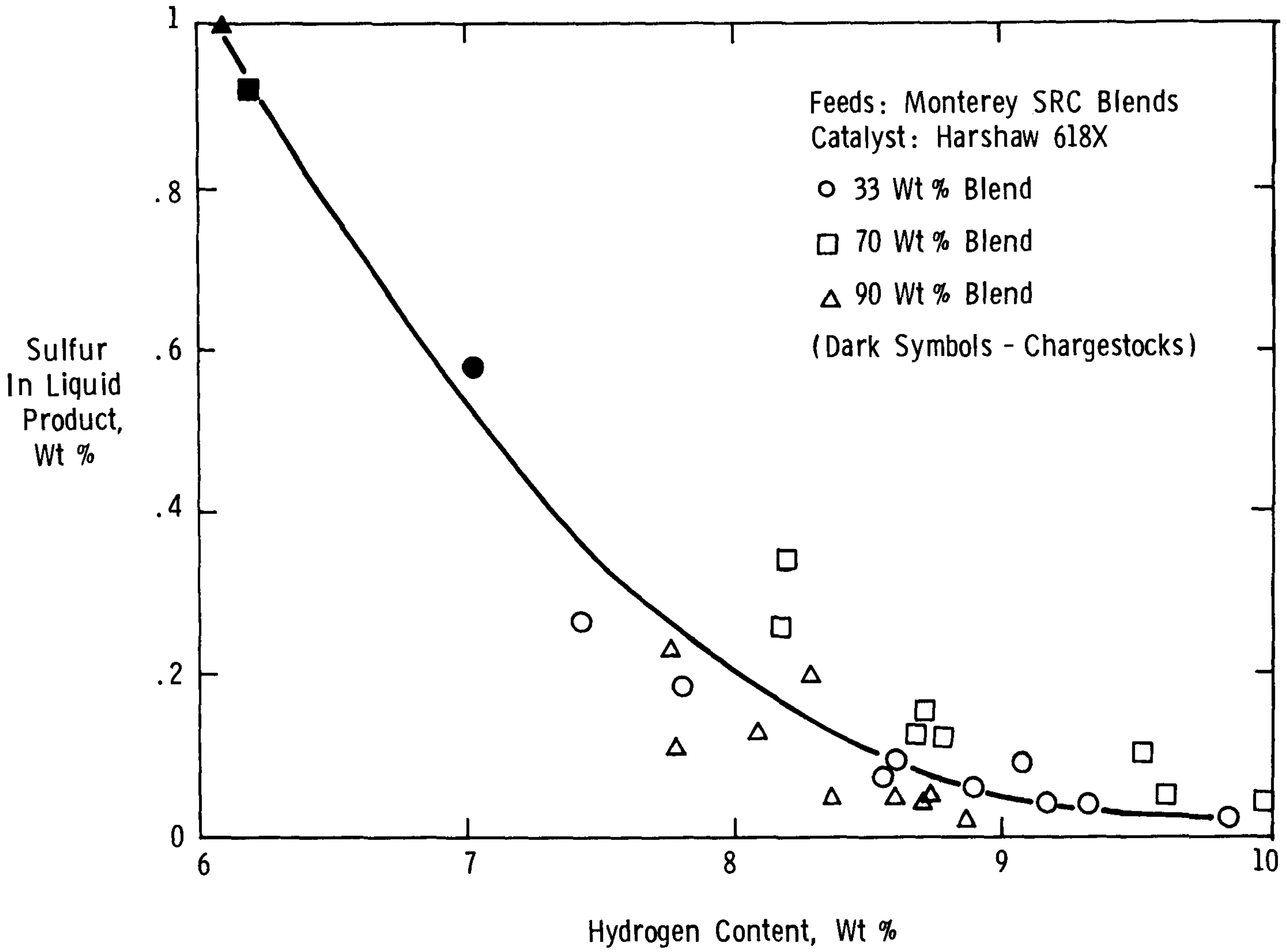

Figure 4-16. Sulfur Content of Hydroprocessed Monterey SRC Blends as a Function of Hydrogen Content

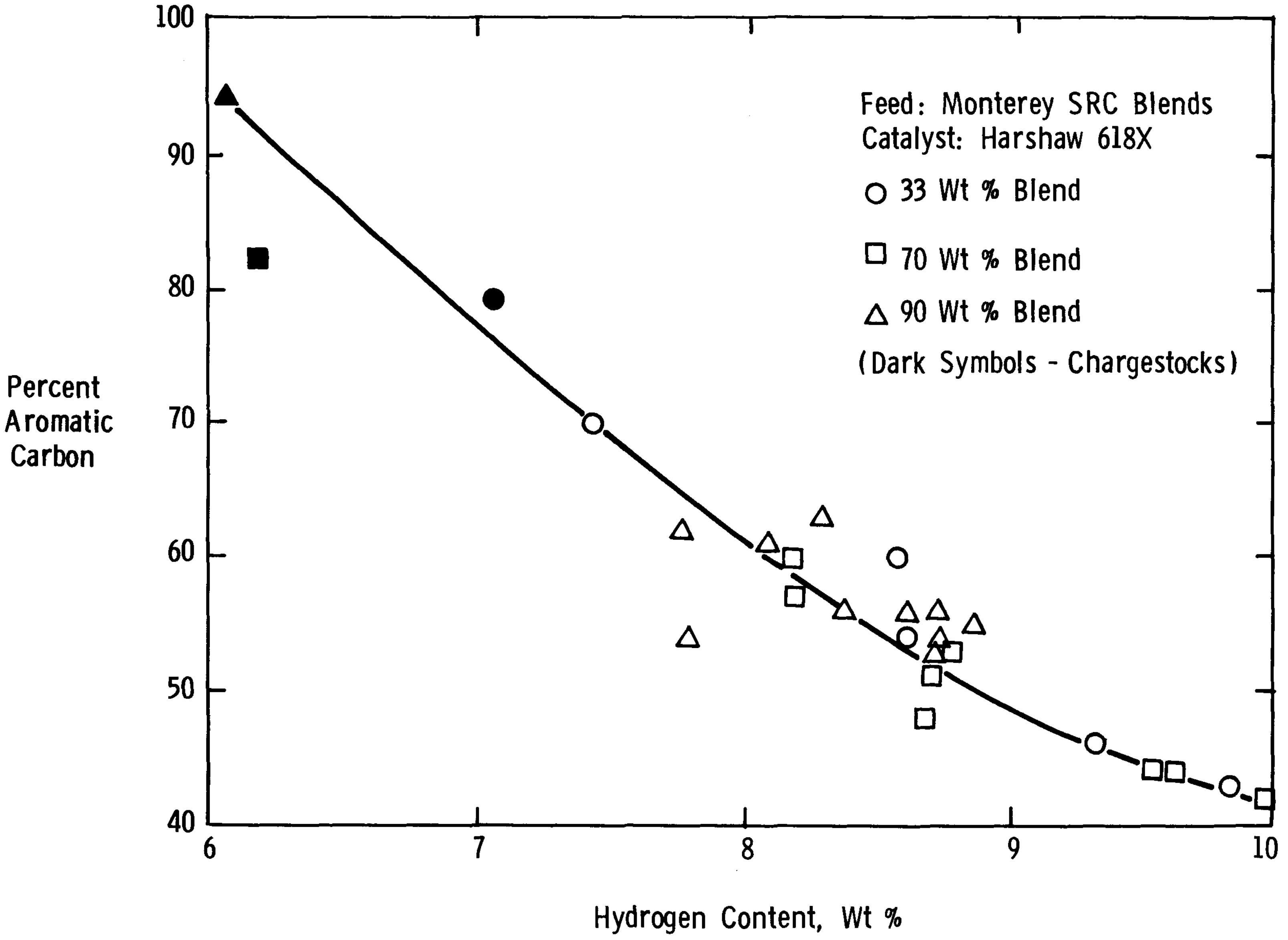

Figure 4-17. Percent Aromatic Carbon as a Function of Hydrogen Content for Hydroprocessed Monterey SRC Blends

Kinetic Model Predictions for Heteroatom Removal from Recycle Solvent and Monterey SRC Blends

750°F, 2000 psia, 1.0 LHSV

Feed	Recycle Solvent (1)	33% SRC Blend (2)	70% SRC Blend (2)	90% SRC Blend (2)
% Heteroatom Removal				
Nitrogen	89	45	31	21
Oxygen	98	52	46	36
Sulfur	97	81	64	66

---

(1) Cyanamid HDS-9A catalyst

(2) Harshaw 618X catalyst

The CCR in the blend can be attributed to the SRC component. It has been shown that CCR correlates very well with resins plus asphaltenes in the blend.

Figure 4-18 shows a plot of CCR conversion as a function of percent SRC in the blends at an equivalent total space velocity of 0.25 (0.45 WHSV) at temperatures of 725 and 775°F. Figure 4-19 shows a similar plot, but for data taken at equivalent SRC space velocities at 775°F. These data are taken from the two studies charging blends containing 33% Monterey SRC in two different recycle solvents and from the 70% and 90% Monterey SRC aging studies. No corrections have been made for slight differences in temperature or space velocity or for aging. The limited GEC data on the conversion of resins and asphaltenes for the SRC products support these trends.

Figure 4-18 shows that as the concentration of SRC is increased at a given total space velocity, the CCR (i.e., SRC) conversion remains approximately constant up to SRC concentrations of about 60%. Above 60 wt % SRC, there is a gradual decline in CCR conversion with increasing SRC concentration. These lines would be expected to be flat for a first order reaction. The lower conversion at the higher concentrations indicates that the SRC may be inhibiting its own conversion. Limited GEC analyses support this conclusion.

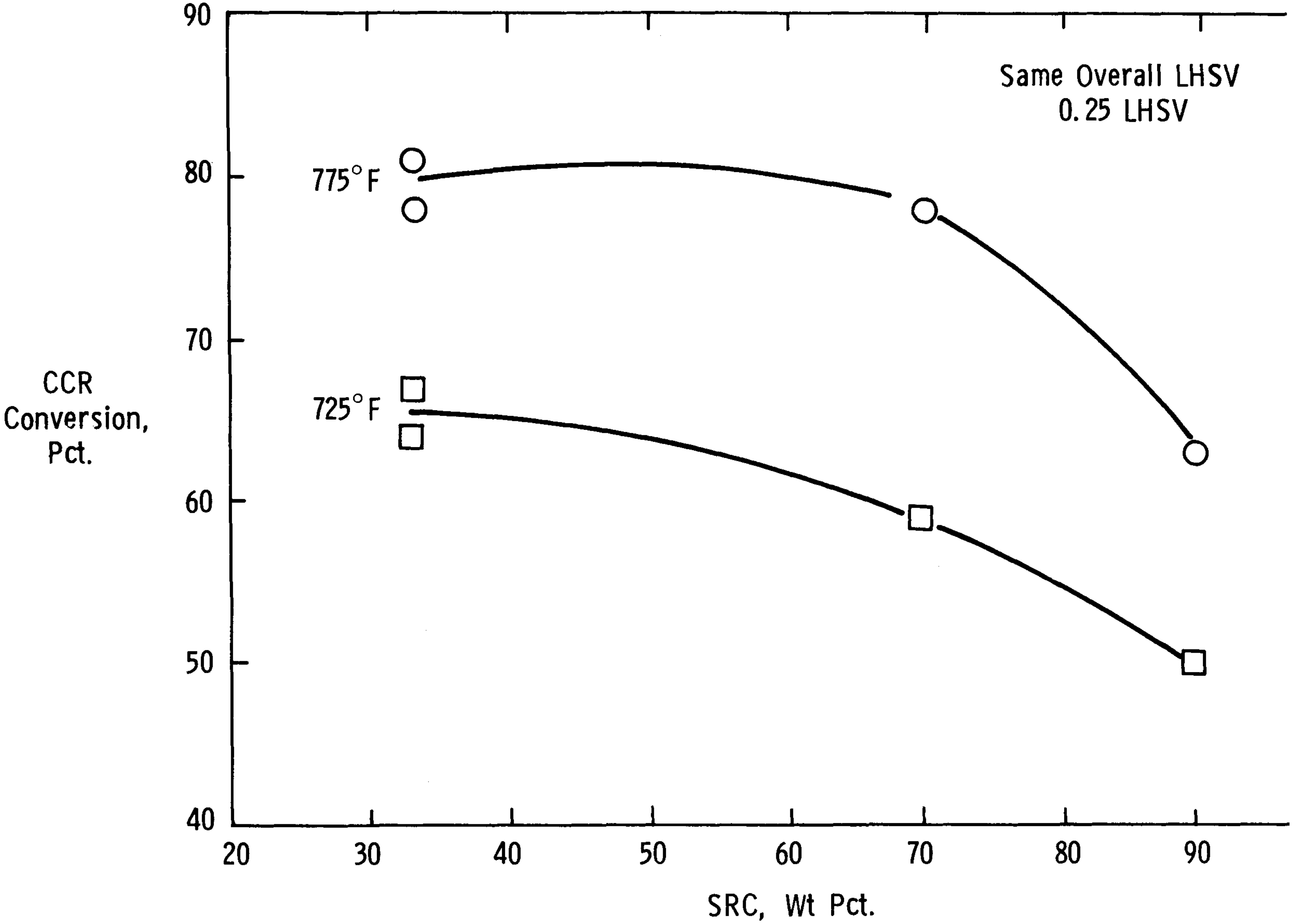

Figure 4-18. CCR Conversion for Monterey SRC Blends at Equivalent Overall LHSVs

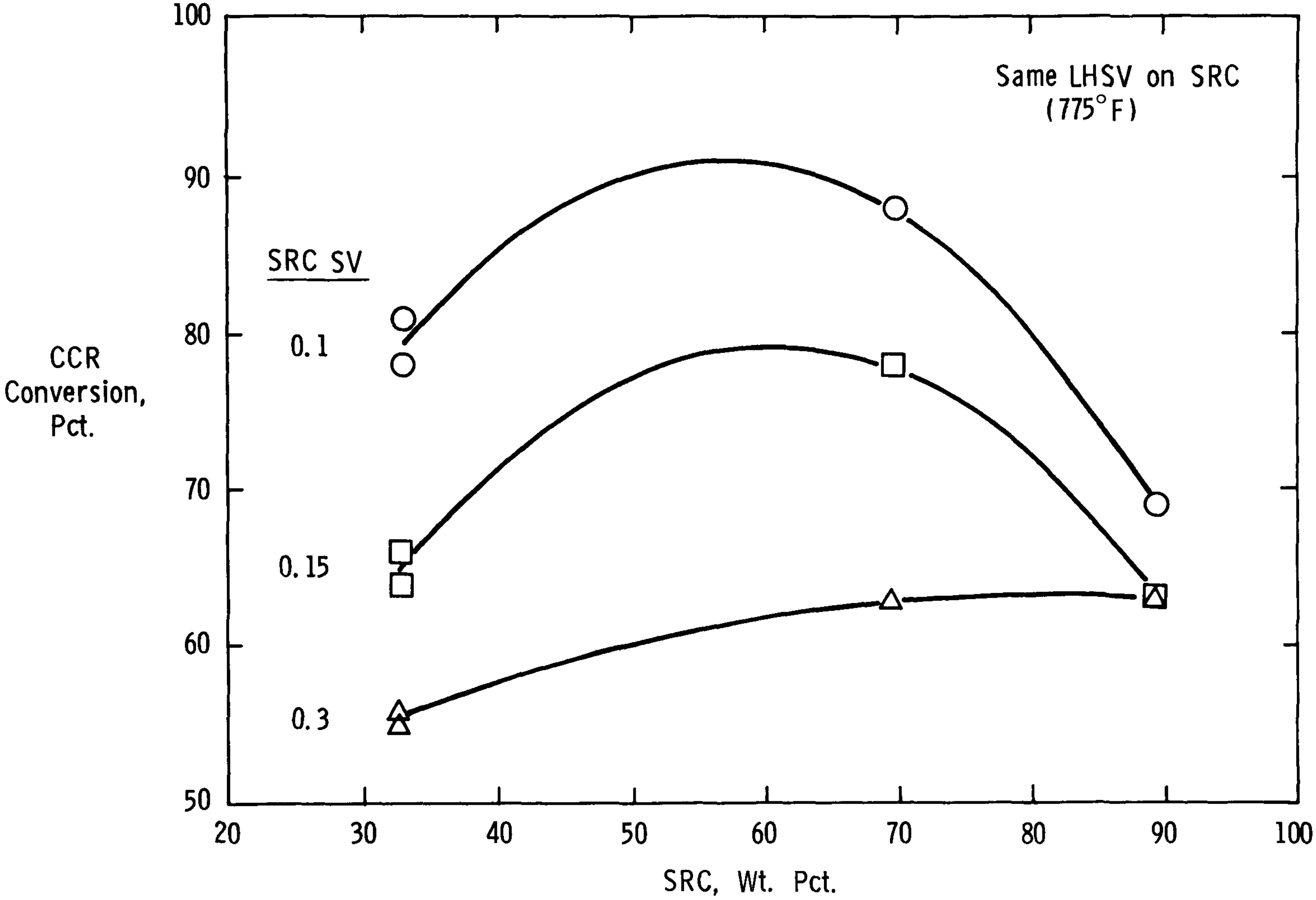

Figure 4-19. CCR Conversion for Monterey SRC Blends at Equivalent LHSVs on SRC

Figure 4-19 shows that if the CCR conversion is plotted versus the SRC concentration, a maximum in CCR conversion is observed at about 60 wt % SRC concentration. This means that for a given unit and feed rate of SRC, more SRC conversion can be achieved by diluting the feed to 60% SRC concentrations in recycle solvent than by running a 100% SRC feed.

4.4 SHORT CONTACT TIME SOLVENT REFINED COAL

Coal can be solubilized at very short residence times in the SRC process. Table 4-39 shows some data obtained at Wilsonville and at Mobil under EPRI Contract RP410. The short contact time (3-4 minutes) runs show considerably less hydrogen consumption and gas make than the regular SRC (40 minute residence time). The coal conversion is still very high at short residence times. The short contact time products, however, contain too much sulfur and further upgrading is required to yield an acceptable fuel.

Two drums of short contact time SRC produced from Monterey Run 98 (77D-13) and West Kentucky Run 88 (76D-3653) were received from Wilsonville via EPRI. Both SRC's were ground and riffled to obtain uniform consistency. Elemental and detailed gradient elution chromatographic analyses of the two SCT SRC's are shown in Table 4-40. The regular Monterey SRC analyses are also shown for comparison. The elemental analyses show that the short contact time Monterey SRC is slightly lower in hydrogen and higher in heteroatoms content than the regular Monterey SRC from Wilsonville. The sulfur content of the SCT sample is three times that of the regular SRC. The Kentucky SCT SRC contains less sulfur, but more nitrogen than the Monterey SCT SRC. Figure 4-20 shows a graphical comparison of the GEC analyses. The two short contact time SRC's are similar in composition; however, they are significantly different from the regular Monterey SRC. The short contact time SRC's generally contain less of Cuts 2-10 and more of Cuts 11-13 than regular SRC. For example, the non-eluted asphaltenes (Cut 13) account for 29.4-31.9% of the SCT SRC's and only 5.16 wt % of the regular SRC. Similarly, the eluted asphaltenes (Cut 7) are from 10.7 to 12.2 wt % of the SCT SRC's and 25.71 wt % of the regular SRC.

No further analyses or hydroprocessing studies were performed on the West Kentucky SCT SRC during the 1977 contract year. This material is to be hydroprocessed in high concentrations during the next contract period.

Table 4-39

COMPARISON OF REGULAR AND SHORT CONTACT TIME (SCT) SRC FROM WILSONVILLE

Monterey Coal

Run	Wilsonville 52	Wilsonville 98	Mobil
Date	Oct. 12-13, 1975	Dec. 3-4, 1976	CRD 59
Dissolver Bypass	No	Yes	Yes
Process Conditions			
Temperature, °F			
Preheater Outlet	805	823	800
Dissolver Outlet	855	–	–
Pressure, psig	2400	2430	1375
Residence Time, Min.	40	3-4	4
Results			
Conversion, % MAF Coal	95	89	88
H_2 Consumption, % MAF Coal	2.9	1.8	0.55
SRC Yields, % MAF Coal	51	76	83
Gas Make, % MAF Coal	11.9	4	2

Table 4-40

ANALYSES OF SHORT CONTACT TIME AND REGULAR SRC
FROM MONTEREY COAL

		SCT WEST KENTUCKY SRC 76D3653	SCT MONTEREY SRC 77D13	REGULAR SRC MONTEREY 76D2155
Composition, wt %				
Hydrogen		6.03	6.14	6.22
Sulfur		0.99	2.19	0.70
Oxygen		5.3	5.0	4.0
Nitrogen		1.99	1.66	1.75
Ash		0.26	0.55	0.085
CCR		44.98	48.40	-
GEC Analysis, wt %				
No.	Fraction Designation			
1	Saturates	0.27	0.04	0.02
2	MNA + DNA Oil	0.21	0.19	1.08
3	PNA Oil	0.21	1.07	1.79
4	PNA Soft Resin	7.54	3.09	4.83
5	Hard Resin	1.53	0.81	2.41
6	Polar Resin	3.53	2.12	4.86
7	Eluted Asphaltenes	12.18	10.67	25.71
8	Polar Asphaltenes	9.94	8.62	15.93
9	" "	2.84	4.62	5.52
10	" "	4.95	6.59	10.97
11	" "	17.58	21.56	17.73
12	" "	9.83	9.11	3.99
13	Non-eluted + Loss	29.39	31.87	5.16
	Total	100.00	100.00	100.00
Trace Elements, ppm				
Sodium		12	110	-
Potassium		10	31	100
Calcium		84	390	<100
Vanadium		15.7	11.4	3.9
Iron		375	470	140

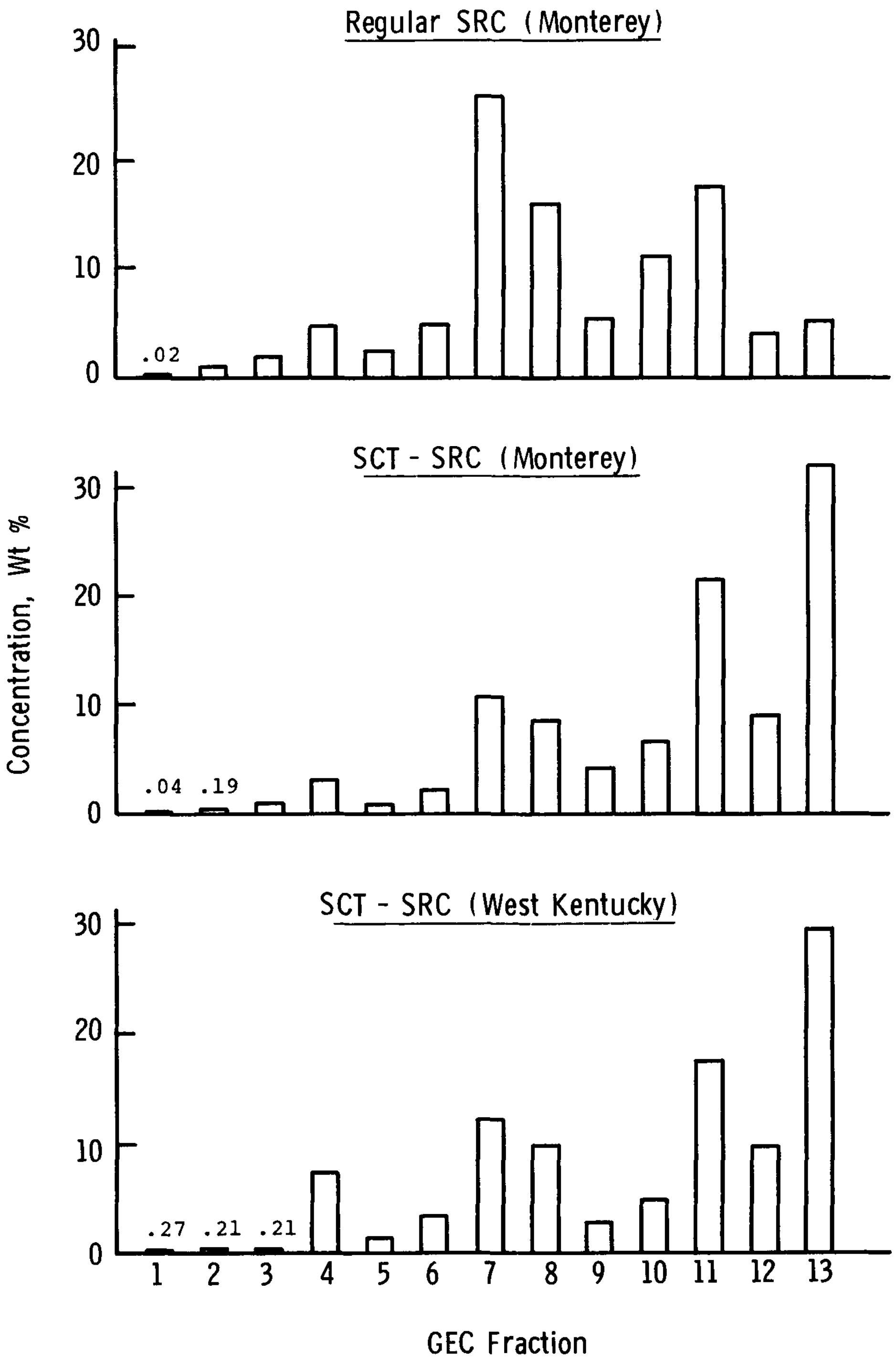

Figure 4-20. GEC Analyses of Regular and Short Contact Time (SCT) Wilsonville SRCs

The SCT Monterey SRC was further characterized by determining the elemental compositions and molecular weights for the GEC fractions. These data are shown in Table 4-41. In comparison with regular Monterey SRC, the higher heteroatom content of short contact time SRC is reflected in a similar increase in heteroatom content of the cuts. However, the heteroatom concentration profiles (Figure 4-21) remain similar for both SRC's. This is also true of the molecular weight profiles (Figure 4-22). The main difference is that cuts 9-12 of short contact time SRC have significantly higher molecular weights. From these data longer contact times appear to: (1) reduce the heteroatom concentration, especially sulfur; (2) lower the molecular weight of the SRC, especially for polar asphaltenes; (3) increase the amount of elutable material from nearly 70% to about 95%.

Figure 4-23 shows that there appears to be a relationship between the molecular weight and oxygen content of resins and asphaltenes from coal liquids. Figure 4-23 includes data from SCT and regular SRC, along with H-Coal fuel oil. Oxygen removal appears to be important in converting the resins and asphaltenes to lower molecular weight, hexane soluble material.

A single 33 wt % SRC blend was used for all the hydroprocessing studies performed on short contact time SRC during this contract year. A half drum quantity of the blend was prepared using two parts of the 400°F recycle solvent and one part of the short contact time Monterey SRC. Considerable difficulty was experienced in solubilizing this SRC in the recycle solvent as compared to regular SRC. Continuous stirring in a hot box at 240°F for several days was not sufficient to dissolve all the SRC. Heating to 300°F was required before a homogeneous, single phase charge stock was produced. This blend was considerably more viscous than typical 33% blends made with regular SRC.

The initial attempt to hydroprocess the SCT SRC blend over the small pore, CoMo/Al_2O_3 HDS-1441A catalyst, previously used for 33 wt % regular SRC blends, was unsuccessful. The unit was shut down after 48 hours on-stream due to plugging downstream of the reactor. Solid precipitation caused severe plugging in the liquid level controller and high pressure separator lines after the reactor. Although plugging of the reactor itself did not cause the shutdown, examination of the catalyst bed after dismantling showed signs of imminent plugging. A similar phenomenon occurred previously when hydroprocessing a 50% Burning Star SRC over the small pore NiMo catalyst Ketjen 153S. Use of the large pore

Table 4-41

ELEMENTAL COMPOSITION AND MOLECULAR WEIGHTS OF SHORT CONTACT TIME MONTEREY SRC

		H (Wt %)	S (Wt %)	N (Wt %)	O (Wt %)	M.W. (No. Avg.)
	77D13	6.14	2.19	1.66	5.0	(c)
	GEC Fractions					
1	Saturates	(a)	(a)	(a)	(a)	(a)
2	MNA + DNA Oil	11.73	(a)	(a)	0.7	(a)
3	PNA Oil	7.89	1.26	0.05	0.7	(a)
4	PNA Soft Resin	6.68	1.67	0.48	1.5	310
5	Hard Resin	6.60	1.89	1.38	2.1	(a)
6	Polar Resin	6.85	1.82	2.20	2.1	511
7	Eluted Asphaltenes	6.97	2.04	2.17	5.5	639
8	Polar Asphaltenes	6.71	2.01	1.64	7.1	810
9	Polar Asphaltenes	6.10	2.00	1.76	6.6	1305
10	Polar Asphaltenes	6.06	2.12	1.64	7.3	(b)
11	Polar Asphaltenes	6.05	1.87	1.41	8.3	1300
12	Polar Asphaltenes	5.95	2.02	1.53	9.6	1316

(a) Insufficient sample.

(b) Incomplete THF solubility.

(c) Analysis not determined.

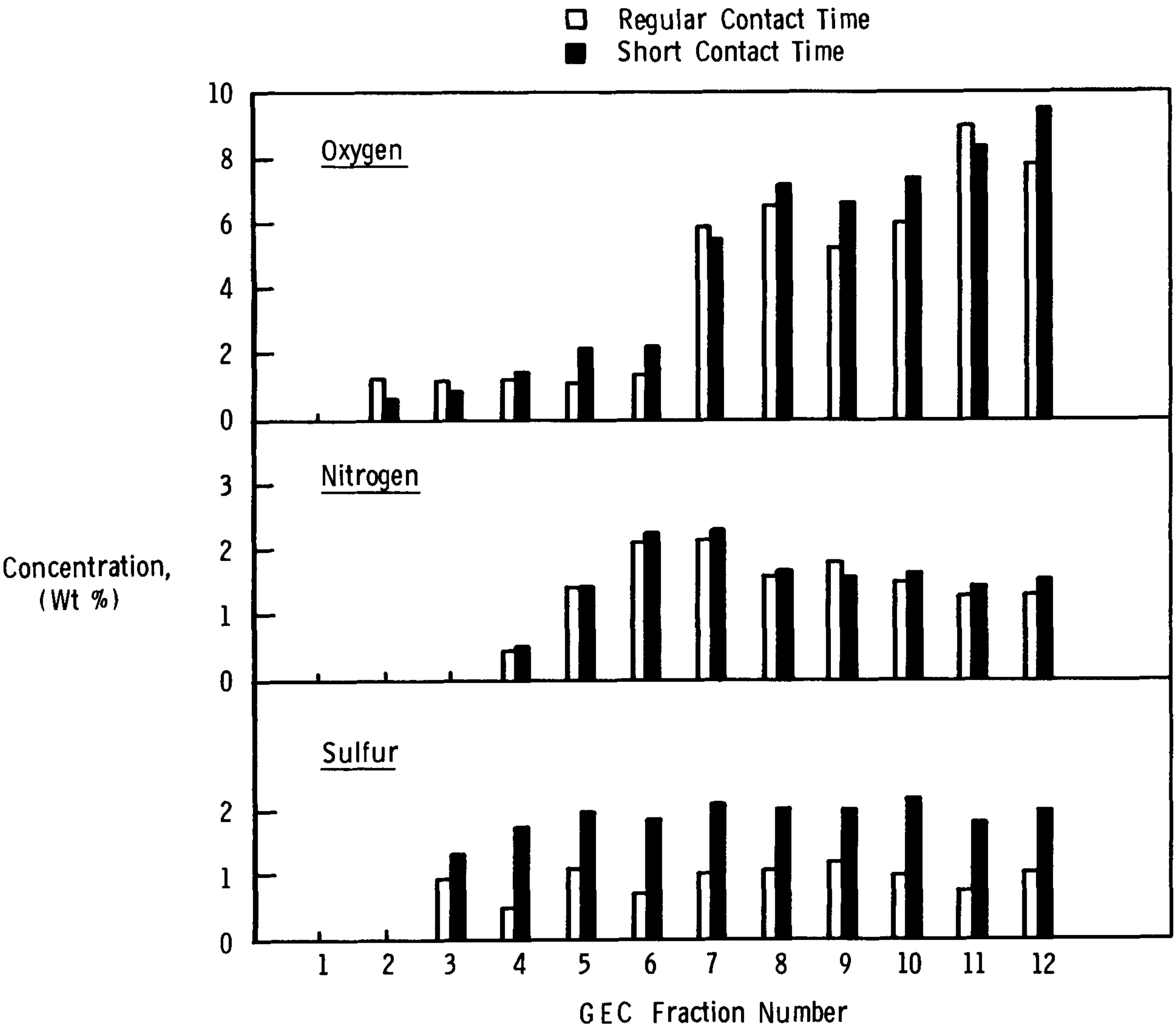

Figure 4-21. Elemental Composition of Short Contact Time and Regular Monterey SRC

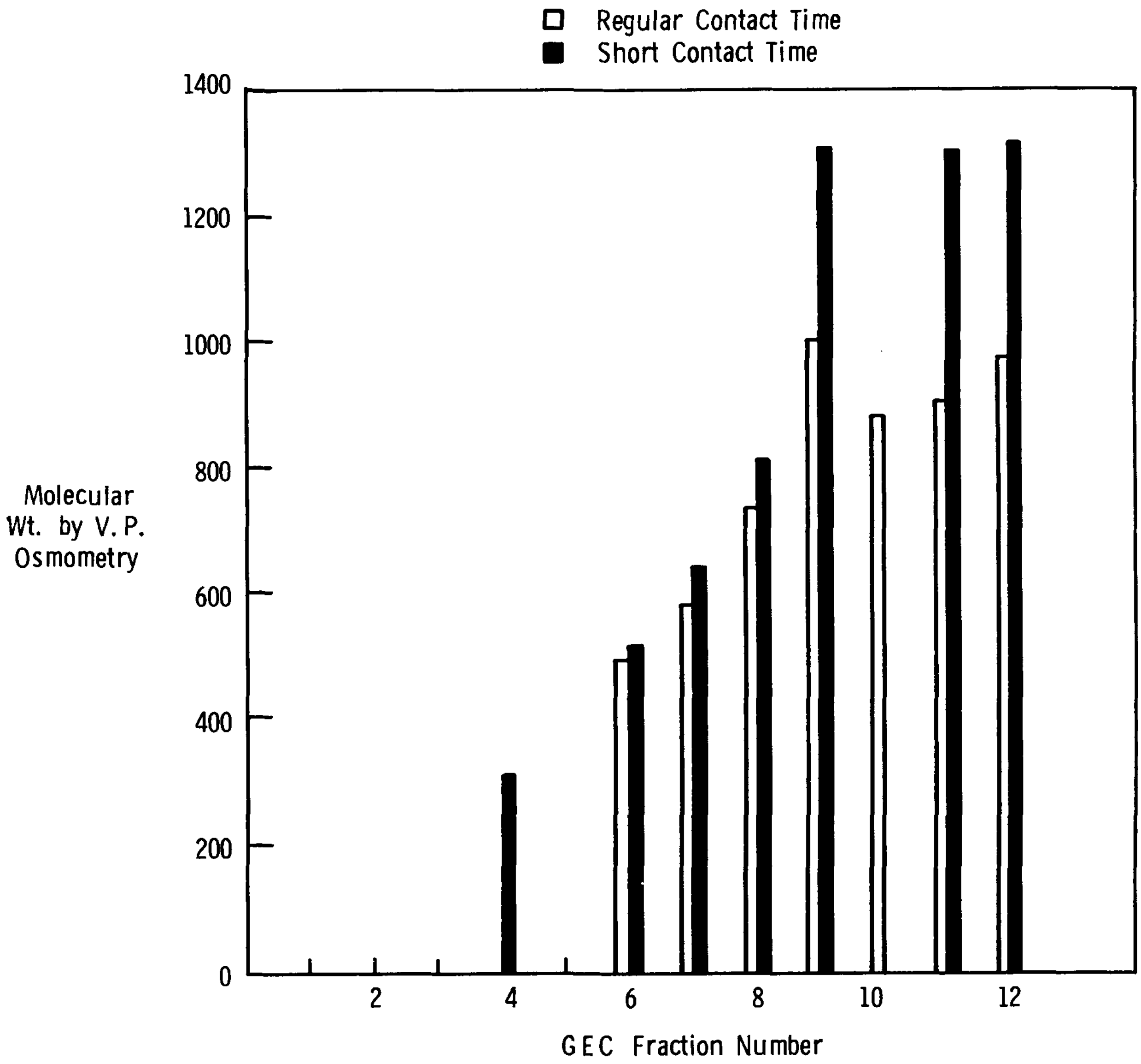

Figure 4-22 Molecular WT of GEC Cuts from Short Contact Time and Regular Monterey SRC

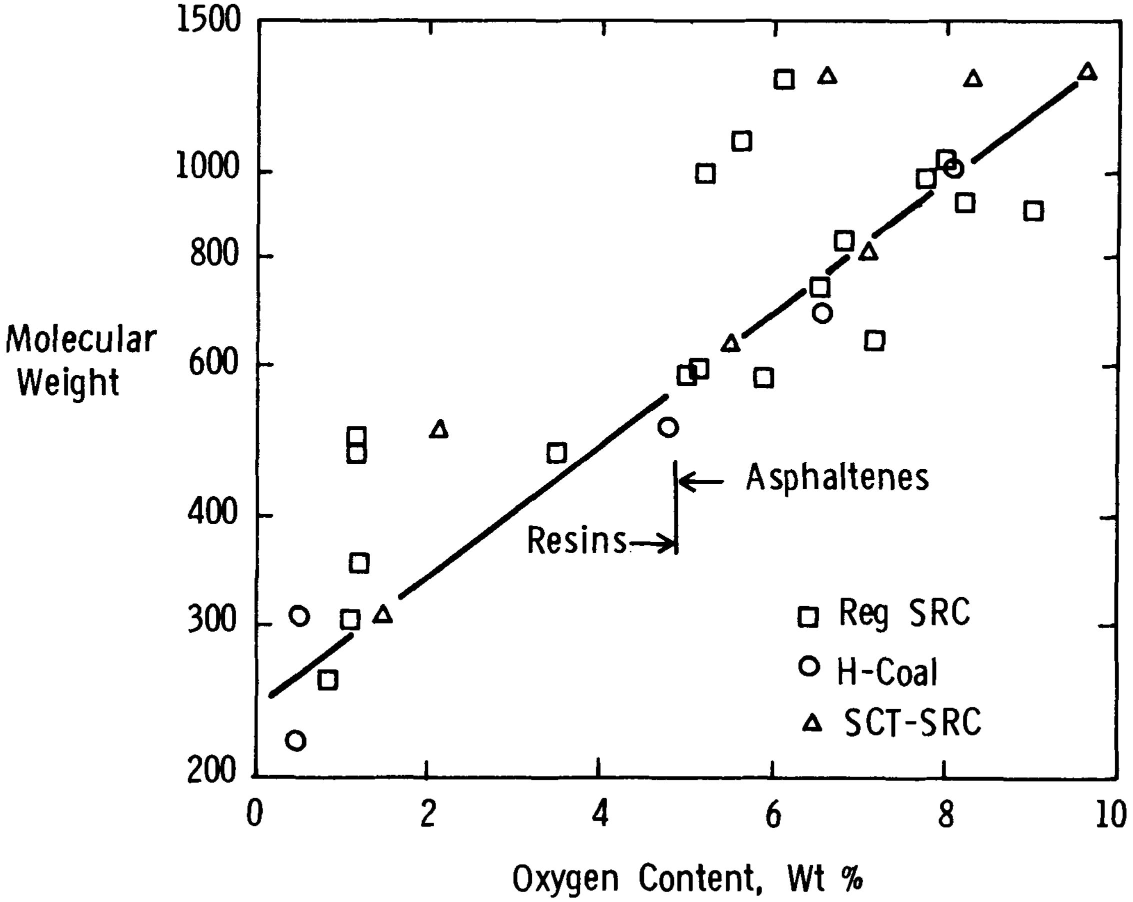

Figure 4-23. Molecular Weight Versus Oxygen Content for Resins and Polar Asphaltenes in Raw Coal Liquids

catalyst, Harshaw 618X, prevented the solid deasphalting in that case, and proved successful in this case as well. This helped to confirm that the pore size distribution is the critical catalyst parameter for hydroprocessing residual coal liquids.

Since the large pore catalyst was necessary to hydroprocess the SCT 33 wt % Monterey SRC blend, the regular 33 wt % Monterey SRC was also hydroprocessed over this catalyst to obtain a base case for comparison with the SCT SRC. The SCT SRC was also hydroprocessed over three Mobil catalysts.

SCT vs Regular Monterey SRC

The elemental and GEC analyses of the 33% blend of short contact time SRC (77D-453) is given in Table 4-42. Similar properties for the 33% blend of regular SRC (77D-1039) are shown for comparison. The SCT SRC blend is generally a heavier, more viscous material containing less hydrogen and more heteroatoms and trace metals than the regular SRC blend. The GEC analysis shows it to contain more than 30% more polar asphaltenes with proportionately less aromatic oils and resins/eluted asphaltenes than the regular blend.

A limited process variable study similar to that reported for the regular SRC blend (Section 4.3) was completed charging the 33 wt % SCT Monterey SRC blend over Harshaw 618X catalyst. A total of eight material balances were obtained over a range of temperatures (673-782°F) and space velocities (.19-90 LHSV) at a pressure of 2000 psig. The operating conditions, hydrogen utilization and liquid product properties for the SCT SCR blend are given in Table 4-43 together with the corresponding charge stock properties. Appendix Table B-13 includes the product yields on charge and the distillations of the liquid products. Tables 4-19 and B-6 in Section 9.3 contain the corresponding information for the regular Monterey SRC blend hydroprocessing study. Corresponding balances from the two studies are given in Table 4-44. These data show that, at a given severity of operation, the products from the regular and SCT SRC are very similar. Due to the lower initial hydrogen and higher heteroatom content, the average overall H_2 consumption is 700-800 scf/B greater for SCT SRC at the same operating severity. This results in a slightly higher hydrogen content for the SCT SRC product. With the scatter of the data considered, it would be very difficult to determine from the product analyses whether the feed was a regular or short contact time SRC.

Table 4-42

COMPARISON OF REGULAR AND SHORT CONTACT TIME 33% MONTEREY SRC BLENDS

	SCT	REGULAR
Mobil Identification	77D453	77D1039
Properties		
Gravity, °API	-5.9	-4.9
Hydrogen, wt %	6.51	7.00
Oxygen, wt %	4.5	3.7
Nitrogen, wt %	1.14	1.12
Sulfur, wt %	1.01	0.58
CCR, wt %	16.75	17.14
KV @ 212°F	415.8	13.4
% Aromatic Carbon	80	79
Ash, wt %	0.15	0.05
Trace Metals Analyses, ppm		
Sodium	39	3.4
Potassium	5	1.4
Calcium	120	4.2
Iron	194	98
Vanadium	7.5	1.5
Lead	1.0	0
Distillation, °F @ % (D-2887)		
IBP	364	351
5	398	397
10	433	418
30	490	483
50	588	591
70	-	-
90	-	-
95	-	-
FBP	-	-
GEC Analysis		
650°F⁻, wt %	49.0	48.3
Cut 1 (saturates)	0.3	0.4
Cut 2-4 (arom. oils)	6.0	10.9
Cut 5-7 (resins/asph.)	8.4	15.9
Cut 8-13 (polar asph.)	36.3	24.5
Total	100.0	100.0

Table 4-43

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33% BLEND SCT MONTEREY SRC/SOLVENT (88D-453)
CATALYST: HARSHAW 618X (J-7242)
PROGRAM: 2490 UNIT: CT-146

	CHARGE	MB-819	MB-820	MB-821	MB-822	MB-823	MB-824	MB-825	MB-826
OPERATING CONDITIONS									
TEMPERATURE, °F	-	720	782	769	721	673	676	725	729
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.42	0.60	0.39	0.21	0.19	0.57	0.30	0.90
DAYS ON STREAM	-	1.9	3.2	4.9	6.1	7.6	8.8	9.8	10.5
LIQUID PRODUCT PROPERTIES									
GRAVITY, °API	-5.9	8.4	12.1	11.6	12.4	6.1	0.4	9.4	3.6
HYDROGEN, WT °/°	6.51	9.05	9.98	9.72	10.28	9.45	8.23	9.62	8.54
SULFUR, WT °/°	1.01	0.15	0.07	0.15	0.08	0.17	0.37	0.12	0.23
NITROGEN, WT °/°	1.14	0.46	0.30	0.44	0.28	0.53	0.81	0.37	0.75
OXYGEN, WT °/°	4.5	1.4	0.8	1.3	1.0	2.8	3.0	1.6	2.7
CCR, WT °/°	16.75	8.57	5.43	6.43	4.71	5.67	10.80	5.85	13.51
K.V. AT 100°C	415.80	-	2.21	2.14	2.31	7.54	-	4.17	15.43
AROMATIC CARBON, °/°	80	-	-	49	-	52	-	48	-
HETEROATOM REMOVAL, PCT									
SULFUR	-	86.5	93.4	86.8	93.0	83.6	63.8	88.0	77.2
OXYGEN	-	71.8	83.2	74.6	79.7	39.0	34.2	65.3	41.2
NITROGEN	-	63.4	75.1	64.7	77.5	54.4	29.9	68.4	35.5
HYDROGEN UTILIZATION, SCF/B									
H2 CONSUMED BY C1-C5	-	589	435	634	585	175	65	99	129
H2 CONSUMED BY C6+	-	1562	2215	2015	2437	2026	1198	2154	1384
H2 CONSUMED BY S	-	41	44	41	44	39	30	42	36
H2 CONSUMED BY N	-	116	137	118	142	99	55	125	65
H2 CONSUMED BY O	-	303	351	314	336	165	144	275	174
H2 CONSUMED TOTAL	-	2610	3182	3122	3544	2505	1492	2695	1787

Table 4-44

HYDROPROCESSING REGULAR AND SCT MONTEREY SRC BLENDS

HYDROPROCESSING REGULAR AND SCT MONTEREY SRC BLENDS

Processing Conditions	Regular SRC	SCT SRC
H_2 Pressure, psig	2000	2000
Temperature, °F	777	782
LHSV, V/V/Hr	.50	.57

Liquid Product Properties	Charge	Product	Charge	Product
Gravity, °API	-4.9	12.2	-5.9	12.1
Hydrogen, Wt %	7.0	9.2	6.5	10.0
Sulfur, Wt %	0.58	.04	1.01	.07
Nitrogen, Wt %	1.12	.32	1.14	.30
Oxygen, Wt %	3.7	1.0	4.5	.8
CCR, Wt %	17.1	6.2	16.8	5.4
KV (100°C)	13.4	1.9	416	2.2
650^{-}°F	48.3	60.7	49.0	66.2
650^{+}°F Residue				
Cut 1 (Saturates)	0.4	1.4	0.3	2.1
Cut 2-4 (Arom. Oils)	10.9	24.9	6.0	19.7
Cut 5-7 (Resins/Asphalt)	15.9	9.9	8.4	8.2
Cut 8-13 (Polar Asphalt)	24.5	3.1	36.3	3.8

The products from this SCT SRC run were distilled to yield an IBP-650°F cut and a 650°F^{+} bottoms. The elemental analyses obtained on these cuts are given in Table 4-45. Similar cut properties were previously reported for the regular Monterey SRC study (Table 4-21). Calculating TLP hydrogen values from the 650°F^{-} and 650°F^{+} fractions, tends to give values $\sim$0.5 wt % lower than the measured values, indicating loss of material during the distillations, possibly some very light ends. Figure 4-24 shows that for a given hydrogen content in the 650°F^{-}, or solvent range material, the 650°F^{+} fraction will contain roughly the same hydrogen content. The data in this plot scatters due to a temperature effect on this selectivity. The charge and four of the 650°F^{+} fractions from this study were analyzed by our GEC technique (Table 4-46). Previously, we had reported the change in GEC composition of a 33 wt % blend of regular Monterey SRC as a function of hydroprocessing severity (Figures 4-3 and 4-4). Figures 4-25 and 4-26 show the SCT SRC data from Table 4-46. The SCT SRC seems to represent a step back the reaction path observed for regular SRC. Since this is a very steep portion of this path, this step is accomplished at very mild severity. The polar and non-eluted asphaltenes fraction from the hydroprocessed SCT SRC is only slightly higher than for the regular SRC at the same total hydrogen content.

The elemental characterization of the 650°F^{+} GEC fractions from two of these balances plus some comparison runs from the regular 33 wt % Monterey SRC run over Harshaw 618X are shown in Table 4-47. Comparison of the two charge stocks show the typical decrease in hydrogen content going from oils (Cuts 1-4) to resins (Cuts 5-7) to asphaltene (Cuts 8-12) fractions. Oxygen and sulfur contents are seen to increase with the higher molecular weight fractions, with the nitrogen content peaking in the resin cut as was noted previously. The only apparent abnormalities observed (other than those attributed to heteroatom concentration differences in the charge stocks), are the slightly higher hydrogen contents in the resin and asphaltene fractions of the SCT SRC blend as compared to the regular blend. These differences tend to confirm that during the non-catalytic preparation of solvent refined coal, longer times in the dissolver tend to further condense the already large aromatic molecules or to remove hydrogen-rich alkyl-side chains.

The hydroprocessed samples also tend to show the higher molecular weight components of the SCT blend to have higher hydrogen contents than the corresponding GEC fractions of the regular blend, with the saturate and oil hydrogen contents lower. Hydrogenation does occur preferentially in the lighter cuts for both blends.

Table 4-45

ANALYSES OF TOTAL LIQUID PRODUCT AND DISTILLATION CUTS FROM 33% MONTEREY SCT SRC BLEND

	CHARGE 77D453	MB-820	MB-821	MB-823	MB-825
Process Conditions					
Temperature, °F	-	782	769	673	725
LHSV	-	0.57	0.32	0.19	0.31
H_2 Consumed, scf/B	-	3198	2940	2505	2693
Days On-Stream	-	3.2	4.9	7.6	9.8
Material Balance, wt %	-	105.7	122.2	99.1	97.7
Total Liquid Product					
Hydrogen, wt %	6.51	9.98	9.34	9.36	9.62
Oxygen, wt %	4.5	0.8	1.25	2.8	1.6
Nitrogen, wt %	1.14	0.3	0.44	0.53	0.37
Sulfur, wt %	1.01	0.07	0.146	0.169	0.101
$650°F^-$, wt %	49.04	66.19	58.18	56.16	62.93
IBP-650°F Cut					
Hydrogen, wt %	7.39	10.22	9.68	9.65	9.73
Oxygen, wt %*	2.9	0.39	1.1	2.6	1.1
Nitrogen, wt %	0.6	(a)	(a)	0.04	(a)
Sulfur, wt %	0.31	0.015	0.021	0.011	0.012
$650°F^+$ Bottoms					
Hydrogen, wt %	5.86	8.21	7.64	8.14	8.25
Oxygen, wt %	6.0	1.6	1.5	3.0	2.4
Nitrogen, wt %	1.66	0.94	1.15	1.16	1.0
Sulfur, wt %	1.68	0.18	0.32	0.37	0.25

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* - Calculated from wt % hydrogen in total liquid product and $650°F^+$ fraction.

(a) - Calculated value is less than zero.

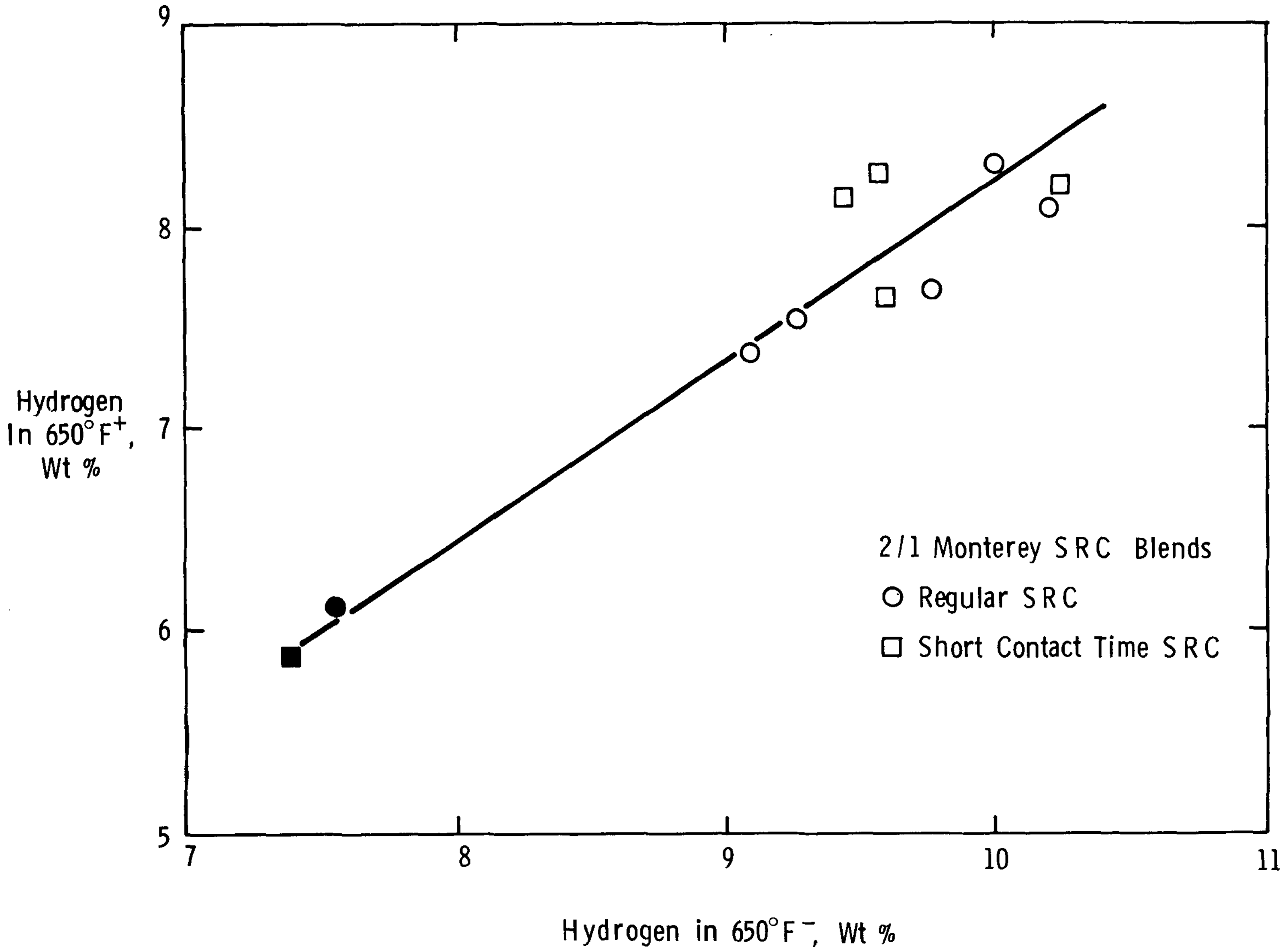

Figure 4-24. Hydrogen in 650°F+ Bottoms as a Function of Hydrogen in 650°F- Overhead

Table 4-46

GEC ANALYSES OF HYDROPROCESSED
33% MONTEREY SCT SRC BLEND

	77D453 CHARGE	MB-821	MB-825	MB-820
Temperature, °F	-	769	725	782
LHSV	-	0.32	0.31	0.57
Pressure, psig	-	2000	2000	2000
H_2 Consumed, scf/B	-	2940	2693	3198
<u>Total Liquid Product</u>				
Hydrogen, wt %	6.51	9.34	9.62	9.98
Oxygen, "	4.5	1.25	1.6	0.80
Nitrogen, "	1.14	0.44	0.37	0.30
Sulfur, "	1.01	0.146	0.101	0.07
650°F$^-$, wt %	49.04	58.18	62.93	66.19
<u>650°F$^+$ Residue</u>				
Cut 1 (saturates)	0.26	1.69	1.40	2.09
Cut 2-4 (arom. oils)	5.95	21.42	17.31	19.73
Cut 5-7 (resins/asph.)	8.42	11.60	11.17	8.15
Cut 8-13 (polar asph.)	36.33	7.11	7.19	3.84
Total	100.00	100.00	100.00	100.00

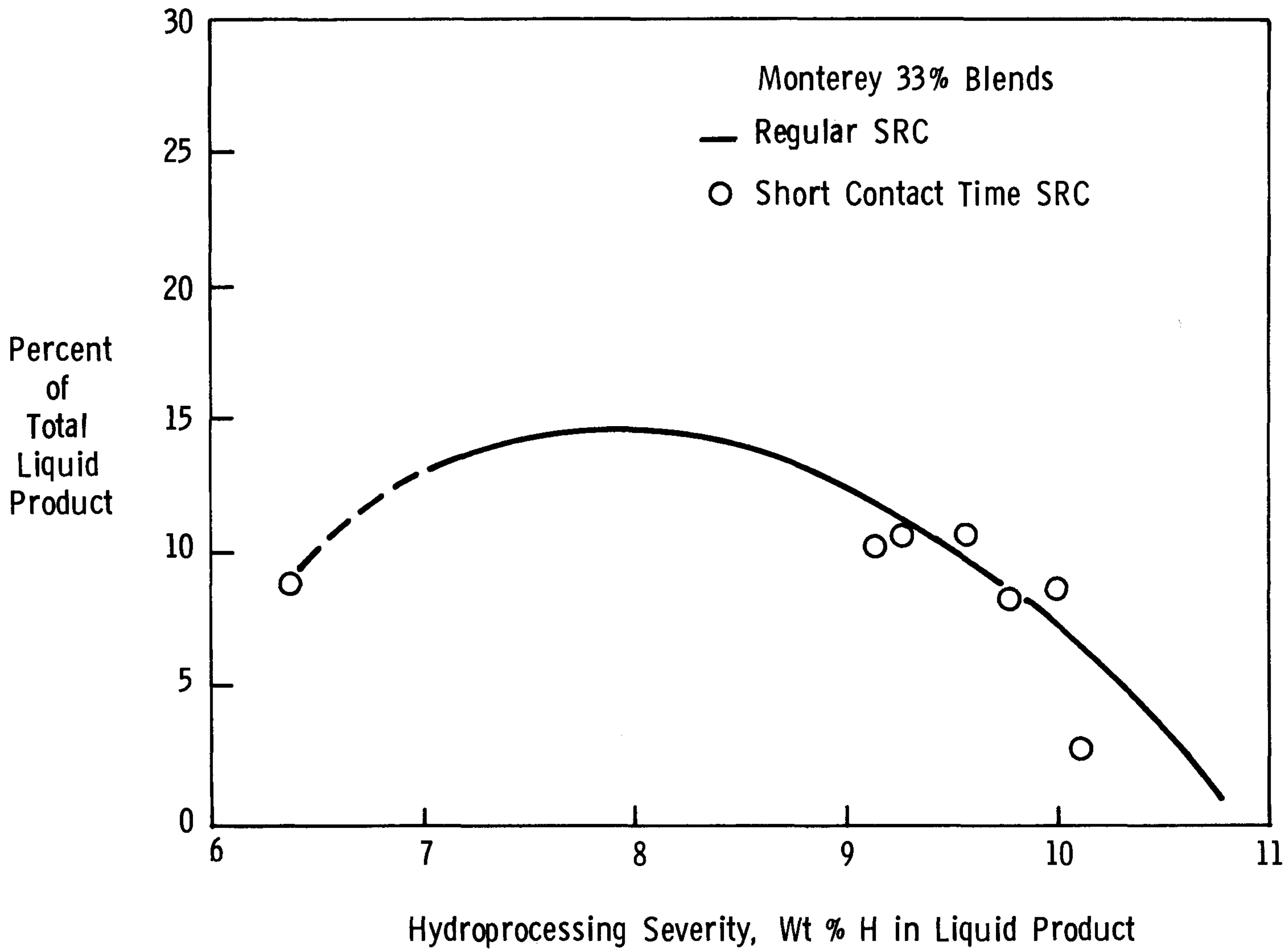

Figure 4-25. Resins and Eluted Asphaltenes in Monterey SRC Blends as a Function of Hydroprocessing Severity

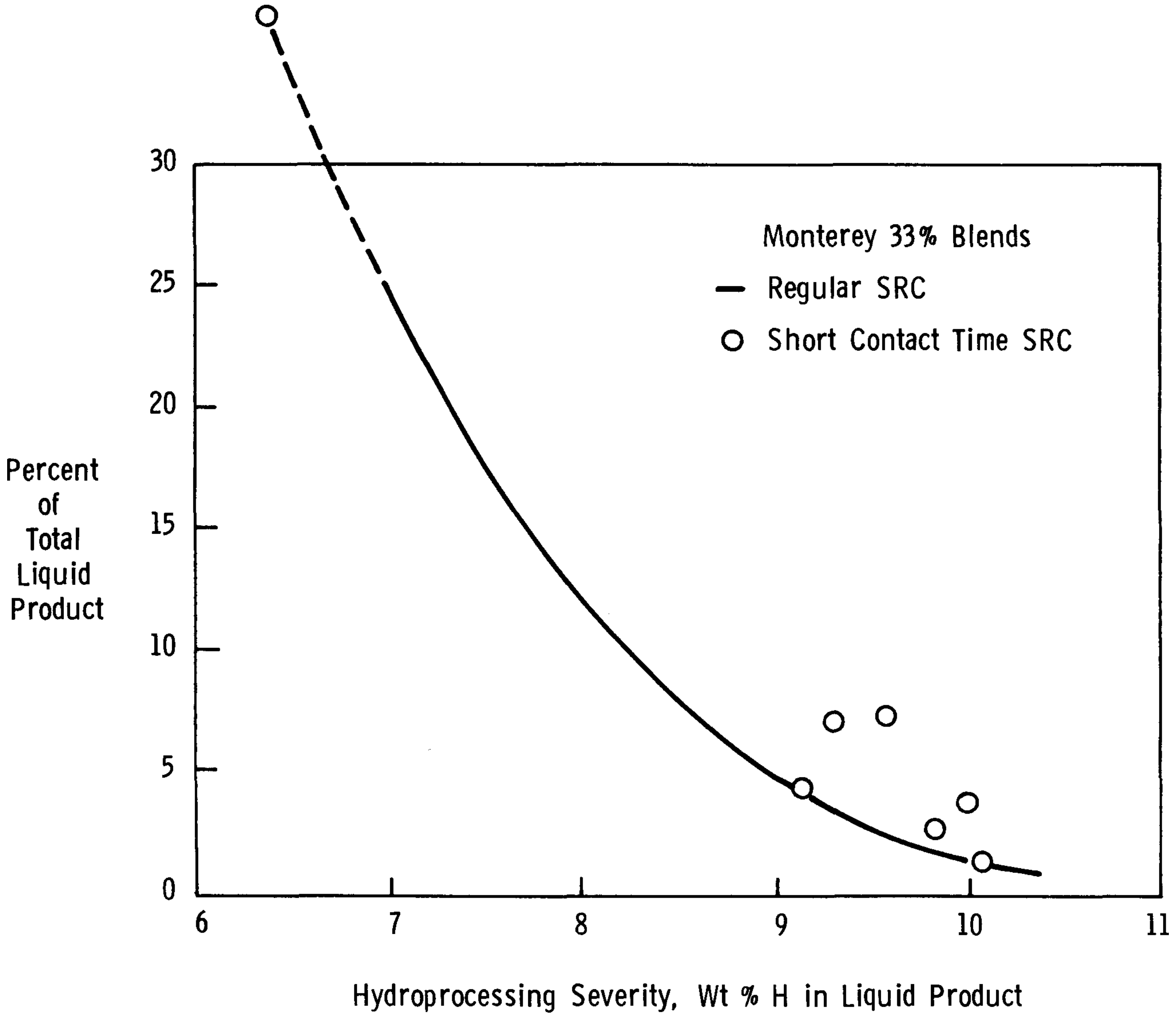

Figure 4-26. Polar and Noneluted Asphaltenes in Monterey SRC Blends as a Function of Hydroprocessing Severity

Table 4-47

ELEMENTAL ANALYSES OF GEC FRACTIONS FROM HYDROPROCESSED 33% MONTEREY SCT AND REGULAR SRC BLENDS

	PROGRAM 2501 REGULAR MONTEREY SRC BLEND			PROGRAM 2490 SCT MONTEREY SRC BLEND		
	77D1039 CHARGE	MB-828	MB-831	77D453 CHARGE	MB-820	MB-825
Process Conditions						
Temperature, °F	--	777	724	--	782	725
LHSV, V/Hr/V	--	0.50	0.25	--	0.57	0.31
Pressure, psig	--	2000	2000	--	2000	2000
H_2 Cons., scf/B	--	2096	2212	--	3198	2693
$650°F^+$ Residue, Wt %	51.64	39.28	38.75	50.96	33.81	37.07
Hydrogen, Wt %						
$650°F^+$ Residue	6.11	7.68	8.31	5.86	8.03	8.25
Cut 1	(a)	12.84	13.02	(a)	12.64	12.78
Cut 2-4	6.84	8.53	8.79	6.54	7.92	9.10
Cut 5-7	6.34	6.62	7.09	6.67	7.04	7.81
Cut 8-12	5.67	5.54	6.28	5.83	6.11	6.64
Oxygen, Wt %						
$650°F^+$ Residue	3.9	0.9	0.7	6.0	1.6	2.4
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	1.0	0.9	0.8	1.0	0.7	0.6
Cut 5-7	3.8	2.0	2.5	5.0	2.2	2.8
Cut 8-12	6.3	3.7	4.0	7.5	4.6	5.6
Nitrogen, Wt %						
$650°F^+$ Residue	1.58	0.68	0.75	1.66	0.94	1.00
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	(0.47)	0.18	0.24	0.44	0.24	0.31
Cut 5-7	2.55	1.85	1.73	2.39	1.89	1.89
Cut 8-12	1.29	1.33	1.13	1.77	1.37	1.13
Sulfur, Wt %						
$650°F^+$ Residue	0.25	0.10	0.09	1.68	0.18	0.25
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	0.59	0.07	<0.03	(a)	0.15	0.18
Cut 5-7	0.75	<0.03	0.03	1.38	0.07	0.23
Cut 8-12	1.03	(a)	(a)	1.86	(a)	(a)

(a) Analysis not determined.

In the case of oxygen, the main difference in its distribution is in the asphaltene fraction, where the SCT products contain a considerably larger amount of oxygen. The oils and resins product cuts from both charge stocks have similar levels of oxygen even though the overall 650°F$^+$ residues have significantly different concentrations. The distribution of nitrogen in the cuts does not appear to be a function of the type of SRC, as both charge stocks have similar initial concentrations.

An interesting phenomenon is observed when hydroprocessing SCT SRC as shown in Figure 4-27, a plot of the viscosity of the hydroprocessed SCT and regular SRC blends. Although the viscosity of the SRC blends differ by a factor of 30, hydroprocessing at moderate to high severity results in products with nearly equivalent viscosities.

Several of the hydroprocessed samples from the SCT SRC study were also analyzed to determine the extent of demetalation that occurred. These data are presented in Table 4-48.

Table 4-48

METALS CONTENT OF HYDROPROCESSED SCT MONTEREY SRC BLEND

	H_2 CONSUMPTION scf/B	V	Fe	Ca	Na	K
33 wt % SCT SRC Blend	-	7.5	194	120	39	5
MB-824	1492	5.5	89	110	17	.6
MB-826	1787	6.0	66	-	18	5
MB-823	2505	2.2	-	-	1.3	-
MB-825	2693	2.2	19	-	-	-
MB-822	3568	-	2.7	3.2	.9	.2

These data show a significant reduction in trace metals, particularly at the high severities. The levels are somewhat higher than for the regular SRC reported in Section 4.2, however, the trend is the same.

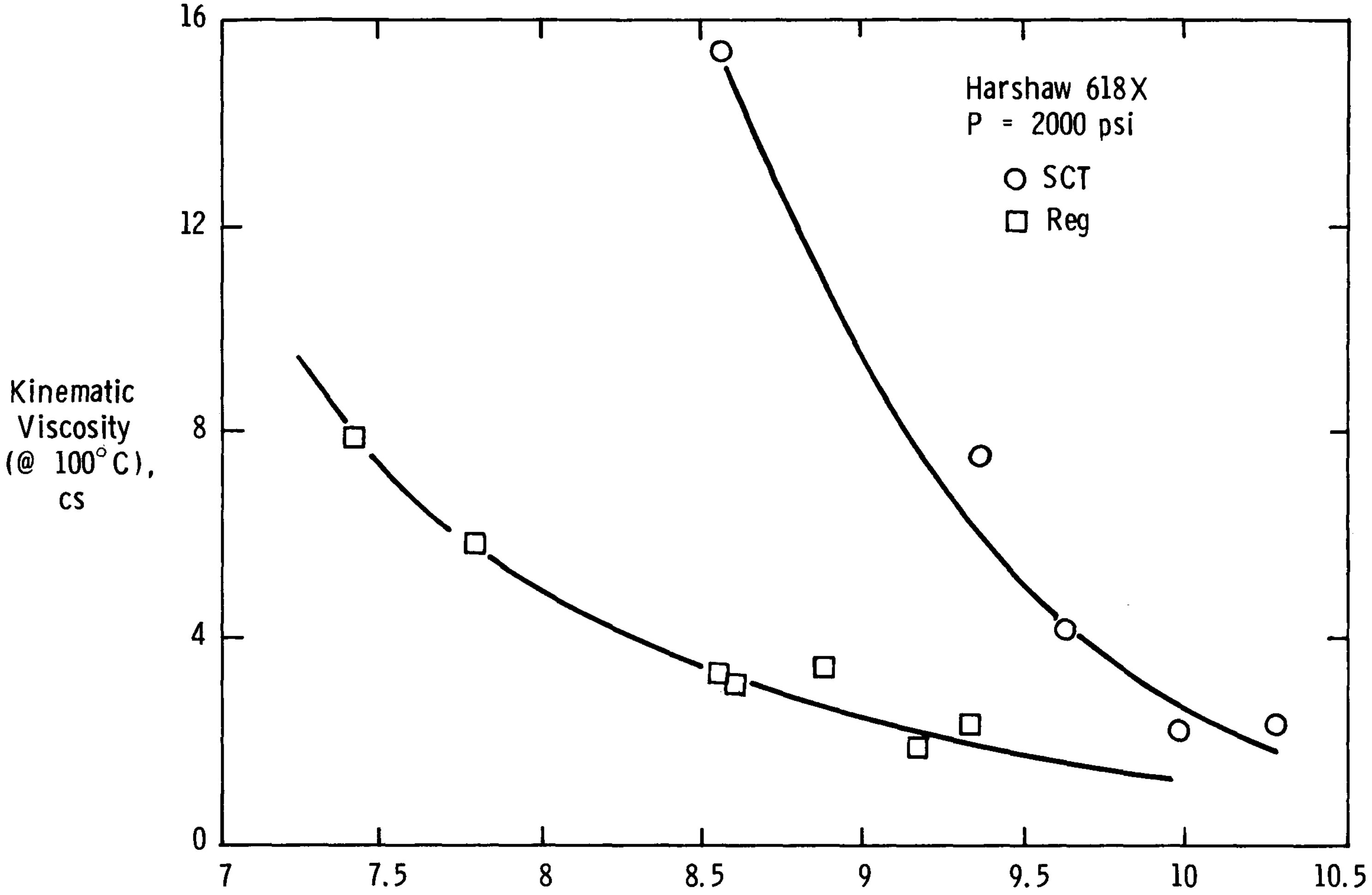

Figure 4-27. Kinematic Viscosity as a Function of Hydrogen Content for Hydroprocessing Regular and Short Contact Time SRC Blends

The data were modeled using a second order rate equation. The kinetic constants determined from our modified Marquardt fitting routine are given in Table 4-49. Comparisons of the rate constant at 750°F ($k_{750°F}$) gives an indication of the relative rates of removal of heteroatom from these liquids. The data from the SCT and regular SRC's were modeled separately and collectively to determine the kinetic constants. The values of $k_{750°F}$ for the regular SRC are consistently higher than for the SCT SRC. This is particularly true for the sulfur. However, the conversion for a second order reaction increases with increasing concentration. Since the sulfur concentration in the SCT SRC blend is almost twice that in the regular SRC blend, its conversion will be high despite its low $k_{750°F}$ Figure 4-28 shows that the sulfur concentration should be low (<0.3 wt %) for either blend when hydroprocessed at moderate conditions (2000 psia, 750°F, <1.5 LHSV). The nitrogen contents will be significantly higher and roughly equivalent for either blend at similar conditions (Figure 4-29).

SCT Monterey SRC: Effect of Catalyst

Use of the commercial Harshaw 618X catalyst proved very successful in hydroprocessing high residual containing coal liquids. No reactor plugging or formation of non-homogeneous products were associated with its use over a wide range of operating conditions. In an attempt to develop leads for a catalyst that would show an improvement over the commercial Harshaw 618X catalyst, short evaluation studies were performed on three Mobil proprietary catalysts; designated Catalysts A, B, and C. A 33 wt % SCT Monterey SRC blend was processed over each catalyst at similar conditions. These studies were meant to obtain relative catalyst activity data on two catalyst concepts; the programs were not designed to test or evaluate relative catalyst aging stabilities. No attempt was made to optimize catalysts specifically for hydroprocessing short contact time SRC coal liquids. All three hydroprocessing studies were conducted in our modified unit (CT-146) at 2000 psi hydrogen pressure. Eight material balances were completed over Catalyst A at conditions ranging from 670-785°F and 1.0 to 0.3 LHSV. Four material balances at the operating conditions of 725 and 775°F and 0.25 and 0.5 LHSV were completed using Catalyst B and four balances were made over Catalyst C at conditions of 775 and 790°F and 0.5 and 0.25 LHSV. All runs were terminated voluntarily.

Table 4-49

CONSTANTS FOR KINETIC MODEL FOR HETEROATOM REMOVAL FROM 33% MONTEREY SCT AND REGULAR SRC BLENDS

Catalyst: Harshaw 618X

Second Order Rate Equation*

		$k_{(750°F)}$	k_o	E, Btu/ # MOLE	AVG. DEVIATION WT %
Nitrogen	- All data	0.674	1.02×10^{6}	34,213	0.067
	- SCT only	0.644	1.18×10^{7}	40,210	0.069
	- REG only	0.734	3.88×10^{5}	31,682	0.048
Oxygen	- All data	0.253	1.32×10^{10}	59,329	0.308
	- SCT only	0.235	6.89×10^{10}	63,487	0.316
	- REG only	0.295	5.84×10^{8}	51,467	0.215
Sulfur	- SCT only	3.687	1.35×10^{9}	47,409	0.035
	- REG only	7.253	1.74×10^{8}	40,856	0.010

* $\frac{dC_i}{dt} = k_o e^{-E/RT} C_i^2$

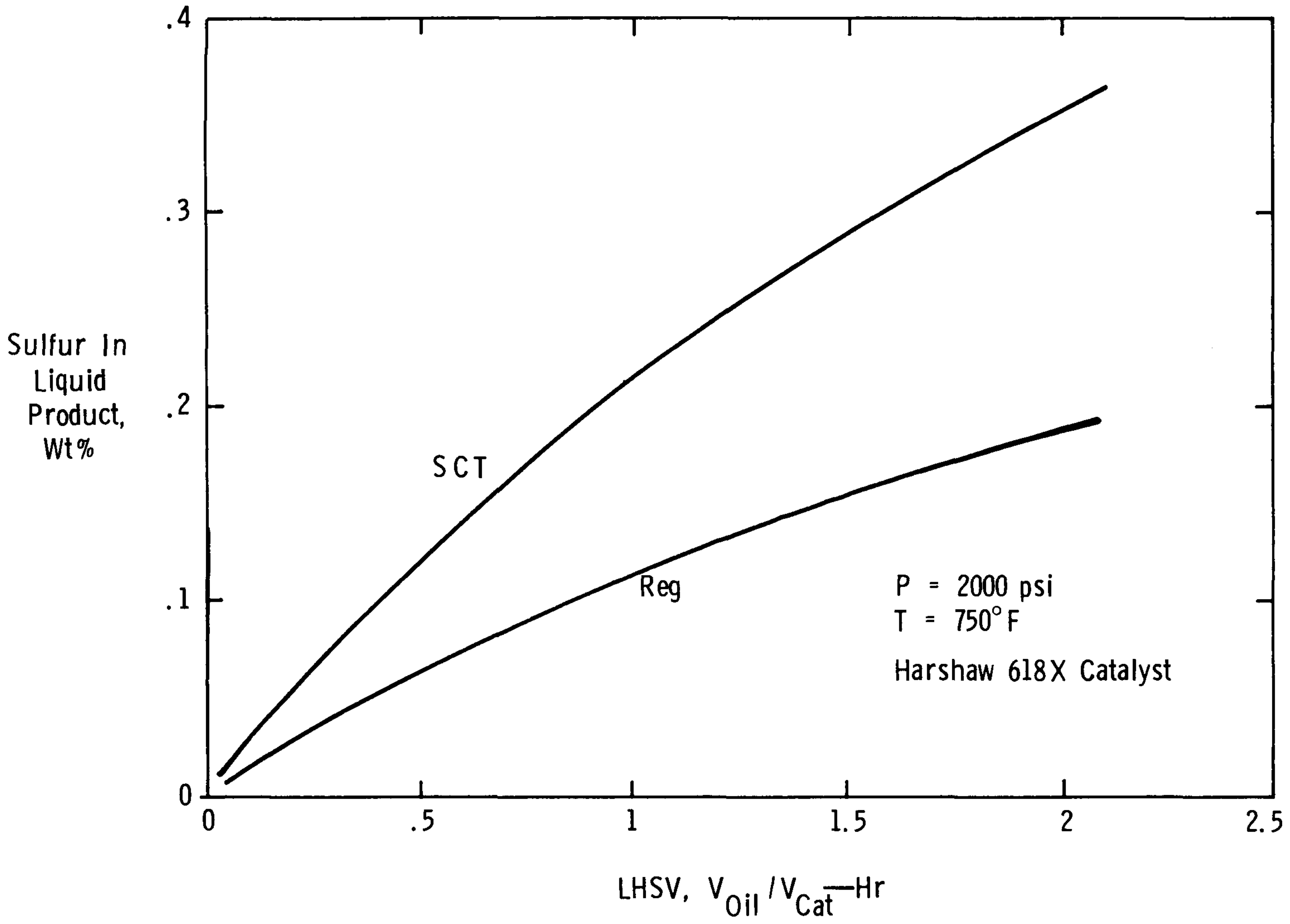

Figure 4-28. Model Prediction of Sulfur Concentration Levels for Hydroprocessing Regular and Short Contact Time SRC Blends

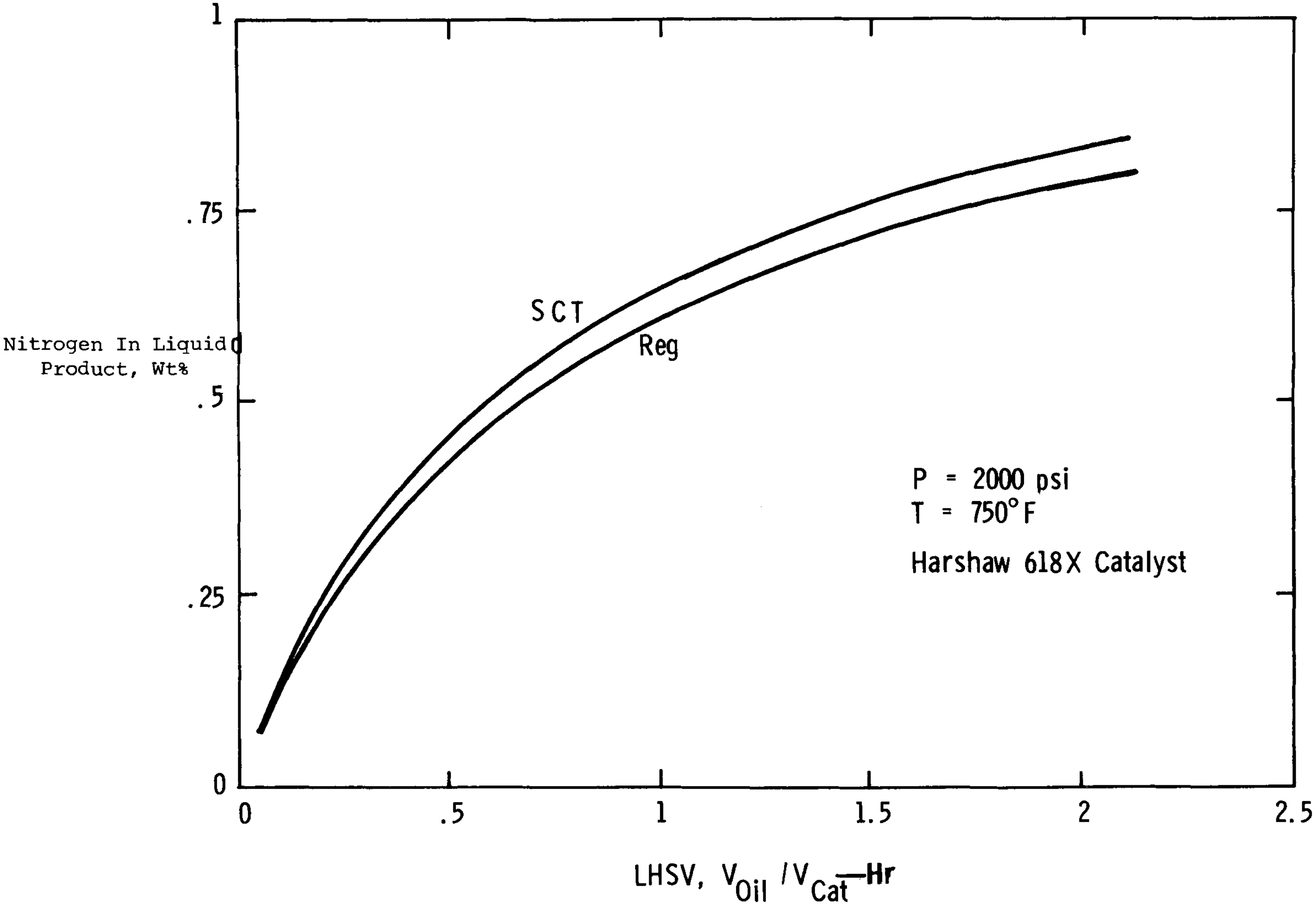

Figure 4-29. Model Prediction of Nitrogen Concentration Levels for Hydroprocessing Regular and Short Contact Time SRC Blends

The operating conditions, hydrogen utilization and liquid product properties for Mobil Catalysts A, B, and C are given in Tables 4-50, 51, and 52 respectively. Appendix Tables B-14, 15, and 16 list the product yields on charge and the distillations of the liquid products for the three catalysts. The product from the runs made with Catalyst C contained a fine-grained solid precipitate that was recovered by vacuum filtration. The yields of liquid and wet solid based on the total product from each of the balances are included in Table 4-50 together with the elemental analyses of the separate phases. The combined properties of the liquid and solid phases were utilized in determining the material balances listed in Table 4-52.

Table 4-54 compares the properties of the liquid product from the three Mobil catalysts with the Harshaw 618X catalyst at nearly equivalent processing conditions using the same SCT SRC blend. The elemental analyses show that at essentially equivalent operating conditions, the total product from Catalyst B and the liquid fraction from Catalyst C gave products as good as or better than the product obtained from the best commercial catalyst evaluated (Harshaw 618X). However, since Catalyst C also yielded a solid precipitate, the total product was of lower quality.

The activity for heteroatom removal for the Mobil Catalysts A & B relative to Harshaw 618X is shown in Figures 4-30 to 32. In all three figures the solid line is the percent heteroatom removal for the Harshaw 618X catalyst as determined by the second order rate constants presented in Table 4-49 (for SCT SRC) at 2000 psig hydrogen pressure. These figures show the denitrogenation, deoxygenation, and desulfurization as a function of space velocity at 725°F. The points on these plots are the actual percent heteroatom removal for the three Mobil catalysts at the corresponding space velocities. These data show that the Mobil B catalyst is equal to or better than the Harshaw 618X catalysts, while the Mobil A catalysts is not as good.

The GEC compositional data on the $650°F^+$ products from the material balances at equivalent operating severities for the four catalysts are also included in Table 4-54. Figure 4-33 shows a graphical comparison of the GEC composition data, based on total product, for the four catalysts.

Table 4-50

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33% BLEND SCT MONTEREY SRC/SOLVENT (77D-543)
CATALYST: MOBIL CATALYST 'A'
PROGRAM: 2525 UNIT: CT-146

	CHARGE	*MB-836*	*MB-837*	*MB-838*	*MB-839*	*MB-840*	*MB-841*	*MB-842*	*MB-843*
OPERATING CONDITIONS									
TEMPERATURE, °F	-	728	779	673	671	727	784	723	726
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.41	0.52	0.51	1.03	0.97	1.07	0.51	0.30
DAYS ON STREAM	-	1.6	2.6	3.6	4.2	4.7	5.2	6.1	7.2
LIQUID PRODUCT PROPERTIES									
GRAVITY, °API	-5.9	7.4	11.2	-0.4	1.5	0.5	3.6	1.9	5.6
HYDROGEN, WT °/°	6.51	8.88	9.18	7.33	7.07	7.50	7.90	7.74	8.77
SULFUR, WT °/°	1.01	0.22	0.14	0.50	0.59	0.43	0.26	0.38	0.19
NITROGEN, WT °/°	1.14	0.56	0.46	0.88	0.93	0.87	0.82	0.81	0.60
OXYGEN, WT °/°	4.5	1.9	1.1	3.5	4.1	3.3	2.4	3.0	2.0
CCR, WT °/°	16.75	8.74	6.97	10.64	13.62	13.01	10.72	12.80	8.42
K.V. AT 100°C	415.80	3.45	2.59	17.51	28.00	10.36	3.90	6.74	2.95
AROMATIC CARBON, °/°	80	-	55	67	-	71	-	66	-
HETEROATOM REMOVAL, PCT									
SULFUR	-	79.1	87.1	51.3	42.1	60.0	75.3	63.3	83.0
OXYGEN	-	59.0	76.7	23.5	9.7	31.0	48.6	34.9	59.5
NITROGEN	-	52.0	61.5	24.1	19.2	28.2	30.7	30.6	52.1
HYDROGEN UTILIZATION, SCF/B									
H2 CONSUMED BY C1-C5	-	145	313	51	49	312	237	140	517
H2 CONSUMED BY C6+	-	1587	1687	540	367	643	836	776	1416
H2 CONSUMED BY S	-	37	41	24	20	28	36	30	39
H2 CONSUMED BY N	-	95	112	44	35	52	56	56	95
H2 CONSUMED BY O	-	249	323	99	41	131	205	147	251
H2 CONSUMED TOTAL	-	2114	2477	759	512	1165	1369	1149	2319

Table 4-51

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33% BLEND SCT MONTEREY SRC/SOLVENT (77D-453)
CATALYST: MOBIL CATALYST 'B'
PROGRAM: 2522 UNIT: CT-146

	CHARGE	*MB-844*	*MB-845*	*MB-846*	*MB-847*
OPERATING CONDITIONS					
TEMPERATURE, °F	-	724	778	726	777
PRESSURE, PSIG	-	2000	2000	2000	2000
LHSV	-	0.50	0.50	0.24	0.18
DAYS ON STREAM	-	1.6	2.6	4.1	5.5
LIQUID PRODUCT PROPERTIES					
GRAVITY, °API	-5.9	9.1	14.2	10.2	16.3
HYDROGEN, WT °/°	6.51	9.06	9.76	9.15	10.07
SULFUR, WT °/°	1.01	0.15	0.06	0.10	0.09
NITROGEN, WT °/°	1.14	0.53	0.32	0.45	0.18
OXYGEN, WT °/°	4.5	1.6	0.8	1.2	0.5
CCR, WT °/°	16.75	8.62	4.83	5.74	1.21
K.V. AT 100°C	415.80	3.54	1.60	2.36	1.37
AROMATIC CARBON, °/°	80	-	46	-	43
HETEROATOM REMOVAL,PCT					
SULFUR	-	86.0	94.7	90.3	91.7
OXYGEN	-	65.7	83.1	74.5	92.3
NITROGEN	-	55.3	73.1	62.5	85.3
HYDROGEN UTILIZATION,SCF/B					
H2 CONSUMED BY C1-C5	-	139	412	290	520
H2 CONSUMED BY C6+	-	1719	2039	1674	2104
H2 CONSUMED BY S	-	41	45	43	45
H2 CONSUMED BY N	-	101	133	114	156
H2 CONSUMED BY O	-	277	350	314	519
H2 CONSUMED TOTAL	-	2276	2980	2435	3343

Table 4-52
FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33% BLEND SCT MONTEREY SRC/SOLVENT (77D-453)
CATALYST: MOBIL CATALYST 'C'
PROGRAM: 2528   UNIT: CT-146

	CHARGE	MB-848	MB-849	MB-850	MB-851
<u>OPERATING CONDITIONS</u>					
TEMPERATURE, °F	-	775	793	792	771
PRESSURE, PSIG	-	2000	2000	2000	2000
LHSV	-	0.45	0.51	0.24	0.28
DAYS ON STREAM	-	2.3	4.3	5.6	7.0
<u>LIQUID PRODUCT PROPERTIES</u>					
GRAVITY, °API	-5.9	12.6	11.0	13.6	12.4
HYDROGEN, WT °/°	6.51	9.82	9.42	9.88	9.44
SULFUR, WT °/°	1.01	0.32	0.12	0.02	0.15
NITROGEN, WT °/°	1.14	0.43	0.40	0.24	0.36
OXYGEN, WT °/°	4.5	2.1	1.1	0.5	1.4
CCR, WT °/°	16.75	8.20	6.69	4.57	7.35
K.V. AT 100°C	415.80	1.81	1.75	1.48	1.78
AROMATIC CARBON, °/°	80	47	49	46	45
<u>HETEROATOM REMOVAL,PCT</u>					
SULFUR	-	69.5	88.9	97.9	86.0
OXYGEN	-	55.1	77.2	89.6	70.8
NITROGEN	-	63.7	67.2	80.3	70.3
<u>HYDROGEN UTILIZATION,SCF/B</u>					
H2 CONSUMED BY C1-C5	-	441	593	611	645
H2 CONSUMED BY C6+	-	2226	1760	2032	1761
H2 CONSUMED BY S	-	33	42	46	41
H2 CONSUMED BY N	-	116	123	147	128
H2 CONSUMED BY O	-	232	325	378	298
H2 CONSUMED TOTAL	-	3048	2843	3214	2873

Table 4-53

ANALYSES OF HYDROPROCESSED 33% MONTEREY SCT SRC BLEND - MOBIL CATALYST C

	MB-848		MB-849		MB-850		MB-851	
Operating Conditions								
Temperature, °F	775		793		792		771	
Pressure, psig	2000		2000		2000		2000	
LHSV	0.45		0.51		0.24		0.26	
	FILTRATE	RESIDUE	FILTRATE	RESIDUE	FILTRATE	RESIDUE	FILTRATE	RESIDUE
Product Properties								
Wt % TLP, No Loss Basis	76	24	85	15	96	4	88	12
Gravity, °API	12.6	-	11.2	-	13.6	-	14.0	-
Hydrogen, wt %	10.08	8.99	9.55	8.68	10.01	6.76	9.88	6.24
Sulfur, wt %	0.07	1.10	0.08	0.34	0.01	0.25	0.08	0.61
Nitrogen, wt %	0.29	0.89	0.32	0.86	0.22	0.72	0.29	0.86
Oxygen, wt %	0.80	6.40	0.80	2.70	0.50	1.50	1.10	3.70
CCR, wt %	5.17	17.80	5.50	13.42	3.64	26.93	5.31	22.31
KV @ 100°C	1.81	-	1.75	-	1.48	-	1.78	-
Arom. Carbon, wt %	45	55	48	56	45	64	44	53

Table 4-54

EFFECT OF CATALYST ON ELEMENTAL ANALYSIS AND GEC COMPOSITION OF HYDROTREATED 33% MONTEREY SCT SRC BLEND

	CHARGE 77D453	HARSHAW 618X	MOBIL CAT. A	MOBIL CAT. B	MOBIL CAT. C		
					TOTAL PRODUCT	FILTRATE ONLY*	SOLID ONLY
Process Conditions							
Temperature, °F	-	782	779	778	775		
LHSV, V/Hr/V	-	0.57	0.52	0.50	0.45		
H_2 Consumed, scf/B	-	3198	2477	2980	3049		
Total Liquid Product							
Gravity, °API	-5.9	12.1	11.2	14.2	11.3	12.6	-
Hydrogen, wt %	6.51	9.98	9.18	9.76	9.82	10.1	8.99
Oxygen, wt %	4.5	0.80	1.10	0.80	2.1	0.8	6.4
Nitrogen, wt %	1.14	0.30	0.46	0.32	0.43	0.29	0.89
Sulfur, wt %	1.01	0.07	0.14	0.06	0.32	0.07	1.2
KV @ 100°C	415.8	2.2	2.6	1.6	-	1.8	-
650°F$^-$, wt %	49.04	66.19	63.30	64.96	50.99	67.09	-
650°F$^+$ Residue							
Cut 1 (saturates)	0.26	2.09	1.24	2.41	3.60	2.23	7.92
Cut 2-4 (arom. oils)	5.95	19.73	17.71	22.05	26.47	19.86	47.41
Cut 5-7 (resins/asphaltenes)	8.42	8.15	11.20	8.28	7.65	7.40	8.44
Cut 8-13 (polar asphalt/non-eluted)	36.33	3.84	6.55	2.30	11.29	3.42	36.23**

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*76% of total product.

**54% of this fraction is from Cut 13.

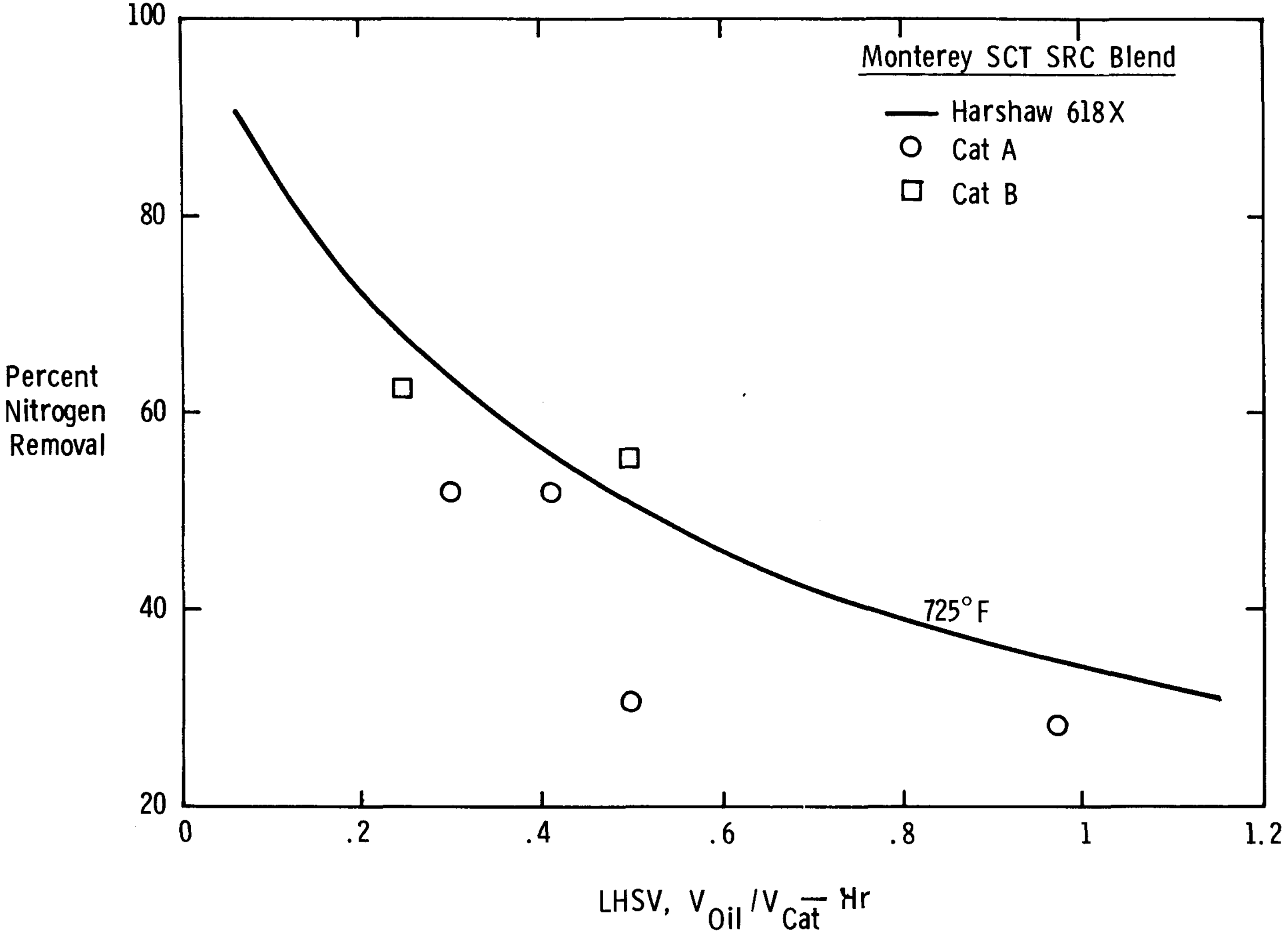

Figure 4-30. Catalyst Comparison for Denitrogenation of Monterey SCT SRC Blend

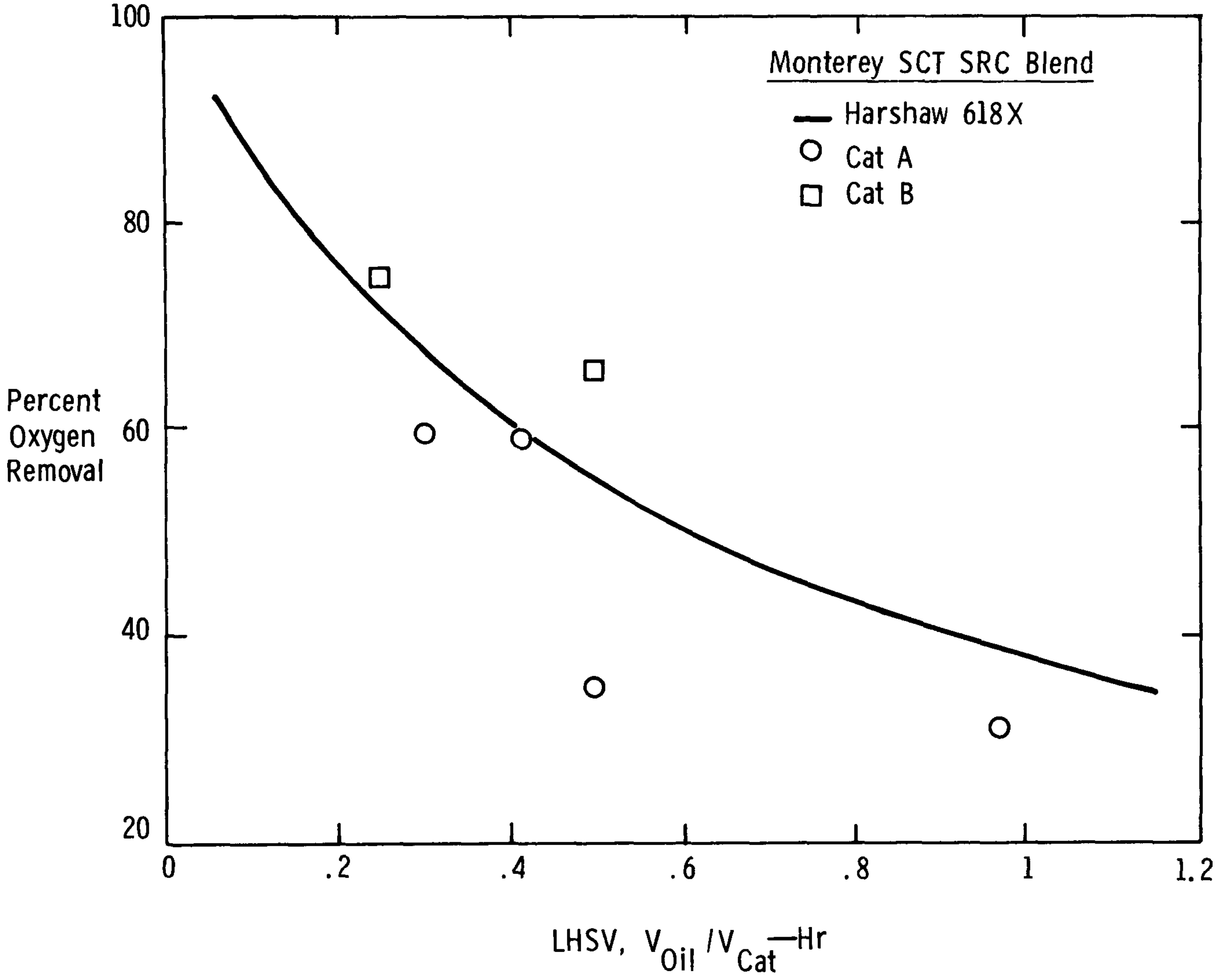

Figure 4-31. Catalyst Comparison for Deoxygenation of Monterey SCT SRC Blend

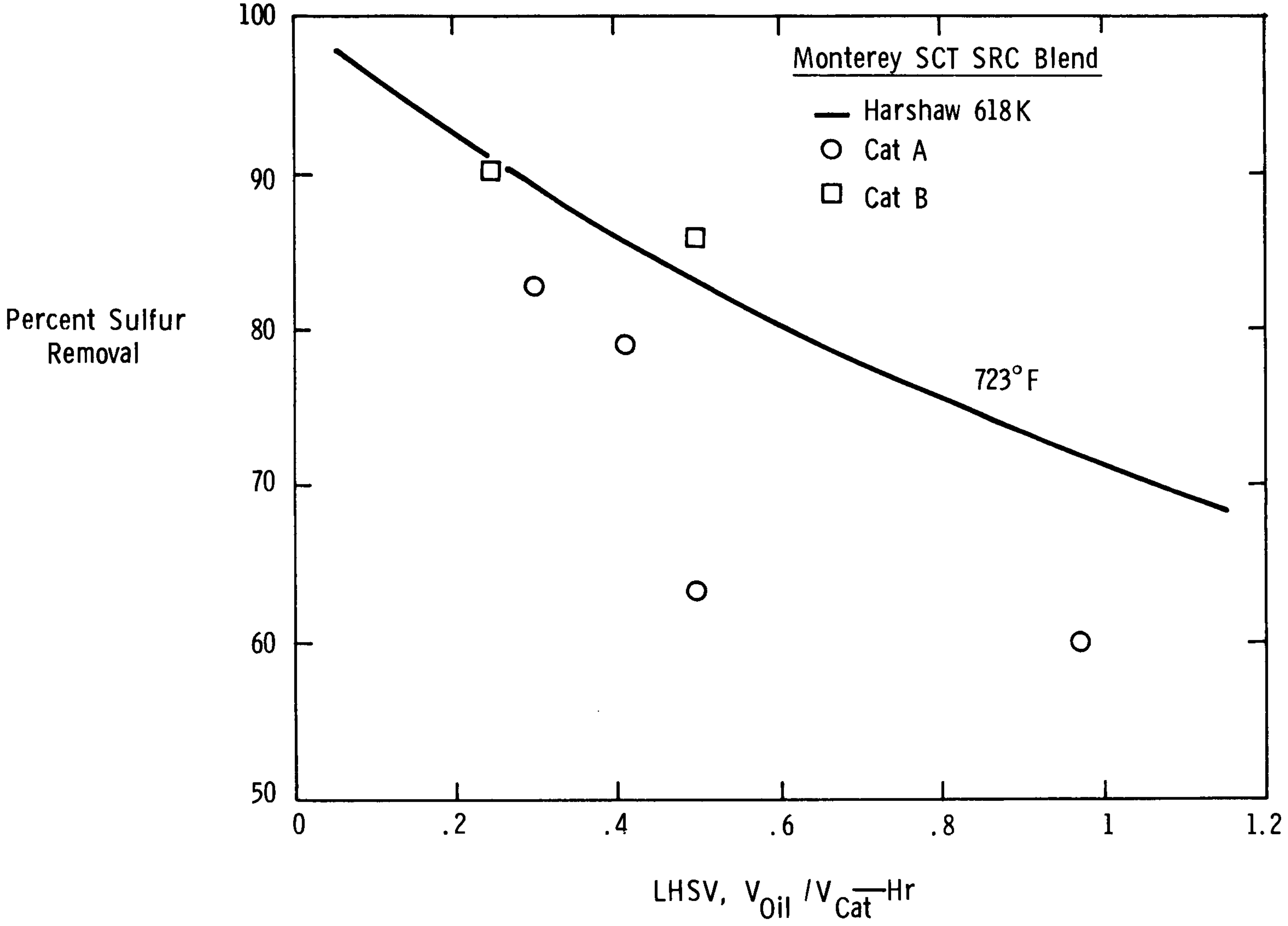

Figure 4-32. Catalyst Comparison for Desulfurization of Monterey SCT SRC Blend

All catalysts are shown to significantly increase the amount of aromatic oils while reducing the polar plus non-eluted asphaltenes of the charge. As would be predicted by our process model for the oils-resins-asphaltenes reaction scheme, presented in Section 4.3, the amount of resins found in the product is of the same magnitude as the charge. On comparing the proprietary catalysts with the Harshaw catalyst, several differences are observed. Coupling the observed higher amounts of resins and polar asphaltenes fractions and the lower aromatic oil fraction with the fact that the product has a significantly lower hydrogen content, suggest that Catalyst A is not as good a hydrogenation catalyst as the Harshaw 618X which is more selective in progressing the reaction, polar asphaltenes ---> resins/eluted asphaltenes ---> oil, along the path at the same severity. Catalyst B, on the other hand, produces more aromatic oils while converting more polar asphaltenes without consuming as much hydrogen (less light saturates are formed). Since its heteroatom removal ability is as good, this would suggest that Mobil B type catalysts may lead to an improved catalyst for hydroprocessing SRC or coal liquid residua materials. Hydroprocessing with Catalyst C resulted in products containing finely dispersed solid suspensions. The product analysis presented here, for the lowest severity case made during the run, shows a significant difference between the filtrate and solid residue portions of the total product -- the filtrate showing properties very similar to the product obtained with the Harshaw catalyst and the solid resin and asphaltene fraction yields identical to the original charge stock. Since two phases result, and the liquid phase is not significantly better than the single product obtained by using the commercial catalyst, it would rule out the use of Catalyst C for the present application.

Additional GEC analyses on products from the Mobil Catalyst "B" study were determined. These results are shown in Table 4-55 along with comparison runs from the Harshaw 618X catalyst study. Although the severities of operation cannot be matched exactly, these data tend to confirm the findings that the Mobil catalyst has a greater reactivity toward converting the asphaltene fraction than the commercial Harshaw catalyst. Comparing the Mobil B catalyst with the Harshaw 618X shows that the polar and non-eluted asphaltenes (Cuts 8-13) is lower for the Mobil B catalyst at all three processing conditions. This confirms the advantage for the Mobil catalyst for hydrotreating high resid containing charge stocks.

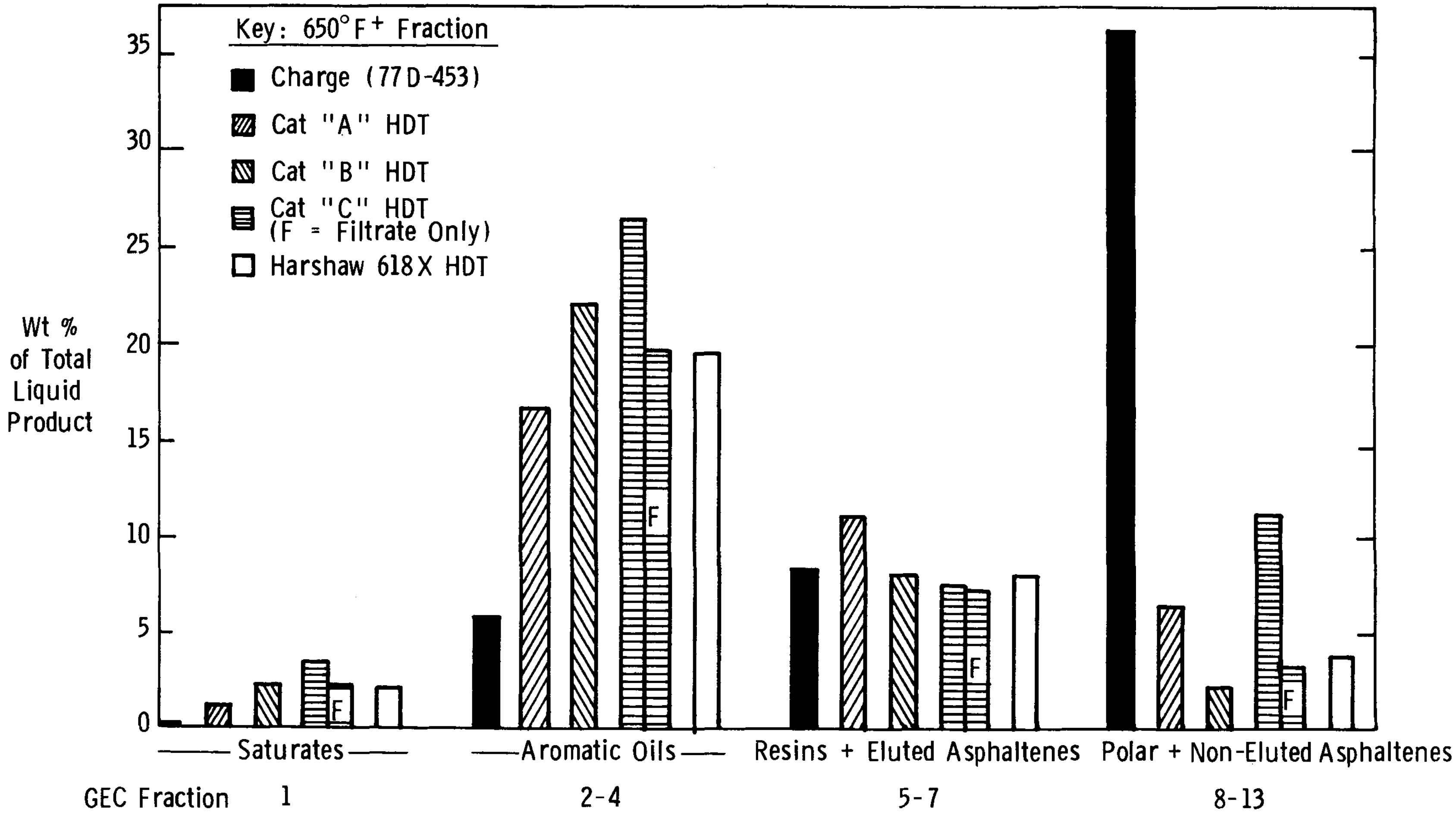

Figure 4-33. Effect of Catalyst on GEC Composition of Hydrotreated SCT Monterey SRC Blend

The elemental analyses of the GEC fractions presented in Table 4-55 for balances from each catalyst study are shown in Table 4-56. The properties for the product from the Catalyst "C" study were calculated from the filtrate and solids analyses. It can be seen that the Harshaw 618X catalyst yielded the best 650°F$^-$ fraction in terms of hydrogenation and heteroatom removal. Mobil Catalyst "B", however, is seen to have done a better overall job of removing heteroatoms from the 650°F$^+$ fraction and the individual GEC cuts (oils, resins, and asphaltenes), with the lowest aromatic saturation. Coupled with the yield data from Table 44-18, the Mobil Catalyst "B" appears to be a better catalyst for upgrading the resid components in the SCT SRC blend. Catalyst "A" shows an equivalent nitrogen removal ability as the Harshaw catalyst, but does not do as well with oxygen and sulfur removal. Catalyst "C", producing a product containing 24 wt % solids, is the lowest activity catalyst for overall heteroatoms removal. A comparison of this solid phase with an H-Coal incompatibility sludge is presented in the next section.

4.5 H-COAL FUEL OIL

Secondary consideration has been given to residual coal liquids from the H-Coal process due to the limited number and quantity of samples made available to us for hydroprocessing studies. Last year's annual (3.1) presented the analyses of the H-Coal products received from Hydrocarbon Research, Inc. (HRI). The H-Coal fuel oil overflow product from HRI's anti-solvent solid separation Run 212-20 was found to consist of two phases at ambient temperatures. Heating with agitation to ∿250°F failed to solubilize the two-phase product. To remove any contamination from the antisolvent used in the preparation, distillations to remove up to a 15% overhead were performed. Heating the bottoms product to 300°F for periods up to 65 hours failed to eliminate the two phases. Due to this inherent incompatibility, this H-Coal product was not used for hydroprocessing.

Four gallons of a homogeneous H-Coal 400°F$^+$ fuel oil were obtained from HRI. This material was originally prepared for the ECAS study in solid separation Run 212-15. It is somewhat lighter and contains less residual oil than the nominal 400°F$^+$ H-Coal fuel oil prepared at a space velocity of 78. Middle distillate was recirculated in the PDU hydroclone system to keep the small line clean during the preparation of this sample. Therefore, it is equivalent to a 400°F$^+$ fuel oil prepared at a space velocity of 50-60, rather than the required 78 space velocity. Complete chemical and physical properties of the as-received

Table 4-55

COMPARISON OF HYDROPROCESSING OF 33% MONTEREY SCT SRC BLEND OVER HARSHAW 618X AND MOBIL B CATALYST

	77D453	HARSHAW 618X			MOBIL CATALYST "B"		
	CHARGE	MB-820	MB-821	MB-825	MB-845	MB-847	MB-846
Process Conditions							
Temperature, °F	--	782	769	725	778	777	726
LHSV, V/Hr/V	--	0.57	0.32	0.31	0.50	0.18	0.24
Pressure, psig	--	2000	2000	2000	2000	2000	2000
H_2 Consumed, scf/B	--	3198	2940	2693	2980	3343	2435
Days On-Stream	--	3.2	4.9	9.8	2.6	5.5	4.1
Total Liquid Product							
Hydrogen, Wt %	6.51	9.98	9.34	9.62	9.76	10.07	9.15
Oxygen, Wt %	4.5	0.8	1.25	1.6	0.8	0.5	1.2
Nitrogen, Wt %	1.14	0.30	0.44	0.37	0.32	0.18	0.45
Sulfur, Wt %	1.01	0.07	0.15	0.101	0.06	0.09	0.10
650°F$^-$, Wt %	49.04	66.19	58.18	62.93	64.96	72.25	63.25
650°F$^+$ Residue							
Cut 1 (Saturates)	0.26	2.09	1.69	1.40	2.41	2.63	2.49
Cut 2-4 (Arom. Oils)	5.45	19.73	21.42	17.31	22.05	21.13	20.00
Cut 5-7 (Resins/Asphalt.)	8.42	8.15	11.60	11.17	8.28	2.93	10.45
Cut 8-13 (Polar Asphalt.)	36.33	3.84	7.11	7.19	2.30	1.03	3.81

Table 4-56

EFFECT OF CATALYST ON THE HYDROPROCESSING OF 33% MONTEREY SCT SRC BLEND

	77D453	HARSHAW 618X	CAT A	CAT B	CAT C		
	CHARGE	MB-820	MB-837	MB-845	MB-848 TOTAL	FILTRATE	SOLIDS
Process Conditions							
Temperature, °F	--	782	779	778	775		
LHSV, V/Hr/V	--	0.57	0.52	0.50	0.45		
Pressure, psig	--	2000	2000	2000	2000		
H_2 Cons., scf/B	--	3198	2477	2980	3049		
IBP-650°F C_6^+							
Hydrogen, Wt %	7.39	10.22	9.25	9.87	--	9.96	--
Oxygen, Wt % (by diff.)	2.9	0.4	0.58	0.6	--	0.5	--
Nitrogen, Wt %	0.51	0.12	0.15	0.16	--	0.10	--
Sulfur, Wt %	0.31	0.015	0.015	0.014	--	0.008	--
650°F$^+$ Residue, Wt %	50.96	33.81	36.70	35.04	49.01	32.91	(Total)
Hydrogen, Wt %							
650°F$^+$ Residue	5.86	8.03	7.39	7.83	7.82	7.92	7.71
Cut 1	(a)	12.64	13.44	12.85	12.87	12.59	13.15
Cut 2-4	6.54	7.92	7.88	8.23		8.13	7.73
Cut 5-7	6.67	7.04	7.19	6.86	6.95	6.90	7.08
Cut 8-12	5.83	6.11	6.17	5.57	5.86	6.09	5.79
Oxygen, Wt %							
650°F$^+$ Residue	6.0	1.6	2.0	1.1	1.9	1.4	2.5
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	1.0	0.7	0.8	0.5	0.8	1.0	0.5
Cut 5-7	5.0	2.2	2.8	2.0	2.9	2.7	3.0
Cut 8-12	7.5	4.6	4.6	3.3	5.3	5.2	5.3
Nitrogen, Wt %							
650°F$^+$ Residue	1.66	0.94	0.96	0.67	0.81	0.66	0.96
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	0.44	0.24	0.22	0.23	0.34	0.37	0.30
Cut 5-7	2.39	1.89	1.92	2.09	1.98	1.92	2.15
Cut 8-12	1.77	1.37	1.33	1.86	1.91	1.47	2.04
Sulfur, Wt %							
650°F$^+$ Residue	1.68	0.18	0.36	0.15	0.80	0.20	1.42
Cut 1	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Cut 2-4	(a)	0.15	0.18	0.12	0.16	0.06	0.30
Cut 5-7	1.38	0.07	0.27	<0.03	(a)	0.18	(a)
Cut 8-12	1.86	(a)	(a)	(a)	(a)	(a)	1.18

(a) Analysis not determined.

material are given in Table 4-57, together with an earlier analysis by HRI. The gravity and distillation show the material to be virtually unchanged from the original analysis. Approximately 20% of the liquid boils above 975°F. It is particularly high in titanium (146 ppm) and iron (19 ppm). H-Coal 400°F$^+$ fuel oil is unlike any previously charged coal liquid, having properties which are generally intermediate between H-Coal distillate and SRC recycle solvent, although its boiling range and CCR content are more nearer those of 2/1 blends of recycle solvent/SRC, as shown below:

	H-Coal 400°F$^+$ Fuel Oil	H-Coal Distillate (Fuel Oil Mode)	SRC Recycle Solvent	2/1 Blend Rec Sol/SRC
Properties				
Gravity, °API	4.5	18.6	5.5	-3.0
Hydrogen, wt %	8.49	10.14	7.71	6.84
Oxygen, wt %	1.70	1.20	3.77	3.91
Nitrogen, wt %	0.84	0.38	0.62	1.03
Sulfur, wt %	0.23	0.11	0.40	0.41
CCR, wt %	10.81	0.28	0.51	16.49
Distillation, °F (D-2887)				
IBP	346	271	301	337
5%	398	333	336	390
10%	434	349	360	403
30%	527	397	400	475
50%	618	441	441	584
70%	741	498	509	765
90%	-	626	614	-
95%	-	697	653	-
EP	-	885	830	-

The as-received 400°F$^+$ H-Coal fuel oil was hydroprocessed over the small pore HDS-1441A (CoMo) catalyst. Only six material balances were completed during this program due to the limited amount of charge stock available (< 5 gallons). Some thermal instability of the H-Coal charge was evidenced by a gummy, black sediment in the charge reservoir after completion of the seven-day run. Hydroprocessing

Table 4-57

ANALYSES OF H-COAL 400°F$^+$ FUEL OIL
(ECAS SAMPLE, RUN 212-15 OVERFLOW PRODUCT)

PROPERTIES	MOBIL	HRI(1)
Gravity, °API	4.5	3.2
Flash Point, °F	--	210
CCR, Wt %	10.8	--
Kinematic Viscosity, cs @ 100°F	226.5	--
Elemental Analysis, Wt %		
Hydrogen	8.49	--
Sulfur	0.23	--
Nitrogen	0.84	--
Oxygen	1.70	--
Ash	--	0.014
Aromatic Carbon, %	58	--
Trace Metals, ppmw		
Pb	1.0	--
Fe	19	--
Ti	146	--
Ca	1.3	--
Na	0.92	--
K	0.66	--
V	0.6	--
Distillation, °F @ %	(Sim. Dist)	(Vacuum)
IBP	346	355
5	398	408
10	434	427
30	527	516
50	618	633
70	741	818
		986 @ 83%

---

(1) Original inspection of blend for ECAS (letter from P. H. Kydd, December 13, 1976).

Table 4-58

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: H-COAL FUEL OIL (400+°F) (J-8723)
CATALYST: HDS-1441A (J-7278)
PROGRAM: 2469 UNIT: CT-146

	CHARGE	*MB-812*	*MB-813*	*MB-814*	*MB-815*	*MB-816*	*MB-817*
OPERATING CONDITIONS							
TEMPERATURE, °F	-	674	729	779	675	724	775
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
LHSV	-	1.00	1.01	1.00	0.36	0.35	0.36
DAYS ON STREAM	-	0.7	1.4	2.0	3.3	5.3	6.8
LIQUID PRODUCT PROPERTIES							
GRAVITY, °API	4.5	6.0	8.8	17.4	8.7	10.7	14.4
HYDROGEN, WT °/°	8.49	8.97	9.43	9.76	9.27	10.01	10.16
SULFUR, WT °/°	0.230	0.113	0.064	0.034	0.095	0.052	0.023
NITROGEN, WT °/°	0.840	0.680	0.510	0.380	0.590	0.360	0.180
OXYGEN, WT °/°	1.7	0.7	0.5	0.1	0.7	0.7	0.1
CCR, WT °/°	10.81	9.91	6.77	6.28	9.07	5.55	3.62
K.V. AT 40°C	226.50	46.66	22.44	12.14	37.84	15.83	8.55
AROMATIC CARBON, °/°	58	53	50	45	-	-	39
HETEROATOM REMOVAL,PCT							
SULFUR	-	51.5	72.6	85.5	59.1	77.8	91.2
OXYGEN	-	59.3	71.1	94.2	59.2	59.6	94.8
NITROGEN	-	20.0	40.2	55.6	30.4	58.0	81.2
HYDROGEN UTILIZATION,SCF/B							
H2 CONSUMED BY C1-C5	-	36	84	78	36	200	1120
H2 CONSUMED BY C6+	-	256	558	764	472	941	576
H2 CONSUMED BY S	-	5	7	8	6	8	9
H2 CONSUMED BY N	-	25	50	69	38	72	101
H2 CONSUMED BY O	-	87	104	139	87	88	139
H2 CONSUMED TOTAL	-	409	803	1058	638	1308	1946

severities ranged from 0.35-1.0 LHSV and 675-780°F at a unit pressure of 2000 psig. The operating conditions, hydrogen utilization and liquid product properties, are given in Table 4-58, together with the corresponding charge stock properties. The product yields on charge and the distillations of the liquid products are given in Appendix Table B-17.

Hydrogen consumptions ranged from 400 to 1950 scf/B producing liquid products containing up to 10.16 wt % hydrogen. The most severe run (775°F, 0.36 LHSV at 2000 psi) produced a product containing <0.1 wt % oxygen, 0.02 wt % sulfur, and 0.18 wt % nitrogen.

To achieve a given product hydrogen level, the required hydrogen consumption for H-Coal fuel oil is intermediate between that required for H-Coal distillate and SRC recycle solvent. The hydrogen consumption required to obtain a given product nitrogen content is greater for H-Coal fuel oil than either of the other two coal liquids. For a given hydrogen content, the H-Coal fuel oil products tend to have less percent aromatic carbon than any other coal liquid.

The degree of demetalation of the H-Coal fuel oil accomplished at various processing severities is indicated by the trace metals analyses presented below in Table 4-59.

Table 4-59

TRACE METALS ANALYSIS IN HYDROPROCESSED COAL LIQUIDS

CHARGE STOCK (CATALYST)	SEVERITY HYDROGEN CONSUMPTION (SCF/BBL)	METALS, PPM V	Fe	Ca	Na	K	Total+
H-Coal 400°F+ Fuel Oil (HDS-1441A)							
CHG	-	.6	19	1.3	.9	.7	22.5
MB-812	409	.4	(15)	1.9	.7	.1	18.1
MB-813	803	.1	3	1.4	.7	.1	5.3
MB-814	1058	<.1	4.7	.5	.6	.1	5.9
MB-817	1946	<.1	.7	.6	.8	.1	2.2

The metals in this liquid were all decreased to low levels. However, the alkali metals may still be a problem for gas turbines. The metals have presumably laid down on the catalyst and will contribute to its deactivation.

One property that has been used to evaluate coal liquids is the fuel compatibility test with petroleum oils. In this test, 50/50 blends of coal and petroleum liquids are stirred for six hours at 150°F, then centrifuged at 150°F, and the volume percent sediment determined as an index of incompatibility. Solvent Refined Coal is extremely incompatible with both No. 2 and No. 6 fuel oils, but this incompatibility decreases as the SRC is hydroprocessed and its asphaltene content is reduced. Sometimes, however, a minimum has been observed, after which the amount of incompatible sediment increased with further increase in hydroprocessing severity. In order to understand this phenomenon, the compatibility of the H-Coal fuel oil and three hydroprocessed samples with petroleum No. 6 fuel oil were determined, and the sediment from one of the resulting sediments was analyzed.

The percent of incompatibility sediment obtained from each test are shown below in Table 4-60.

Table 4-60

COMPATIBILITY OF RAW AND HYDROPROCESSED H-COAL $400°F^+$ FUEL OIL WITH PETROLEUM NO. 6 FUEL OIL

50/50 WT BLEND OF NO. 6 F.O. WITH:	INCOMPATIBILITY SEDIMENT, VOL %
Raw H-Coal $400°F^+$ (J8723)	100
Mild HDP H-Coal $400°F^+$ (146-2469-812-1)	6.0
Moderate HDP H-Coal $400°F^+$ (146-2469-814-1)	Trace
Severe HDP H-Coal $400°F^+$ (146-2469-817-1)	Trace

The raw H-Coal $400°F^+$ material was completely incompatible, forming a solid which could not be poured when the 50/50 blend was stirred at 150°F. Mild hydroprocessing reduced the incompatibility sediment to 6.0 vol % while the moderate and severe hydroprocessed materials formed only trace (<0.05 vol %) amounts of sediment with No. 6 fuel oil. Thus, as a minimum, moderate hydroprocessing conditions would be required to provide a product with some degree of compatibility with petroleum fuels.

The sediment obtained from the mild hydroprocessing condition (i.e., 6 vol % of the mixture) is a very soft, black grease at room temperature. GEC and mass spectrometer type analyses of the sediment and the hydroprocessed H-Coal and petroleum No. 6 fuel oils are shown in Table 4-61. As expected, the sediment shows higher amounts of resins, asphaltenes, and polar asphaltenes than the components of the blend. However, it is surprising that over half of the sediment is made up of saturates and aromatic oils, and over a third of the saturates are paraffins much like the paraffins in the No. 6 fuel oil. Analyses for oxygen, nitrogen, and sulfur compounds (Table 4-62) indicate that these materials concentrate preferentially in the sediment.

This incompatibility may be the result of mixing high and low aromaticity oils (solvents) to give one of intermediate aromaticity which is incapable of dissolving either the highly polar asphaltenes or the waxy paraffins. Aromatic oils and resins, highly attractive to both the fuel oil and sediment phases, would be partitioned between the two phases. They may also be retained in the sediment as entrapped liquid in the micelles.

During a previously discussed hydroprocessing study with short contact time SRC, a significant amount of fine solids slurried with the liquid product were formed. The elemental and GEC analyses of the filtered solids and liquid phase from one of the balances obtained at moderately severe hydroprocessing are compared with the H-Coal fuel oil and its incompatible sludge in Table 4-63. The solid phase obtained by hydroprocessing has a lower hydrogen and a great deal higher heteroatom concentration than does the liquid phase. This relationship is similar to the one seen between an H-Coal fuel oil and the incompatible sludge formed upon mixing with a No. 6 fuel oil. In addition, both of the solid phases are surprisingly high in saturates and aromatic oils, but low in resins/asphaltenes (Cuts 5-7). Further, these saturated and aromatic oil cuts are fluid oils at room temperature, whereas the corresponding cuts from the $650°F^+$ residue are solids.

Table 4-61

ANALYSES OF NO. 6 FUEL OIL, A HYDROPROCESSED H-COAL LIQUID AND THEIR INCOMPATIBLE SEDIMENT

CUT NO.	GEC FRACTION	EL PALITO NO. 6 FUEL OIL	CT 146-2469-812 (H-COAL FUEL OIL)	INCOMPATIBLE SEDIMENT*
		Wt %	Wt %	Wt %
1	Saturates	30.48	12.22	16.85
2-4	Aromatic Oils	49.57	56.18	40.57
5-7	Resins/Asphaltenes	14.48	16.80	18.21
8-13	Polar Asphaltenes	5.47	14.80	24.37
	Total	100.00	100.00	100.00
Mass Spec. Type Analysis				
Saturates (Cut 1)				
	Paraffins, % w/w	38.7	28.0	36.0
	Mono-Naphthenes	30.4	30.3	26.8
	Poly-Naphthenes	30.1	39.4	35.5
	Mono-Aromatics	0.8	2.3	1.8
		100.0	100.0	100.1
Paraffin Avg. Mol Wt		415	304	392
Aromatic Oils (Cuts 2-4)				
	Mono-aromatics	38.4	40.6	36.5
	Di-Aromatics	27.6	32.2	37.1
	Tri-Aromatics	10.9	13.1	14.0
	Tetra-Aromatics	5.3	8.3	4.9
	Penta-Aromatics	4.5	3.4	2.7
	Arom. Sulfur Types	9.2	2.5	4.8
	Unidentified Arom.	4.1	0.0	0.0
		100.0	100.1	100.0

---

*Sediment-centrifuged from a 50/50 w/w mixture at 150°F (M 1006).

Table 4-62

ELEMENTAL ANALYSES OF GEC FRACTIONS FROM NO. 6 FUEL OIL, A HYDROPROCESSED H-COAL FUEL OIL AND THEIR INCOMPATIBLE SEDIMENT

	EL PALITO NO. 6 FUEL OIL Wt %	CT 146-2469-812 (H-COAL FUEL OIL) Wt %	INCOMPATIBLE SEDIMENT Wt %
As Received			
Oxygen	0.30	0.70	1.51
Nitrogen	0.41	0.68	0.77
Sulfur	1.96	0.11	1.30
GEC Fractions			
Cut 2-4 (Arom. oils)			
Oxygen	0.21	0.6	0.8
Nitrogen	0.18	0.19	0.19
Sulfur	2.74	0.02	1.26
Cut 5-7 (Resins/asph.)			
Oxygen	1.35	3.9	3.6
Nitrogen	1.48	2.65	1.85
Sulfur	2.64	<0.2	2.15
Cut 8-12 (Polar asph.)			
Oxygen	2.54	6.4	5.7
Nitrogen	(a)	1.68	1.81
Sulfur	(a)	0.48	1.55

(a) Analysis not determined.

Table 4-63

ANALYSES OF INCOMPATIBLE SOLIDS FROM COAL LIQUIDS

		CT146-2528-848		CT146-2469-812	INCOMPATIBLE*
		SCT MONTEREY SRC BLEND		H-COAL	SLUDGE
		LIQUID PHASE	SOLID PHASE	FUEL OIL	
	Hydrogen, Wt %	10.08	7.71	8.97	(a)
	Oxygen, Wt %	0.8	2.5	0.7	1.5
	Nitrogen, Wt %	0.29	0.96	0.68	0.77
	Sulfur, Wt %	0.07	1.42	0.11	1.30
Cut No.	GEC Fraction, Wt %	650°F+ Resid	As Rec'd.	650°F+ Resid	As Rec'd.
1	Saturates	6.78	7.92	7.34	16.85
2-4	Aromatic Oils	60.34	47.41	47.92	40.57
5-7	Resins/Asphaltenes	22.49	8.44	26.32	18.21
8-13	Polar Asph./Non-Eluted	10.39	36.23	18.42	24.37
	Total	100.00	100.00	100.00	100.00

---

*Sediment centrifuged from a 50-50 w/w mixture of H-Coal fuel oil and El Palito No. 6 F.O. at 150°F.

(a) Analysis not determined.

Section 5

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APPENDIX A

FINAL REPORT

June 1, 1976-December 31, 1977

AN INVESTIGATION OF THE UTILIZATION OF COAL-DERIVED LIQUID FUELS IN A COMBUSTION TURBINE ENGINE

Prepared Under Contract

RP 361-2

for

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Electric Power Research Institute

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Section A.6

CONCLUSIONS AND RECOMMENDATIONS

It can be generally concluded from this series of tests that coal liquid fuels could be utilized in current combustion turbines provided an allowance for increased heat transfer to the combustor wall is made. Only the most severely hydrotreated H-Coal fuel showed no significant effect on combustor wall temperature. On the other hand, no fundamental barrier to the use of even the lowest quality coal liquid has been identified suggesting that even the recycle solvent could be a satisfactory gas turbine fuel if combustor modification, and possibly, more liberal emission standards and lower turbine inlet temperatures, can be allowed. The cost of upgrading coal liquids to meet current gas turbine requirements would be a substantial and continuing cost. The cost of redesigning the gas turbine to accommodate raw coal liquids would also be quite large and the probability of getting the emission regulations lowered small. The optimum approach probably lies somewhere between these two extremes including both fuel upgrading and gas turbine component redesign.

Specific conclusions based on the results of this work are:

- NO_x emissions were over 100% higher for the raw SRC recycle solvent (0.62 wt. % fuel bound nitrogen) compared to the base petroleum No. 2 fuel (0.008 wt. % fuel bound nitrogen). Hydroprocessing both the H-Coal and SRC recycle solvent decreased the fuel bound nitrogen content producing coal-derived fuels with NO_x emissions equivalent to or less than the base petroleum No. 2 fuel.

- Coal liquid fuels over the range tested can be readily forwarded and atomized.

- Based on calculations the lighter fuel fractions may perturb the spray, producing fuel rich pockets conducive to coke formation.

- Corrosive, erosive and deposit forming contaminants in the raw coal liquids are present in sufficient concentrations to require further turbine tolerance studies.

- Coke formation on the nozzle and in the combustor is a major concern with the raw and least severely hydroprocessed fuels.

- CO emissions are acceptable.

- Unburned hydrocarbons are acceptably low, increasing slightly with increasing fuel hydrogen.
- The smoke production was also low showing no relationship to fuel hydrogen.
- The combustor wall temperature was higher when burning coal liquids than for the base line #2 distillate test except for H-Coal fuel sample J8830 (11.66 wt. % H). Combustor wall temperatures increased with decreasing fuel hydrogen content.
- Flame temperatures were not significantly different for any of the fuels as determined by optical pyrometry.
- H-Coal fuels performed better than fuels derived from SRC recycle solvent.

The results to date have been encouraging and have helped to identify coke formations, combustor cooling and turbine tolerance as design areas needing further work.

In order to establish the most effective program for utilization of coal liquids, consideration should be given to fuel quality, combustion processes and the tolerance of turbine hot parts to the resultant operating environment. Those considerations which should be included in a balanced program are:

1. Fuels - additional characterization of the fuels is required to determine the auto ignition point as a function of temperature, pressure, and fuel/air ratio.

The ignition delay of the coal liquids at gas turbine operating conditions must be established to permit combustor development work.

Redefinition of the distillation range of interest in gas turbine applications is required to minimize highly volatile and heavy fractions.

Further evaluation of chemical and physical methods of upgrading fuel and the costs associated with this upgrading are needed.

Consideration should be given to blending coal liquids with lower cost, more available fuels in order to produce a suitable gas turbine fuel.

2. Combustion - improvements in the performance of existing can type combustors is possible through redesign of nozzles, sweep air flows, primary air distribution

wall cooling and primary zone residence time. These improvements should be investigated in parallel with the fuel studies.

Advanced combustor concepts should be developed to permit the utilization of the lowest quality fuel possible. Specific concepts which merit further evaluation include:

- hot wall combustors
- prevaporized/premixed combustors
- staged combustion
- external fluid bed combustors

3. Turbine Protection - evaluation of the alloys and coatings being used in gas turbines should be carried out in a turbine expansion gas produced through the combustion of coal liquids in order to establish the tolerance of the turbine to the fuel contaminants and to develop methods of increasing this tolerance. Such an experimental evaluation of corrosion should be supported by an analysis of the corrosive mechanisms acting and projection of the turbine tolerance as a function of pressure, temperature, contaminant level and turbine alloy.

The tendency of fuel contaminants to deposit on turbine hot parts or to erode these parts must also be investigated. The use of existing models to predict erosion and deposition coupled with bench scale determination of the erosive characteristics of the fuel ash and cascade tests of air foils under actual gas turbine operating conditions can define the tolerance of the combustion turbine to erosion and deposition.

Methods for controlling damage due to particulates also needs to be studied. Included should be hot gas clean-up, air foil design and deposit removal techniques.

It is necessary to have parallel and carefully integrated programs in the areas of fuels, combustion and turbine protection in order to effect the proper compromises which will inevitably be required to use coal-derived fuels efficiently and reliably in combustion turbines.

Section A.7

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APPENDIX B

Material Balances for Hydroprocessing Studies

TABLE B-1

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: WYODAK WASH SOLVENT (77D-895)
CATALYST: HDS-1441 (J-7322)
PROGRAM: 2569 UNIT: CT-199

	CHARGE	MB-715	MB-716	MB-717	MB-718	MB-719	MB-720	MB-721
MATERIAL BALANCE, PCT WT	-	103.6	103.7	103.3	99.6	100.4	102.4	100.3
<u>OPERATING CONDITIONS</u>								
TEMPERATURE, °F	-	720	670	767	763	727	677	676
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	1000
LHSV	-	0.58	0.52	0.51	0.94	1.01	1.00	0.95
H2 CIRCULATION, SCF/B	-	7431	8827	8149	8091	7951	7686	6949
DAYS ON STREAM	-	1.8	2.8	3.8	4.6	5.1	5.6	6.4
<u>YIELDS, WT</u> °/°								
C1-C3	-	0.49	0.11	0.74	0.50	0.26	0.08	0.07
C4	-	0.44	0.07	0.23	0.18	0.31	0.08	0.07
C5	-	0.16	0.09	0.20	0.16	0.19	0.10	0.12
C6+	100.00	96.26	96.50	96.44	95.96	96.37	96.48	96.29
H2S	-	0.12	0.15	0.16	0.14	0.15	0.15	0.14
NH3	-	0.43	0.42	0.43	0.43	0.43	0.40	0.30
H2O	-	6.53	6.53	6.53	6.53	6.53	6.53	5.99
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)								
IBP	321	179	180	178	178	178	178	178
5 °/°	374	184	184	183	184	183	182	184
10	387	216	216	215	215	215	214	215
30	458	282	283	279	282	281	283	303
50	501	364	368	361	364	362	376	395
70	570	401	405	389	402	402	417	422
90	649	443	450	437	445	447	454	457
95	665	460	465	456	460	461	468	472
EP	841	517	513	512	513	507	509	510

TABLE B-2

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: SRC RECYCLE SOLVENT (75D-3019) W/H2O ADDITION
CATALYST: HDS-1441A (J-7278)
PROGRAM: 2335   UNIT: CT-146

	CHARGE	MB-753	MB-754	MB-755	MB-756	MB-757
MATERIAL BALANCE, PCT WT	-	100.3	98.5	96.9	98.3	91.3
OPERATING CONDITIONS						
TEMPERATURE, °F	-	676	675	675	672	675
PRESSURE, PSIG	-	2500	2500	2500	2500	2500
LHSV	-	1.04	1.02	1.00	1.00	0.98
H2 CIRCULATION, SCF/B	-	4854	4870	4876	4746	5163
DAYS ON STREAM	-	1.4	2.6	3.6	4.6	5.9
YIELDS, WT °/°						
C_1-C_3	-	0.07	0.13	0.11	0.12	0.09
C_4	-	0.02	0.04	0.04	0.05	0.02
C_5	-	0.03	0.04	0.04	0.04	0.03
C_6+	100.00	98.25	98.57	98.56	98.22	98.56
H_2S	-	0.39	0.40	0.40	0.39	0.39
NH_3	-	0.19	0.26	0.30	0.31	0.17
H_2O	-	2.73	2.17	2.17	2.29	2.28
DISTILLATION, °F (D-2887)						
IBP	301	-	-	-	-	-
5 °/°	336	-	-	-	-	-
10	360	-	-	-	-	-
30	400	-	-	-	-	-
50	441	-	-	-	-	-
70	509	-	-	-	-	-
90	614	-	-	-	-	-
95	653	-	-	-	-	-
EP	830	-	-	-	-	-

TABLE B-3

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: SRC RECYCLE SOLVENT (76D-3007) W/H2O ADDITION
CATALYST:HDS-1441A (J-7278)
PROGRAM: 2562 UNIT: CT-146

	CHARGE	MB-860	MB-861	MB-862	MB-863
MATERIAL BALANCE, PCT WT	-	102.4	92.6	91.8	95.0
<u>OPERATING CONDITIONS</u>					
TEMPERATURE, °F	-	722	721	715	727
PRESSURE, PSIG	-	2500	2500	2500	2500
LHSV	-	1.04	1.00	1.00	0.97
H2 CIRCULATION, SCF/B	-	6958	6009	7036	8117
DAYS ON STREAM	-	2.2	3.7	4.4	5.2
<u>YIELDS, WT</u> °/°					
C1-C3	-	0.17	0.17	0.13	0.16
C4	-	0.21	0.05	0.05	0.05
C5	-	0.07	0.03	0.04	0.03
C6+	100.00	99.47	99.33	99.36	99.74
H2S	-	0.31	0.30	0.28	0.28
NH3	-	0.56	0.51	0.51	0.52
H2O	-	2.37	2.14	2.03	1.69
<u>DISTILLATION</u>, °F (D-2887)					
IBP	263	179	180	180	180
5 °/°	327	213	266	268	272
10	349	311	367	379	377
30	389	421	437	439	439
50	409	477	482	484	483
70	440	540	545	545	545
90	461	589	596	598	596
95	474	626	632	634	632
EP	676	729	732	733	719

TABLE B-4

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: H-COAL DISTILLATE(SYNCRUDE) (76D-3521)
CATALYST: KETJEN 153-S (J-7709)
PROGRAM: 2502 UNTT: CT-199

	CHARGE	MB-641	MB-642	MB-643	MB-644	MB-645	MB-646
MATERIAL BALANCE, PCT WT	-	97.9	98.5	102.4	104.8	96.4	98.0
<u>OPERATING CONDITIONS</u>							
TEMPERATURE, °F	-	701	704	698	700	698	702
PRESSURE, PSIG	-	1500	1500	1500	1500	1500	2500
LHSV	-	1.44	1.43	0.53	0.51	0.48	0.47
H2 CIRCULATION, SCF/B	-	2192	6144	5090	5653	5986	6256
DAYS ON STREAM	-	3.3	5.9	7.9	10.9	17.4	19.4
<u>YIELDS, WT</u> °/o							
C1-C3	-	0.05	0.21	0.22	0.24	0.23	0.36
C4	-	0.05	0.09	0.10	0.11	0.10	0.07
C5	-	0.07	0.05	0.06	0.06	0.06	0.06
C6+	100.00	99.10	98.87	99.30	99.26	99.50	100.15
H2S	-	0.14	0.14	0.14	0.14	0.14	0.13
NH3	-	0.37	0.39	0.45	0.46	0.46	0.46
H2O	-	1.47	1.47	1.58	1.58	1.46	1.58
<u>DISTILLATION</u>, °F (D-2887)							
IBP	270	159	180	182	47	181	182
5 °/o	328	254	268	255	293	250	243
10	346	316	32	320	340	317	312
30	396	383	402	395	398	394	380
50	433	423	443	437	428	436	429
70	489	482	494	486	481	484	479
90	590	578	584	574	617	571	564
95	665	638	641	636	694	633	621
EP	942	855	897	884	1039	899	889

TABLE B-4 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: H-COAL DISTILLATE(SYNCRUDE) (76D-3521)
CATALYST: KETJEN 153-S (J-7709)
PROGRAM: 2502 UNIT: CT-199

	MB-647	MB-648	MB-649	MB-650	MB-651	MB-652	MB-653
MATERIAL BALANCE, PCT WT	97.0	98.6	97.5	95.8	96.1	100.8	93.4
<u>OPERATING CONDITIONS</u>							
TEMPERATURE, •F	702	703	705	717	732	697	695
PRESSURE, PSIG	2500	2500	1500	1500	1500	1500	1500
LHSV	0.50	0.54	1.49	1.51	1.45	2.93	2.93
H2 CIRCULATION, SCF/B	6348	2873	4329	6003	6394	4856	1157
DAYS ON STREAM	22.4	29.5	30.4	30.9	31.4	32.1	33.6
<u>YIELDS, WT</u> •/•							
C1-C3	0.20	0.14	0.19	0.20	0.25	0.04	0.01
C4	0.10	0.13	0.07	0.10	0.11	0.01	0.01
C5	0.06	0.08	0.11	0.13	0.06	0.03	0.02
C6+	100.21	100.01	98.83	99.18	99.20	99.60	99.52
H2S	0.14	0.14	0.14	0.14	0.14	0.13	0.14
NH3	0.46	0.46	0.37	0.38	0.41	0.18	0.15
H2O	1.58	1.58	1.47	1.24	1.35	0.90	0.79
<u>DISTILLATION</u>, •<u>F</u> (<u>D</u>-2887)							
IBP	159	31	183	140	164	164	176
5 •/•	228	65	268	236	244	301	315
10	303	179	334	308	313	342	348
30	380	346	400	392	395	403	402
50	433	410	437	437	440	449	446
70	487	473	489	490	494	504	498
90	566	563	580	583	585	594	587
95	616	613	634	640	641	651	643
EP	826	812	767	830	832	839	841

TABLE B-5

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33°/° BLEND WYODAK SRC/SOLVENT (76D-2917)
CATALYST: HDS-1441A (J-7278)
PROGRAM: 2418 UNIT: CT-146

	CHARGE	MB-772	MB-773	MB-774	MB-775	MB-776	MB-777	MB-778	MB-779	MB-780	MB-781
MATERIAL BALANCE, PCT WT	-	99.4	106.3	100.3	95.3	99.2	99.2	94.9	105.6	93.2	94.3
<u>OPERATING CONDITIONS</u>											
TEMPERATURE, °F	-	676	731	784	772	723	670	671	724	775	770
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.94	0.98	0.97	0.45	0.51	0.51	0.16	0.18	0.29	0.07
H2 CIRCULATION, SCF/B	-	6315	5423	6311	6552	6979	5910	8390	8041	5745	25770
DAYS ON STREAM	-	1.6	2.6	3.6	4.8	6.0	7.2	8.8	10.4	11.9	13.4
<u>YIELDS, WT</u> °/°											
C1-C3	-	0.16	0.40	0.90	1.15	0.41	0.07	0.32	0.67	1.53	4.39
C4	-	0.21	0.24	0.98	0.38	0.31	0.43	0.38	1.10	0.57	2.30
C5	-	0.09	0.11	0.76	0.12	0.29	0.32	0.15	0.90	0.30	0.91
C6+	100.00	98.35	97.72	95.62	97.31	98.57	98.34	98.24	96.53	97.02	92.78
H2S	-	0.25	0.27	0.30	0.29	0.28	0.25	0.29	0.30	0.30	0.30
NH3	-	0.27	0.42	0.72	0.83	0.62	0.26	0.60	0.89	0.79	1.17
H2O	-	1.85	2.41	3.53	3.73	2.95	1.75	2.85	3.85	3.18	4.06
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)											
IBP	334	231	240	198	245	289	220	218	184	183	171
5 °/°	387	371	358	289	328	387	365	318	262	285	218
10	403	403	402	359	381	416	404	385	335	355	273
30	473	468	469	447	454	500	476	458	445	458	435
50	569	559	560	537	537	597	572	542	526	537	499
70	801	696	686	642	644	749	858	709	671	673	572
90	-	-	-	-	-	-	-	-	-	-	765
95	-	-	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-	-	-

TABLE B-6

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33°/° BLEND MONTEREY SRC/SOLVENT (77D-1039)
CATALYST: HARSHAW 618X (J-7242)
PROGRAM: 2501 UNIT: CT-146

	CHARGE	*MB-827*	*MB-828*	*MB-829*	*MB-830*	*MB-831*	*MB-832*	*MB-833*	*MB-834*	*MB-835*
MATERIAL BALANCE, PCT WT	-	103.2	105.3	100.7	100.7	112.8	99.0	103.9	108.3	99.6
<u>OPERATING CONDITIONS</u>										
TEMPERATURE, °F	-	724	777	622	623	724	776	788	775	730
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.47	0.53	0.53	0.25	0.29	0.28	0.95	1.04	0.49
H2 CIRCULATION, SCF/B	-	7556	7361	6915	3617	6807	8035	8359	8401	5844
DAYS ON STREAM	-	1.6	2.6	3.6	4.7	6.3	7.8	8.6	9.1	10.1
<u>YIELDS, WT</u> °/°										
C1-C3	-	0.34	1.67	0.18	0.13	1.45	1.71	1.20	1.11	0.49
C4	-	0.74	0.57	0.04	0.03	0.48	0.61	0.52	0.76	0.29
C5	-	0.25	0.18	0.01	0.04	0.18	0.24	0.17	0.30	0.09
C6+	100.00	97.19	95.75	98.85	98.77	95.86	95.51	95.98	96.25	97.56
H2S	-	0.52	0.58	0.34	0.42	0.58	0.59	0.56	0.54	0.52
NH3	-	0.80	0.99	0.28	0.65	0.99	1.16	0.84	0.70	0.71
H2O	-	2.76	3.09	0.83	0.94	3.41	3.74	3.20	2.44	2.30
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)										
IBP	351	258	258	261	261	258	140	257	183	257
5 °/°	397	305	306	400	393	331	234	306	332	340
10	418	351	363	408	413	369	325	354	381	390
30	483	424	437	446	478	434	437	436	436	435
50	591	477	479	581	557	476	502	482	480	472
70	-	600	575	-	718	587	592	603	680	602
90	-	-	-	-	-	-	-	-	-	830
95	-	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-	-

TABLE B-7

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 70°/° BLEND MONTEREY SRC/SOLVENT (J-8864)
CATALY: HARSHAW 618X (J-7242)
PROGRAM: 2539   UNIT: CT-146

	CHARGE	MB-852	MB-853	MB-854	MB-855	MB-856	MB-857	MB-858	MB-859
MATERIAL BALANCE, PCT WT	-	98.2	101.8	97.1	106.2	106.6	106.3	100.7	100.0
<u>OPERATING CONDITIONS</u>									
TEMPERATURE, °F	-	726	768	772	723	720	772	781	779
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.44	1.17	0.20	0.30	0.14	0.13	0.41	0.42
H2 CIRCULATION, SCF/B	-	7017	3250	10521	7817	15199	15486	7412	9186
DAYS ON STREAM	-	2.2	3.2	4.5	6.0	8.5	11.5	13.6	14.4
<u>YIELDS, WT</u> °/°									
C1-C3	-	1.04	0.81	2.20	0.96	3.04	3.60	1.72	1.69
C4	-	0.31	0.19	0.98	0.64	2.51	1.61	0.58	1.40
C5	-	0.02	0.06	0.42	0.60	1.85	0.66	0.23	1.03
C6+	100.00	96.30	95.71	93.40	95.65	90.47	91.30	94.74	93.36
H2S	-	0.63	0.72	0.93	0.83	0.88	0.94	0.86	0.86
NH3	-	0.90	1.17	1.51	1.05	1.44	1.73	1.10	1.08
H2O	-	3.40	3.90	4.99	3.74	4.64	5.22	4.09	4.20
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)									
IBP	-	363	260	180	307	234	200	233	331
5 °/°	-	416	358	330	423	416	368	412	419
10	-	454	416	395	446	442	416	444	440
30	-	573	484	483	564	559	514	572	555
50	-	738	591	587	694	674	610	703	651
70	-	-	811	754	-	909	732	-	806
90	-	-	-	-	-	-	-	-	-
95	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-

TABLE B-8

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGES: 70,80,AND 90°/° BLENDS MONTEREY SRC/SOLVENT
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2593 UNIT: CT-146

	J-8977	MB-864	MB-865	J-8978	MB-866	MB-867	MB-868	J-8979	MB-869
°/° SRC	70			80				90	
MATERIAL BALANCE, PCT WT	-	104.6	116.3	-	99.8	102.6	97.5	-	104.7
OPERATING CONDITIONS									
TEMPERATURE, °F	-	723	792	-	719	781	780	-	781
PRESSURE, PSIG	-	2000	2000	-	2000	2000	2000	-	2000
LHSV	-	0.21	0.32	-	0.28	0.22	0.41	-	0.38
H2 CIRCULATION, SCF/B	-	12042	7905	-	7289	11180	12445	-	14515
DAYS ON STREAM	-	1.9	2.7	-	3.6	4.6	5.2	-	5.9
YIELDS, WT °/°									
C1-C3	-	0.89	1.62	-	0.52	5.13	3.53	-	3.59
C4	-	0.79	0.70	-	0.37	2.27	1.37	-	4.31
C5	-	0.10	0.28	-	0.06	0.71	0.36	-	2.70
C6+	100.00	96.10	94.61	100.00	96.28	89.03	91.75	100.00	88.00
H2S	-	0.70	0.81	-	0.69	0.83	0.76	-	0.83
NH3	-	1.52	1.89	-	1.20	1.90	1.45	-	1.28
H2O	-	2.33	3.32	-	2.89	4.45	3.61	-	3.34
DISTILLATION, °F									
IBP	560	214	186	537	306	196	280	511	321
5 °/°	619	375	352	594	404	365	434	610	401
10	713	431	388	628	477	445	485	688	457
30	1015	586	535	926	574	551	655	994	541
50	-	809	691	-	740	737	837	-	667
70	-	-	892	-	901	916	-	-	768
90	-	-	-	-	-	-	-	-	874
95	-	-	-	-	-	-	-	-	909
EP	-	-	-	-	-	-	-	-	987

TABLE B-9

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 90°/° BLEND MONTEREY SRC/SOLVENT (J-8991)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2628 UNIT: CT-146

	CHARGE	MB-871	MB-872	MB-873	MB-874	MB-875	MB-876	MB-877	MB-878	MB-879	MB-880
MATERIAL BALANCE, PCT WT	-	102.1	107.2	105.6	172.0	105.3	116.0	99.5	95.7	104.8	95.5
<u>OPERATING CONDITIONS</u>											
TEMPERATURE, °F	-	720	778	781	769	748	777	775	798	800	770
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.24	0.28	0.46	0.40	0.17	0.22	0.20	0.15	0.38	0.11
H2 CIRCULATION, SCF/B	-	7565	9538	7130	25448	19682	8841	12084	10769	10978	13871
DAYS ON STREAM	-	2.2	3.7	4.6	5.1	6.0	7.0	8.0	9.0	9.7	10.5
<u>YIELDS, WT</u> °/°											
C1-C3	-	0.23	1.24	2.08	1.89	1.47	0.44	2.46	3.23	3.53	1.91
C4	-	0.04	3.90	1.44	1.57	0.93	0.34	1.25	1.44	1.30	1.30
C5	-	0.02	1.77	0.70	0.29	0.21	0.04	0.06	0.27	0.17	0.12
C6+	100.00	96.92	94.33	93.30	93.67	94.36	95.78	93.12	91.49	92.42	93.15
H2S	-	0.95	1.01	0.87	0.83	1.01	1.01	1.01	1.04	0.94	1.02
NH3	-	1.11	1.60	1.27	0.88	1.29	1.47	1.53	1.80	1.16	1.63
H2O	-	2.77	4.32	3.50	3.26	3.67	4.09	4.11	4.53	3.49	4.33
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)											
IBP	-	334	292	293	365	324	306	275	232	294	346
5 °/°	-	456	414	434	486	472	436	418	379	420	455
10	-	519	473	503	537	522	509	493	452	486	508
30	-	737	615	689	755	709	680	670	581	684	658
50	-	967	788	873	-	864	829	819	720	865	788
70	-	-	-	-	-	-	-	-	-	-	-
90	-	-	-	-	-	-	-	-	-	-	-
95	-	-	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-	-	-

TABLE B-10

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 50°/° BLEND BURNING STAR SRC/SOLVENT (76D-2351)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2455 UNIT: CT-146

	CHARGE	MB-806	MB-807	MB-808	MB-809	MB-810	MB-811
MATERIAL BALANCE, PCT WT	-	91.0	99.0	97.6	88.7	96.2	100.0
OPERATING CONDITIONS							
TEMPERATURE, °F	-	672	729	780	673	725	777
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
LHSV	-	0.84	0.84	0.77	0.22	0.19	0.27
H_2 CIRCULATION, SCF/B	-	6609	7558	8027	7670	8317	6488
DAYS ON STREAM	-	1.0	1.5	1.8	3.0	4.6	6.0
YIELDS, WT °/°							
C_1-C_3	-	0.16	0.41	1.17	0.42	0.96	2.52
C_4	-	0.15	0.22	0.49	0.21	0.50	0.69
C_5	-	0.05	0.01	0.14	0.03	0.15	0.23
C_6+	100.00	98.20	97.76	96.04	97.60	96.63	94.72
H_2S	-	0.31	0.37	0.43	0.37	0.44	0.46
NH_3	-	0.45	0.64	1.23	0.93	1.14	1.20
H_2O	-	2.30	3.30	4.09	3.53	4.41	4.52
DISTILLATION, °F (D-2887)							
IBP	329	204	205	180	175	181	176
5 °/°	392	343	293	269	281	265	216
10	405	385	378	350	373	340	275
30	481	426	448	444	448	441	414
50	583	475	528	527	525	507	487
70	-	548	705	649	667	589	575
90	-	-	-	-	-	-	-
95	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-

TABLE B-11

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 70 AND 90°/° BLENDS BURNING STAR SRC/SOLVENT (J-9031/J-9032)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2655 UNIT: CT-146

	CHARGE	MB-881	MB-882	CHARGE	MB-883	MB-884
MATERIAL BALANCE, PCT WT	-	100.0	93.7	-	116.3	106.5
<u>OPERATING CONDITIONS</u>						
TEMPERATURE, °F	-	724	773	-	722	773
PRESSURE, PSIG	-	2000	2000	-	2000	2000
LHSV	-	0.84	0.43	-	0.55	1.04
H2 CIRCULATION, SCF/B	-	5022	9465	-	4473	4255
DAYS ON STREAM	-	2.6	3.1	-	7.2	8.2
<u>YIELDS, WT</u> °/°						
C1-C3	-	0.17	1.30	-	0.42	1.94
C4	-	0.46	0.69	-	0.30	0.58
C5	-	0.29	0.15	-	0.08	0.36
C6+	100.00	97.63	95.75	100.00	99.44	96.51
H2S	-	0.30	0.47	-	0.30	0.29
NH3	-	0.46	0.93	-	0.57	0.51
H2O	-	1.86	2.86	-	0.38	1.79
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)						
IBP	-	-	383	-	-	-
5 °/°	-	-	469	-	-	-
10	-	-	516	-	-	-
30	-	-	668	-	-	-
50	-	-	908	-	-	-
70	-	-	-	-	-	-
90	-	-	-	-	-	-
95	-	-	-	-	-	-
EP	-	-	-	-	-	-

TABLE B-12

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 70°/° BLEND WYODAK SRC/SOLVENT (J-9075)
CATALYST: HARSHAW 618X (J-7243)
PROGRAM: 2671   UNIT: 146

	CHARGE	MB-885	MB-886	MB-887	MB-888	MB-889	MB-890
MATERIAL BALANCE, PCT WT	-	107.2	98.7	124.4	100.4	101.5	100.2
<u>OPERATING CONDITIONS</u>							
TEMPERATURE, °F	-	739	739	775	727	799	723
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
LHSV	-	0.70	0.58	0.25	0.38	0.32	0.40
H_2 CIRCULATION, SCF/B	-	4099	4819	31981	5877	6865	7950
DAYS ON STREAM	-	1.2	1.7	2.5	4.0	5.0	5.9
<u>YIELDS, WT</u> °/°							
C_1-C_3	-	1.42	0.43	9.91	0.58	2.95	0.57
C_4	-	0.79	0.24	2.87	0.30	0.98	0.32
C_5	-	0.64	0.08	0.25	0.02	0.24	0.12
C_6+	100.00	95.85	97.62	85.90	97.56	93.91	97.26
H_2S	-	0.13	0.13	0.16	0.13	0.16	0.14
NH_3	-	0.63	0.63	1.14	0.77	1.26	0.62
H_2O	-	2.57	2.74	3.80	2.96	3.75	2.75
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)							
IBP	-	340	354	338	313	224	334
5 °/°	-	412	411	424	394	357	417
10	-	441	438	451	429	394	444
30	-	591	555	586	543	517	588
50	-	-	-	790	-	670	-
70	-	-	-	-	-	-	-
90	-	-	-	-	-	-	-
95	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-

TABLE B-13

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33°/° BLEND SCT MONTEREY SRC/SOLVENT (77D-453)
CATALYST: HARSHAW 618X (J-7242)
PROGRAM: 2490 UNIT:CT-146

	CHARGE	MB-819	MB-820	MB-821	MB-822	MB-823	MB-824	MB-825	MB-826
MATERIAL BALANCE, PCT WT	-	115.9	105.8	121.9	105.9	99.1	102.0	97.6	99.7
<u>OPERATING CONDITIONS</u>									
TEMPERATURE, °F	-	720	782	769	721	673	676	725	729
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.42	0.60	0.39	0.21	0.19	0.57	0.30	0.90
H2 CIRCULATION, SCF/B	-	8207	6977	5318	9903	9975	6194	8009	9258
DAYS ON STREAM	-	1.9	3.2	4.9	6.1	7.6	8.8	9.8	10.5
<u>YIELDS, WT</u> °/°									
C1-C3	-	1.50	1.86	2.00	1.70	0.57	0.26	0.39	0.55
C4	-	1.99	0.88	1.67	1.86	0.55	0.18	0.28	0.34
C5	-	1.07	0.30	1.06	0.89	-	-	0.05	-
C6+	100.00	93.53	94.98	93.86	94.22	98.44	98.69	97.70	98.03
H2S	-	0.93	1.00	0.93	1.00	0.90	0.69	0.95	0.83
NH3	-	0.88	1.04	0.90	1.07	0.75	0.41	0.95	0.49
H2O	-	3.64	4.21	3.78	4.04	1.98	1.73	3.31	2.09
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)									
IBP	364	181	180	179	184	244	256	273	63
5 °/°	398	310	258	250	335	390	400	339	363
10	433	381	324	315	399	401	422	381	386
30	490	467	449	446	472	478	493	436	478
50	588	531	516	511	530	554	590	480	566
70	-	641	612	605	625	725	-	582	696
90	-	-	-	-	-	-	-	-	-
95	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-

TABLE B-14

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33°/° BLEND SCT MONTEREY SRC/SOLVENT (77D-543)
CATALYST: MOBIL CATALYST 'A'
PROGRAM: 2525 UNIT: CT-146

	CHARGE	MB-836	MB-837	MB-838	MB-839	MB-840	MB-841	MB-842	MB-843
MATERIAL BALANCE, PCT WT	-	97.2	100.0	101.0	99.1	114.6	102.2	102.9	97.8
<u>OPERATING CONDITIONS</u>									
TEMPERATURE, °F	-	728	779	673	671	727	784	723	726
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
LHSV	-	0.41	0.52	0.51	1.03	0.97	1.07	0.51	0.30
H2 CIRCULATION, SCF/B	-	6751	5824	5258	5906	5367	6158	6577	9300
DAYS ON STREAM	-	1.6	2.6	3.6	4.2	4.7	5.2	6.1	7.2
<u>YIELDS, WT</u> °/°									
C1-C3	-	0.68	1.54	0.20	0.17	0.23	1.08	0.60	0.56
C4	-	0.31	0.46	0.20	0.20	1.49	0.39	0.35	2.35
C5	-	0.03	0.13	0.01	0.04	0.94	0.15	0.08	1.48
C6+	100.00	97.27	95.52	98.54	99.06	96.31	96.52	97.63	94.10
H2S	-	0.85	0.93	0.55	0.45	0.64	0.81	0.68	0.89
NH3	-	0.72	0.85	0.33	0.27	0.39	0.43	0.42	0.72
H2O	-	2.99	3.89	1.19	0.49	1.57	2.46	1.77	3.02
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)									
IBP	364	200	29	317	332	267	205	237	203
5 °/°	398	330	161	392	392	391	389	391	363
10	433	391	340	402	401	400	399	400	392
30	490	452	439	476	473	475	475	475	466
50	588	521	507	556	550	556	566	554	526
70	-	637	609	775	774	758	809	765	644
90	-	-	-	-	-	-	-	-	-
95	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-

TABLE B-15

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33•/• BLEND SCT MONTEREY SRC/SOLVENT (77D-453)
CATALYST: MOBIL CATALYST 'B'
PROGRAM: 2522 UNIT: CT-146

	CHARGE	MB-844	MB-845	MB-846	MB-847
MATERIAL BALANCE, PCT WT	-	101.0	103.9	100.8	101.0
OPERATING CONDITIONS					
TEMPERATURE, •F	-	724	778	726	777
PRESSURE, PSIG	-	2000	2000	2000	2000
LHSV	-	0.50	0.50	0.24	0.18
H2 CIRCULATION, SCF/B	-	7573	7851	8228	9506
DAYS ON STREAM	-	1.6	2.6	4.1	5.5
YIELDS, WT •/•					
C1-C3	-	0.72	1.87	1.27	2.36
C4	-	0.18	0.72	0.40	0.92
C5	-	0.06	0.26	0.32	0.32
C6+	100.00	97.09	94.93	95.68	92.49
H2S	-	0.92	1.02	0.97	1.01
NH3	-	0.77	1.01	0.87	1.18
H2O	-	3.33	4.21	3.78	6.23
DISTILLATION, •F (D-2887)					
IBP	364	-	178	211	180
5 •/•	398	-	275	340	315
10	433	-	348	395	378
30	490	-	431	457	447
50	588	-	488	513	499
70	-	-	576	612	573
90	-	-	-	-	735
95	-	-	-	-	830
EP	-	-	-	-	-

TABLE B-16

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: 33°/° BLEND SCT MONTEREY SRC/SOLVENT (77D-453)
CATALYST: MOBIL CATALYST 'C'
PROGRAM: 2528   UNIT: CT-146

	CHARGE	MB-848	MB-849	MB-850	MB-851
MATERIAL BALANCE, PCT WT	-	95.5	101.3	104.2	104.9
<u>OPERATING CONDITIONS</u>					
TEMPERATURE, °F	-	775	793	792	771
PRESSURE, PSIG	-	2000	2000	2000	2000
LHSV	-	0.45	0.51	0.24	0.28
H2 CIRCULATION, SCF/B	-	9225	6736	7082	6912
DAYS ON STREAM	-	2.3	4.3	5.6	7.0
<u>YIELDS, WT</u> °/°					
C1-C3	-	2.04	2.79	3.06	3.20
C4	-	0.73	0.89	0.86	0.91
C5	-	0.25	0.34	0.17	0.22
C6+	100.00	96.64	94.01	93.53	94.06
H2S	-	0.75	0.95	1.05	0.92
NH3	-	0.88	0.93	1.11	0.97
H2O	-	2.79	3.91	4.54	3.59
<u>DISTILLATION</u>, °F (D-2887)					
IBP	364	182	189	190	181
5 °/°	398	302	309	318	280
10	433	360	365	374	350
30	490	450	450	457	454
50	588	508	511	519	525
70	-	590	594	607	629
90	-	-	-	-	-
95	-	-	-	-	-
EP	-	-	-	-	-

TABLE B-17

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
CHARGE: H-COAL FUEL OIL(400°F+) (J-8723)
CATALYST: HDS-1441A (J-7278)
PROGRAM: 2469 UNIT: CT-146

	CHARGE	MB-812	MB-813	MB-814	MB-815	MB-816	MB-817
MATERIAL BALANCE, PCT WT	-	105.3	96.8	98.1	86.4	99.5	104.0
<u>OPERATING CONDITIONS</u>							
TEMPERATURE, °F	-	674	729	779	675	724	775
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
LHSV	-	1.00	1.01	1.00	0.36	0.35	0.36
H2 CIRCULATION, SCF/B	-	7736	5385	6384	4294	9710	6542
DAYS ON STREAM	-	0.7	1.4	2.0	3.3	5.3	6.8
<u>YIELDS, WT</u> °/°							
C1-C3	-	0.12	0.34	0.25	0.11	0.68	3.04
C4	-	0.15	0.21	0.23	0.16	0.60	3.85
C5	-	0.05	0.10	0.14	0.06	0.34	2.47
C6+	100.00	98.80	98.57	98.33	99.02	98.36	90.60
H2S	-	0.13	0.18	0.21	0.14	0.19	0.22
NH3	-	0.20	0.41	0.57	0.31	0.59	0.83
H2O	-	1.14	1.36	1.80	1.13	1.14	1.82
<u>DISTILLATION</u>, °<u>F</u> (<u>D</u>-2887)							
IBP	346	273	227	215	272	219	213
5 °/°	398	399	398	382	400	391	385
10	434	433	427	412	437	424	425
30	527	525	523	506	527	517	512
50	618	611	609	584	609	596	578
70	741	730	732	684	725	699	661
90	-	-	-	-	-	-	875
95	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-