Upgrading of Coal Liquids for Use as Power Generation Fuels

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ABSTRACT

Residual coal liquids were hydroprocessed in a fixed bed unit to upgrade them to power generation fuels. A series of catalysts was evaluated for the desulfurization of short contact time (SCT) SRC. Low sulfur (0.4 wt %) boiler fuels were produced with hydrogen consumptions as low as 800 and 1200 scf/B from Indiana V regular SRC and W. Kentucky SCT SRC, respectively. Constant temperature aging runs were conducted with both regular and short contact time SRC. A kinetic aging model was developed to estimate process conditions and yields in either a fixed bed or ebullated bed reactor.

Chemical characterization indicated that W. Kentucky SCT SRC molecules are significantly larger and more polar than those of Indiana V regular SRC. The high conversion of asphaltenes to less polar material does not appear necessary for SRC desulfurization. A structural comparison was made between SCT SRC recycle solvent and hydroprocess-generated solvent to assess the potential of process solvent in an integrated short contact time SRC/hydrotreating scheme. The alkali metals found in SRC distillate were determined not to be organically bound and are easily removed by water washing or distillation. A heating value correlation was developed from a wide range of raw and hydroprocessed coal liquids, based on elemental composition.

EPRI PERSPECTIVE

PROJECT DESCRIPTION

This is a final report issued under Research Project (RP) 361-2. This three-year project followed an earlier study (RP361-1) also co-funded by EPRI and Mobil Research and Development Corporation, which concluded that the raw products from the solvent refined coal (SRC) (RP1234) and the H-Coal (RP238) processes would require considerable upgrading to meet current specifications for combustion turbine fuels. Initially, the current contract was aimed at the use of laboratory-scale, continuous equipment for the application of various petroleum-refining operations to the upgrading of hydrogen-deficient, raw coal liquids to acceptable turbine fuels. Much of the work centered around use of catalytic hydroprocessing as an upgrading step. The feedstocks were chosen from various distillate and distillate-residual blends from the SRC-I pilot plant at Wilsonville and the H-Coal process development unit. Small-scale combustor tests were run on limited available quantities of raw and upgraded distillate fuels in order to obtain a preliminary assessment of the extent of upgrading required to arrive at acceptable turbine fuels. Some preliminary hydroprocessing studies using dilute SRC-I and recycle solvent blends were also carried out during this period.

The subject of this report, covering the third and final year of the project, involves the investigation of catalytic hydroprocessing as a means of upgrading both conventional and short contact time SRC (SCT SRC) to low sulfur boiler fuel. A major portion of the study was concerned with the evaluation of catalysts for the upgrading of these heavy residual coal liquids. In the case of the SCT SRC, the upgrading step is an integral part of the short contact time processing approach being investigated at the Wilsonville SRC pilot plant (RP1234) and at a smaller-scale by Conoco Coal Development Company (RP1134 and 779-22) and by the Kerr-McGee Corporation (RP1134 and 1715). The integration of short contact time coal dissolution with a subsequent catalytic upgrading step offers the potential for reduced overall hydrogen consumption, which would have a favorable impact on utility fuel costs. Mobil is also carrying out a laboratory study (RP1655) aimed at further understanding the chemistry of the catalytic liquefaction and hydroprocessing of heavy coal liquids into high hydrogen content liquid fuels.

PROJECT OBJECTIVE

The overall objective of this project was to evaluate various conventional petroleum-refining operations as a means of upgrading a variety of raw coal liquids to acceptable power generation fuels. In addition to the evaluation of technical feasibility, information on which to base a preliminary evaluation of the economic feasibility of the various upgrading operations was sought. The specific objective of the work performed during the last year of the contract was to further evaluate catalytic hydroprocessing as a means of upgrading several varieties of raw, solvent-refined coal products to higher quality utility boiler fuels while evaluating a number of catalysts as to their suitability for use in this processing step.

PROJECT RESULTS

During this reporting period, the project demonstrated that raw, residual coal liquids ranging from SCT SRC through conventional (with respect to first-stage hydroliquefaction processing severity) critical solvent deashed SRC (CSD SRC) can be upgraded to higher quality boiler fuels via catalytic hydroprocessing. Despite differences in hydrogen consumption, hydroprocessing severity, and catalyst requirements, low sulfur boiler fuel can be prepared from each of the three qualities (SCT SRC, filtered SRC, and CSD SRC) of feedstock studied. This permits greater flexibility in the choice of several processing routes for the production of high quality boiler fuels.

Hydroprocessing of SCT SRC was found to be more difficult than it was in the case of the other two feedstocks. Its high concentration of highly polar, high-molecular weight fractions is believed to be responsible for its high viscosity (which makes it difficult to pump), its lessened reactivity, and its propensity for rapid catalyst deactivation. However, based on the proper selection of pore size and pore size distribution, it was possible to substantially increase catalyst activity for heteroatom (sulfur, nitrogen, and oxygen) removal while significantly reducing Conradson carbon content of the upgraded products.

Of particular significance to the catalytic hydroprocessing of residual coal liquids is the selective removal of the most hard-to-convert material through the critical solvent deashing process. When CSD SRC replaced filtered SRC as the hydroprocessing unit feedstock, a three-fold increase in reactor throughput was obtained while maintaining 70 percent sulfur removal. As the critical solvent deashing process has also been shown (RP1134-2) to be capable of fractionating SCT SRC into lighter and heavier components, it should be possible also to enhance the hydroprocessability of SCT SRC in a similar manner. As the most hard-to-convert components are believed to contribute strongly to catalyst deactivation, further improvements in a two-stage process that integrates short contact time solvent refining, critical solvent deashing, and catalytic hydroprocessing may be possible. Demonstration of such an integrated process is planned at Kerr-McGee under RP1715-1.

Finally, expansion of the kinetic process model developed earlier in the project to include the new feedstocks as well as catalyst-aging effects has expanded the capabilities for relating processing conditions to product properties. This model will be used to estimate the incremental cost of upgrading SRC to higher quality boiler fuels.

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Section 1

SUMMARY

1.1 INTRODUCTION

This section is a three-year summary of research project RP 361-2. Details of the final year are given in Sections 2 through 8. The overall objective of this project has been to evaluate the feasibility, both technical and economic, of upgrading coal liquids via hydroprocessing into power generation fuels. It is anticipated that the results and conclusions obtained from this project will provide basic information for possible use in the planning and designing of integrated coal liquefaction/upgrading plants.

The coal liquids evaluated and processed consisted primarily of solvent refined coal (SRC), SRC process solvent, and H-Coal distillates. These liquids were catalytically hydroprocessed in a down-flow fixed bed pilot unit to yield products ranging from low sulfur solid boiler fuel to high quality turbine fuel. Both fresh catalyst activities and long-term aging were studied. The combustion characteristics of the liquid products were also investigated.

In the upgrading of SRC to produce low sulfur boiler fuel, the hydroprocessing of regular as well as short contact time SRC was studied. Also, the effects of coal source on SRC upgrading requirements were examined. A comprehensive process development program was carried out to identify optimal operating conditions, maximum SRC concentration feeds, and catalysts with high activity and efficient hydrogen utilization. Kinetic models were developed to describe the heteroatom removal rates and to translate process design conditions from fixed bed to ebullated bed reactors.

An automatic gradient elution chromatograph (GEC) system was developed in this project to characterize the functional classes of raw and hydrotreated SRC's. These analyses elucidate the chemical changes which take place during hydroprocessing and serve as an indicator of catalyst selectivity and activity.

1.2 UPGRADING DISTILLATE COAL LIQUIDS

Feedstock

Elemental and distillation analyses of representative samples of distillate

TABLE 1-1

PROPERTIES OF COAL-DERIVED DISTILLATES

	SRC	SRC	SRC	H-COAL DISTILLATE	
	LIGHT ORGANIC LIQUID	RECYCLE SOLVENT	WASH SOLVENT	FUEL OIL MODE	SYNCRUDE MODE
ANALYSIS					
GRAVITY, API	35.4	5.5	14.5	18.6	17.1
HYDROGEN, WT °/°	11.30	7.71	8.68	10.14	9.80
SULFUR "	0.40	0.40	0.16	0.11	0.13
NITROGEN "	0.23	0.62	0.36	0.38	0.38
OXYGEN "	5.00	3.77	5.90	1.20	1.50
DISTILLATION (D2887), °F					
10 °/°	177	360	349	333	328
30 °/°	250	400	389	397	396
50 °/°	317	441	409	441	433
70 °/°	347	509	440	498	489
90 °/°	399	614	461	626	590

coal liquids evaluated in this project are given in Table 1-1. Light organic liquids from the SRC process have the highest hydrogen content, followed by H-Coal distillates and SRC process recycle solvents. Although there are no specifications of the turbines for coal derived liquids yet, some of the general specifications for petroleum base fuels are given in Table 1-2. As compared with the specifications, the coal liquids are hydrogen deficient, but nitrogen rich.

Processing

As discussed previously, a major requirement in the upgrading of coal liquids to turbine fuels should be an increase in hydrogen content and a reduction in nitrogen content. These low boiling range materials may be efficiently processed with small-pore, high surface area hydrotreating catalysts. In this work both commercial NiMo/Al_2O_3 (American Cyanamid HDS-9A) and CoMo/Al_2O_3 (HDS-1441A) catalysts were tested. Table 1-3 shows the operating conditions necessary to achieve a hydrogen content above 11 wt % in the product. Light organic liquids can be upgraded in hydrogen content from 11.3 to 13 wt % at relatively mild conditions (625°F; 500 psig; 1.0 LHSV). H-Coal distillates and SRC process recycle solvents require more severe conditions (2500 psig, 0.5 LHSV) to yield liquids with hydrogen contents exceeding 12 wt %. Nevertheless, the heteroatom removal is high for all three liquids; desulfurizing and denitrogenation are generally higher than 80%. Therefore, hydrogenation is the key catalytic objective in the upgrading of coal liquids to turbine fuels.

The importance of hydrogenation also causes an economic strain on the use of coal liquids for turbine fuels. The coal liquids hydroprocessed in this project range in hydrogen content from 6.8 to 11.3 wt %. Approximately 70 percent of the hydrogen consumed in the processing directly increases the hydrogen content of the product. In Figure 1-1 the total liquid product hydrogen content is related to total chemical hydrogen consumption for the coal liquids processed. The slope of each line in this figure is indicative of the hydrogenation selectivity for the liquid product. In order to achieve a 12 wt % hydrogen fuel, the hydrogen consumption will be 500 scf/B for the SRC light organic liquids, 1500 scf/B for the H-Coal distillates, and 3000 scf/B for the SRC recycle solvents.

Nitrogen content may also be a potential limitation on the use of coal liquids. However, the problem of nitrogen content is somewhat less than that of the hydrogen content, primarily due to relatively high denitrogenation in the upgrading step. As discussed in the following paragraph (Combustion Test), the NO_x emissions of severely hydrotreated coal liquids (N <0.1 wt %) are similar to the level of emissions for No. 2 fuel oils. Figure 1-2 shows the hydrogen requirements to denitrogenate the distillate coal liquids. This relationship is somewhat dependent on operating

TABLE 1-2

GENERAL ELECTRIC LIQUID FUEL SPECIFICATIONS FOR GAS TURBINES

	DISTILLATES	
	LIGHT	*HEAVY*
KIN. VISC., CS, 100°F, MIN.	0.5	1.8
KIN. VISC., CS, 100°F, MAX.	5.8	30.0
DIST. TEMP., 90°/° POINT, °F, MAX.	650	-
CARB. RES. (100°/° SAMPLE) WT °/°, MAX.	1.0	1.0
ASH, PPM, MAX.	50	50
TRACE METALS, PPM, MAX.		
SODIUM PLUS POTASSIUM	1	1
LEAD	1	1
VANADIUM (UNTREATED)	0.5	0.5
CALCIUM	2	2
WATER AND SEDIMENT, VOL °/°, MAX.	0.1	0.1
FUEL COMPATIBILITY, TUB NO., (50/50 MIX WITH SECOND FUEL)	-	2
SULFUR, WT °/°, MAX.(A)	0.5	0.5
HYDROGEN, WT °/°, MIN.	12.0	12.0
NITROGEN, WT °/°, MAX.(B)	-	-

(A) *OR COMPLIANCE TO ANY APPLICABLE CODES.*

(B) *FUEL-BOUND NITROGEN MAY BE LIMITED TO MEET ANY APPLICABLE CODES ON TOTAL* NO_X *EMISSION.*

TABLE 1-3

FIXED BED HYDROPROCESSING COAL DISTILLATES TO TURBINE FUELS

	199-552	199-566	199-568	199-482	199-487	199-479	199-505	199-500	199-520	199-531
FEED CHARGE	SRC LT ORGANIC LIQUID			SRC RECYCLE SOLVENT					H-COAL DISTILLATE	
CATALYST	← HDS-9A →						← HDS-1441A →			
OPERATING CONDITIONS										
TEMPERATURE, °F	624	625	615	721	731	729	727	726	726	724
PRESSURE, PSIG	500	1000	1000	1800	1200	2500	1200	1800	2500	1200
LHSV, VO/HR/VCAT	1.02	2.00	3.97	0.46	0.99	0.45	1.01	0.50	0.50	0.46
HYDROGEN CIRC., SCF/B	5000	5000	5000	5000	5000	5000	5000	5000	5000	5000
LIQUID PRODUCT PROPERIES										
GRAVITY, API	45.3	44.7	41.4	24.7	17.4	27.7	17.2	32.8	27.4	24.6
HYDROGEN, WT °/°	12.93	12.92	12.26	11.30	9.46	12.05	9.55	10.67	11.80	11.04
SULFUR ''	0.06	0.02	0.01	0.01	0.02	0.01	0.02	0.01	0.01	0.01
NITROGEN, ''	0.03	0.06	0.18	0.09	0.20	0.03	0.27	0.09	0.01	0.04
OXYGEN, ''	0.90	1.10	2.60	0.10	0.90	0.20	0.80	0.30	0.10	0.20
HETEROATOM REMOVAL, °/°										
SULFUR	89.8	96.2	99.4	97.6	94.6	97.6	96.0	98.8	90.3	93.0
NITROGEN	78.5	73.6	37.1	97.6	78.7	95.2	81.3	92.9	99.9	83.5
OXYGEN	89.8	81.8	41.9	85.0	67.1	95.0	56.2	85.1	97.1	88.5
HYDROGEN UTILIZATION, SCF/B										
H2 CONSUMED BY C1-C5	165	234	228	92	94	67	259	79	65	89
H2 CONSUMED BY C6+	579	515	251	2317	1029	2909	1020	1867	984	458
H2 CONSUMED BY S,N,O REMOVAL	291	258	141	434	352	434	352	418	148	128
TOTAL H2 CONSUMPTION	1035	1007	620	2843	1475	3410	1631	2364	1197	678

Figure 1-1

CHEMICAL HYDROGEN
CONSUMPTION REQUIRED TO HYDROPROCESS
COAL LIQUIDS TO FUELS OF VARYING HYDROGEN CONTENT

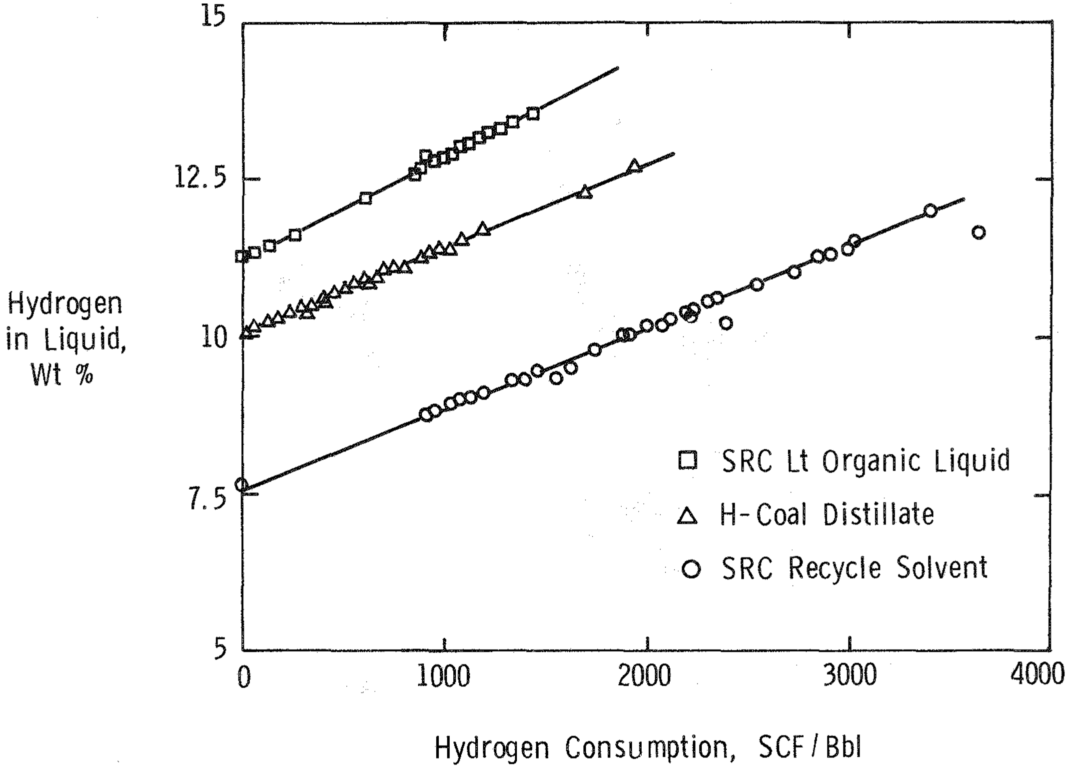
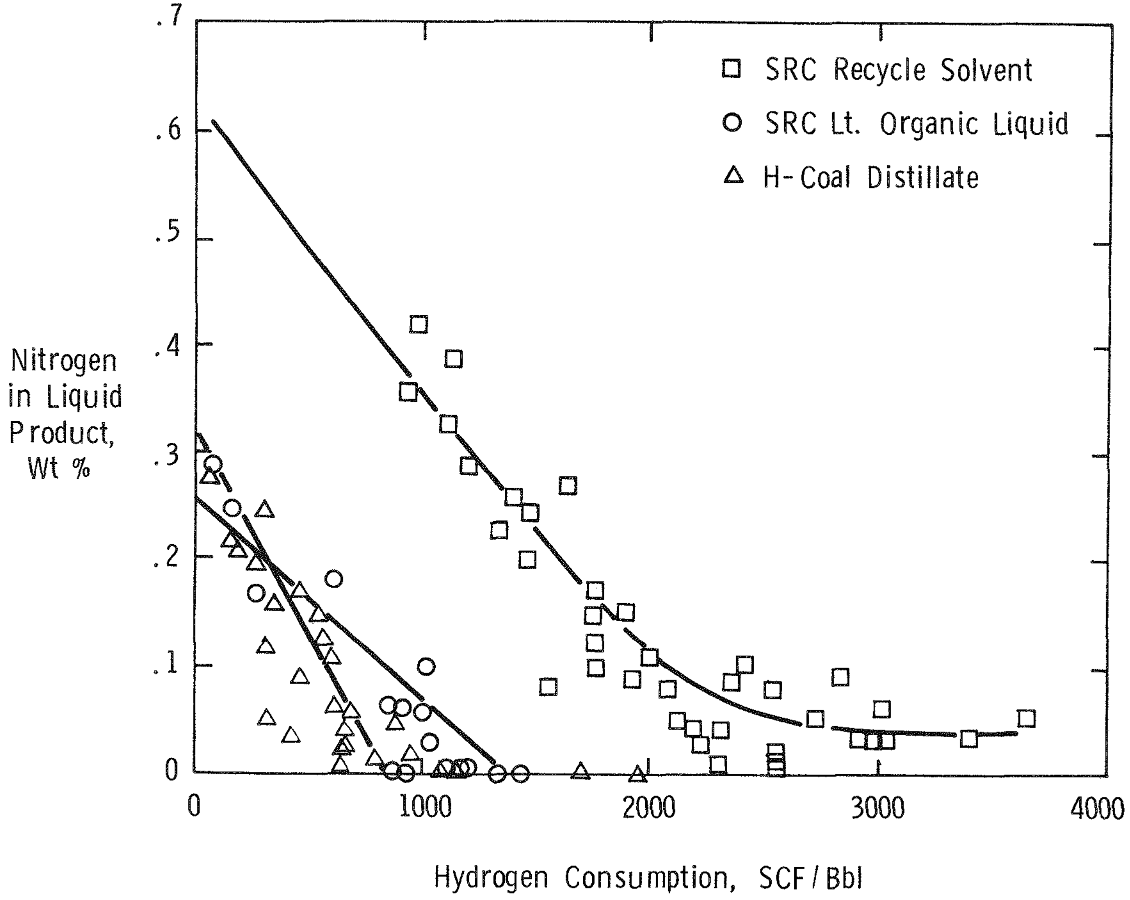


Figure 1-2

CHEMICAL HYDROGEN CONSUMPTION REQUIRED TO HYDROPROCESS COAL LIQUIDS TO FUELS OF VARYING NITROGEN CONTENT



conditions and catalyst. However, the data show that to produce a 0.1 wt % nitrogen fuel, the hydrogen consumption is about 600 scf/B for the H-Coal distillate, 800 scf/B for SRC light organic liquid, and 2,000 scf/B for SRC recycle solvents.

Although the hydrogen consumption is high for the upgrading of distillate coal liquids, the catalyst deactivation is generally mild. The results from a 30-day aging run of H-Coal distillates over Cyanamid HDS-1441A catalyst show a very low catalyst deactivation, 5-10°F/month temperature rise, at moderate operating conditions. Consequently, 6-12 month cycle lengths can be anticipated.

Combustion Test

Raw and hydrotreated distillate coal liquids were tested in an existing pressurized combustor passage, modified to accept small quantities of liquid (1-2 gallons per hour). The hydrogen contents in the liquids tested range from 5.3 wt % to 12 wt %, and the nitrogen contents ranged from 0.62 wt % to 0.04 wt %. A petroleum-derived No. 2 fuel oil was used as a reference. The evaluations include fuel atomization, coke formation, combustion parameters, and emissions.

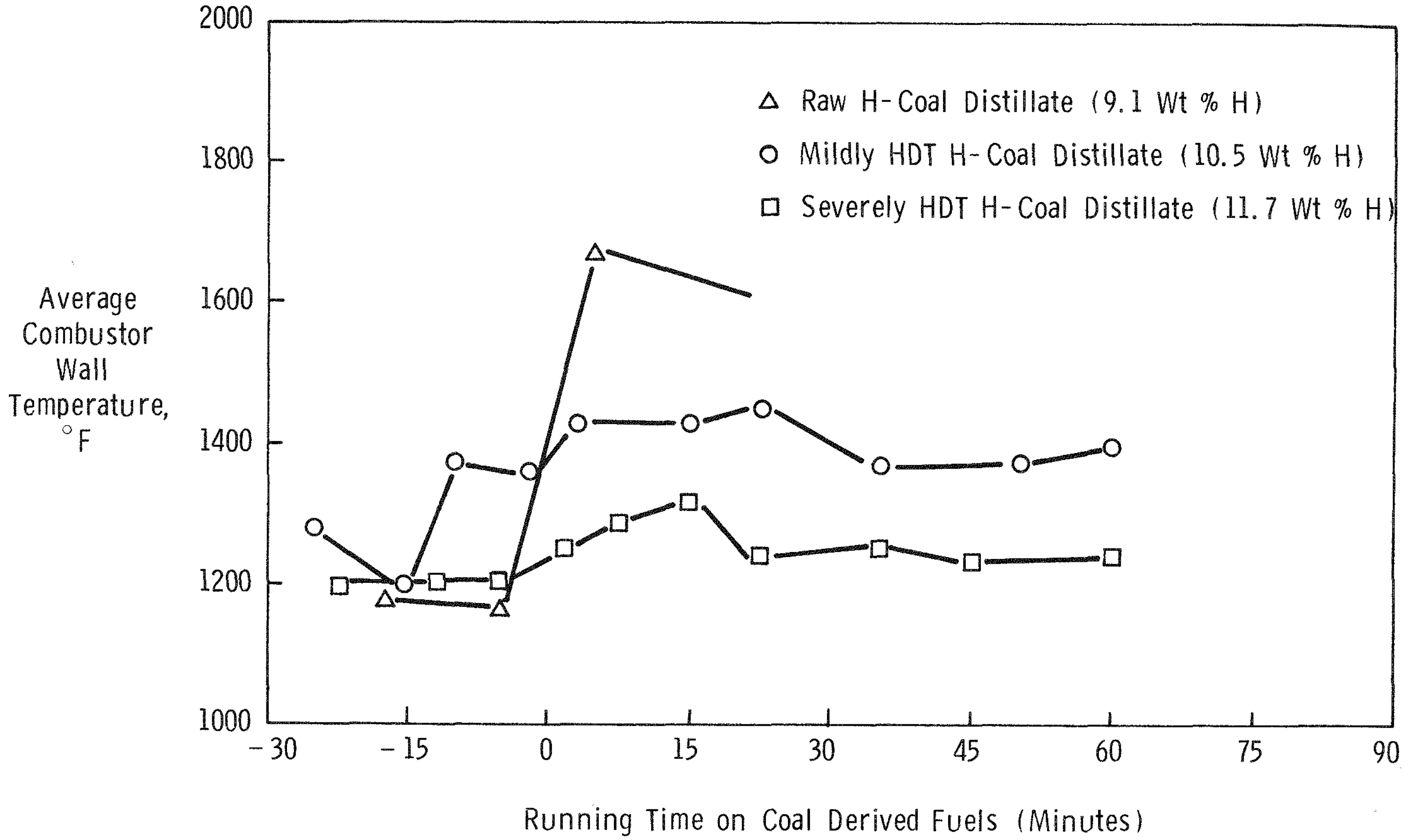
The coal liquid fuels tested, without exception,: were readily forwarded and atomized with no evidence of incompatibility with the #2 oil used for startup purposes; had no problem with deposits in fuel lines and had excellent atomizing characteristics. It is suggested, however, that the lighter fractions 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 negligible for the case of the 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 (1). Subsequent tests have shown that smoking is not a significant problem in large combustors (2).

As shown in Figure 1-3, the average combustor wall temperature was always higher for the coal-derived fuels, compared to No. 2 fuel oil. This is due to the higher thermal radiation from these hydrogen-deficient fuels. The increased heat transfer to the combustor wall will require operation at derated conditions or a redesign of the combustor. Alternatively, higher hydrogen content via hydroprocessing of the coal liquids alleviates this problem (1).

Figure 1-3

AVERAGE COMBUSTOR WALL TEMPERATURE AS A FUNCTION OF TIME FOR THE H-COAL FUELS



The ratio of NO_x measured with coal-liquids to the base line NO_x measured with No. 2 fuel oil is plotted in Figure 1-4 as a function of the nitrogen content of the fuel. It should be noted that the NO_x values observed for the severely hydrotreated SRC recycle solvent (11.0 wt % H) and H-Coal distillates (10.5 and 11.7 wt % H) are lower than or equivalent to No. 2 fuel oil even though the coal liquids have a slightly higher nitrogen content. In these cases the contribution due to fuel-bound nitrogen is compensated for by the reduced thermal NO_x production rate. This is in agreement with the lower exhaust temperatures observed for these fuels (compared to No. 2 fuel oil).

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 (UHC's), 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. However, it should be noted that in the test of raw process solvent, incipient deposition of inorganic material in the fuel was observed. Since the levels of alkali metals present in the fuels are relatively low, longer periods of exposure are required before definitive data on the rate of attack or deposition can be established. The trace alkali metals found in the coal liquids are not organically bound and can be removed by careful distillation. The inorganic meterials in the coal liquids are also significantly reduced by the hydroprocessing, presumably by deposition upon the hydrotreating catalyst. This lowered nonhydrocarbon content should contribute to a longer turbine life.

1.3 UPGRADING SRC TO BOILER FUEL

Feedstocks

Elemental and GEC analyses of representative SRC's used in this project are given in Table 1-4. The sulfur content in the SRC is strongly dependent on the source of coals. For example, Wyodak is a low sulfur coal and yields an SRC with only 0.1 wt % S. Nitrogen and oxygen contents are relatively constant, about 1.8 wt % nitrogen and 4.0 wt % oxygen in the various samples. Both regular and SCT SRC's are listed in Table 1-4. The SCT SRC is significantly higher in oxygen content than are the regular SRC's. However, more distinct differences can be found in the GEC analyses. The SCT SRC contains nearly 30 wt % of non-eluted asphaltenes, compared

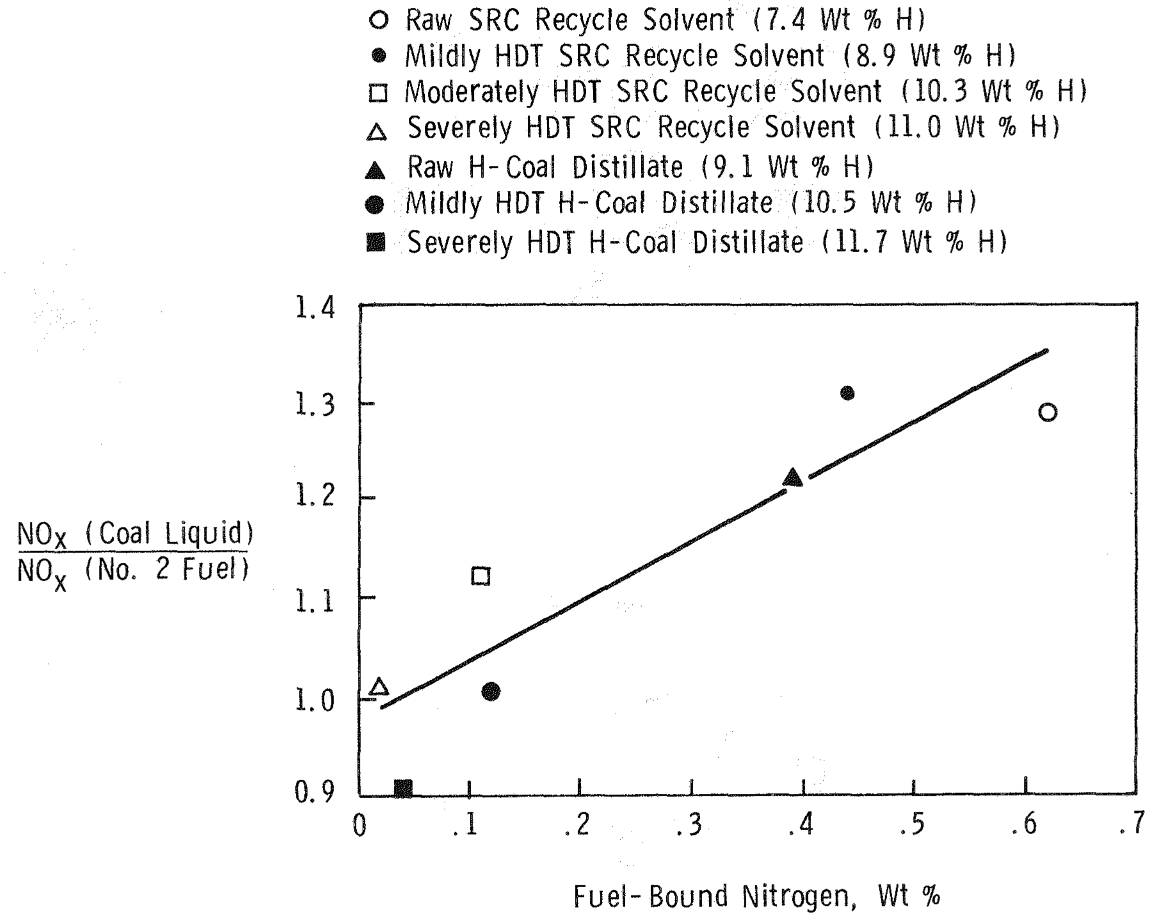
TABLE 1-4

SRC PROPERTIES

	← REGULAR			→	← SCT →
	ILL. NO. 6 BURNING STAR	ILL. NO. 6 MONTEREY	WYODAK AMAX	INDIANA NO. 5	W. KENTUCKY SCT
CHEMICAL PROPERTIES					
HYDROGEN, WT °/°	5.72	6.22	5.60	5.80	6.03
SULFUR, "	0.57	0.70	0.10	0.85	0.97
NITROGEN, "	1.71	1.75	1.70	1.97	1.99
OXYGEN, "	3.5	4.0	3.8	3.3	5.3
GEC ANALYSES, WT °/°					
SATURATES (CUT 1)	0.04	0.02	0.03	0.04	0.27
AROMATIC OILS (CUTS 2-4)	11.9	7.7	9.1	4.4	8.0
RESINS/ASPH. (CUTS 5-7)	30.8	33.0	28.8	33.1	17.2
POLAR ASPH. (CUTS 8-12)	47.3	54.1	50.9	52.7	46.1
NON ELUTED POLAR ASPH. (CUT 13)	10.0	5.2	11.2	9.7	29.4

Figure 1-4

RELATIVE NO$_X$ VALUES AS A FUNCTION OF THE FUEL-BOUND NITROGEN CONTENT OF THE COAL-DERIVED FUELS



to 5-11 wt % of the non-eluted asphaltenes in the regular SRC.

The major purpose of upgrading SRC is to produce a low sulfur boiler fuel (0.4 wt % S in the 900°F$^+$ product). Of the SRC's shown in Table 1-4, only Wyodak AMAX SRC meets this sulfur specification without need for further desulfurization. Four areas of SRC upgrading are discussed in this subsection: general process conditions, effects of SRC concentration, comparison between regular and SCT SRC, and a comparison of Kerr-McGee CSD SRC and filtered SRC. Also, the mechanism of SRC reactions is discussed on the basis of GEC analyses.

Experimental

The hydroprocessing runs were carried out in a continuous fixed bed downflow pilot unit which was extensively modified to enable the processing of highly viscous coal liquids. All lines and valves were heat traced so that temperatures could be controlled up to 500°F. The SRC/recycle solvent charges were prepared in a low pressure blending system. The homogeneity of the charge was achieved either through external circulation or internal mixing.

With these provisions, regular SRC charges up to 90% concentration were smoothly processed in this unit. To process high SRC concentration charges, line temperatures throughout the unit were kept at 420°F. Figure 1-5 shows the viscosity of regular SRC/recycle solvent blends as a function of temperature. Blends of short contact time SRC normally have higher viscosities than do regular SRC's at similar blending ratios.

Effects of SRC Concentration

The effects of SRC concentration in the charge were based on results from upgrading Monterey regular SRC over a commercial NiMo/Al_2O_3 catalyst, Harshaw 618X. Table 1-5 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 heteroatom removal and CCR reduction are also lower for the higher concentration blends. The relative ease of heteroatom removal is sulfur >oxygen>nitrogen. Figure 1-6 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 three blends. Due to the low hydrogen content of the SRC, hydrogen consumption increases with SRC concentration to achieve the same product sulfur level. However, the hydrogen

TABLE 1-5

EFFECT OF SRC CONCENTRATION ON HYDROPROCESSING MONTEREY SRC BLENDS
CATALYST: HARSHAW 618X

	146-828	146-858	146-873
SRC CONC., WT °/°	33	70	90
OPERATING CONDITIONS			
TEMPERATURE, °F	777	781	781
PRESSURE, PSIG	2000	2000	2000
LHSV, VO/HR/VCAT	0.53	0.41	0.46
DAYS ON STREAM	2.6	13.6	4.6
LIQUID PRODUCT ANALYSES			
GRAVITY, API	12.2	5.2	-1.9
HYDROGEN, WT °/°	9.17	8.67	8.28
SULFUR, ''	0.04	0.12	0.20
NITROGEN, ''	0.32	0.79	0.89
OXYGEN, ''	1.00	1.60	1.30
CCR, ''	6.15	16.90	21.32
650°*F+ YIELDS, WT °/°*	40	45	75
SULFUR IN 650°*F+, WT °/°*	0.1	0.3	0.3
HETEROATOM REMOVAL, °/°			
SULFUR	93.4	87.6	81.7
NITROGEN	72.7	54.8	56.1
OXYGEN	74.2	71.1	72.3
CCR REMOVAL	64	59	52
H2 CONSUMPTION, SCF/B	2084	2600	2562

Figure 1-5

COMPARISON OF KINEMATIC VISCOSITY OF 40 AND 70 WT % SHORT CONTACT TIME (SCT) W. KENTUCKY SRC BLENDS WITH REGULAR MONTEREY SRC BLENDS

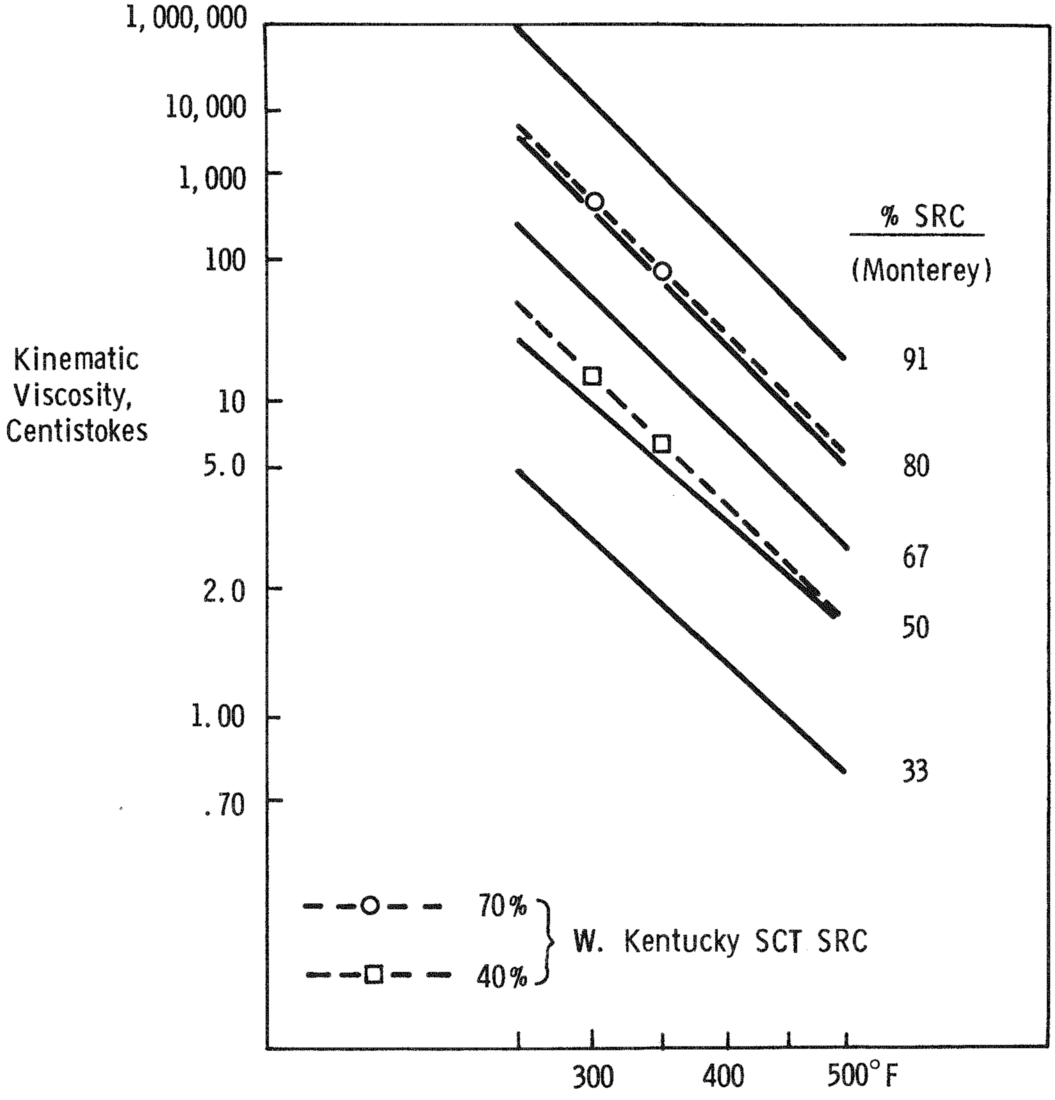
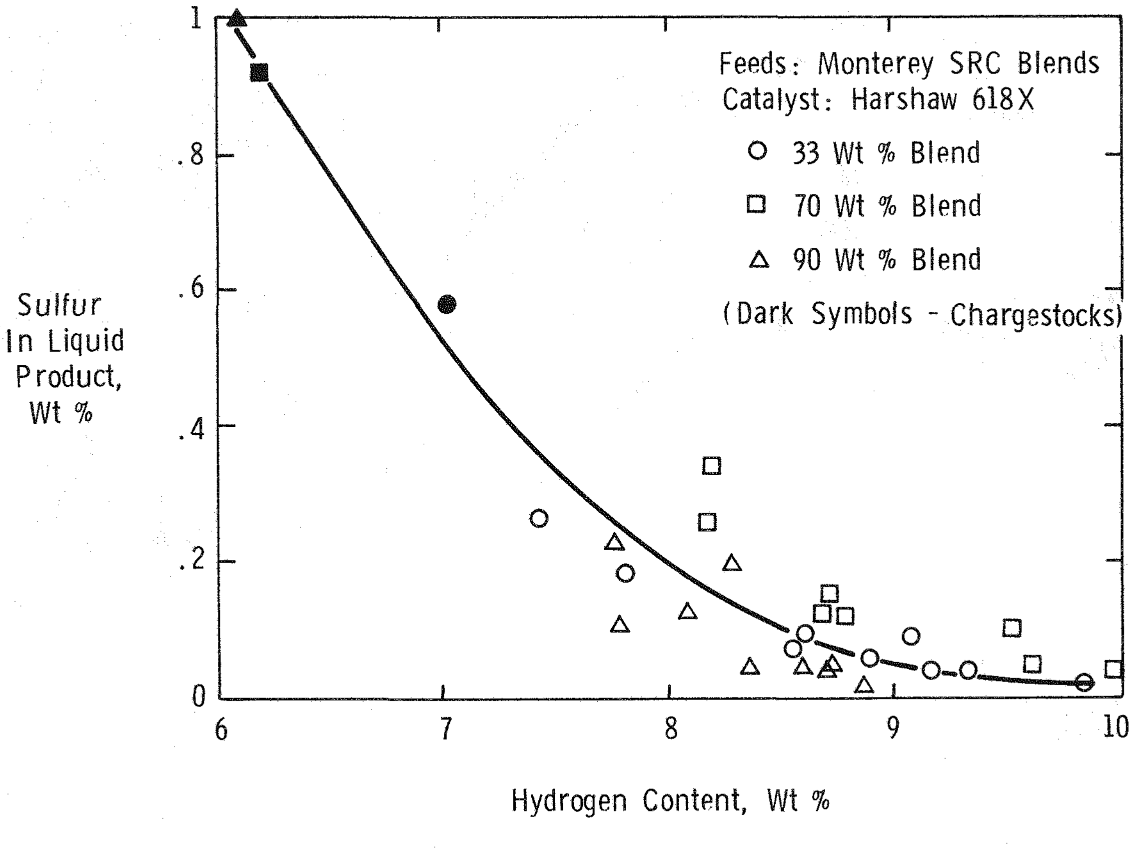


Figure 1-6

SULFUR CONTENT OF HYDROPROCESSED MONTEREY SRC BLENDS AS A FUNCTION OF HYDROGEN CONTENT



utilization distribution (C_1-C_5, C_6^+, etc.) for all blends is similar. Figure 1-7 shows that about 65% of the total hydrogen consumed goes to the C_6^+ liquid products. The 90% blend appears to put somewhat more hydrogen into the gaseous products and less into the C_6^+ liquid, due to higher temperatures required for equivalent desulfurization. However, little difference is seen between the different blends.

The heteroatoms (sulfur, nitrogen, and oxygen) are present both in SRC and recycle solvent. Consequently, heteroatom removal cannot provide an unambiguous indication of the interaction between SRC and recycle solvent. Conradson Carbon Residue (CCR) is present in the SRC only and correlates well with resins and asphaltenes in the blends; consequently, CCR should give a good indication of the effect of SRC concentration on the upgrading. Figure 1-8 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 1-9 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. Figure 1-8 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.

Figure 1-9 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.

Effect of Coal Source

As shown in Table 1-4, sulfur content is the major difference among regular SRC's derived from different coals; the sulfur content is directly related to the organic sulfur in the coals. However, an important factor with respect to processability is the SRC's molecular weight distribution. We have observed that the GEC fractions

Figure 1-7

HYDROGEN UTILIZATION IN HYDROPROCESSING OF MONTEREY SRC

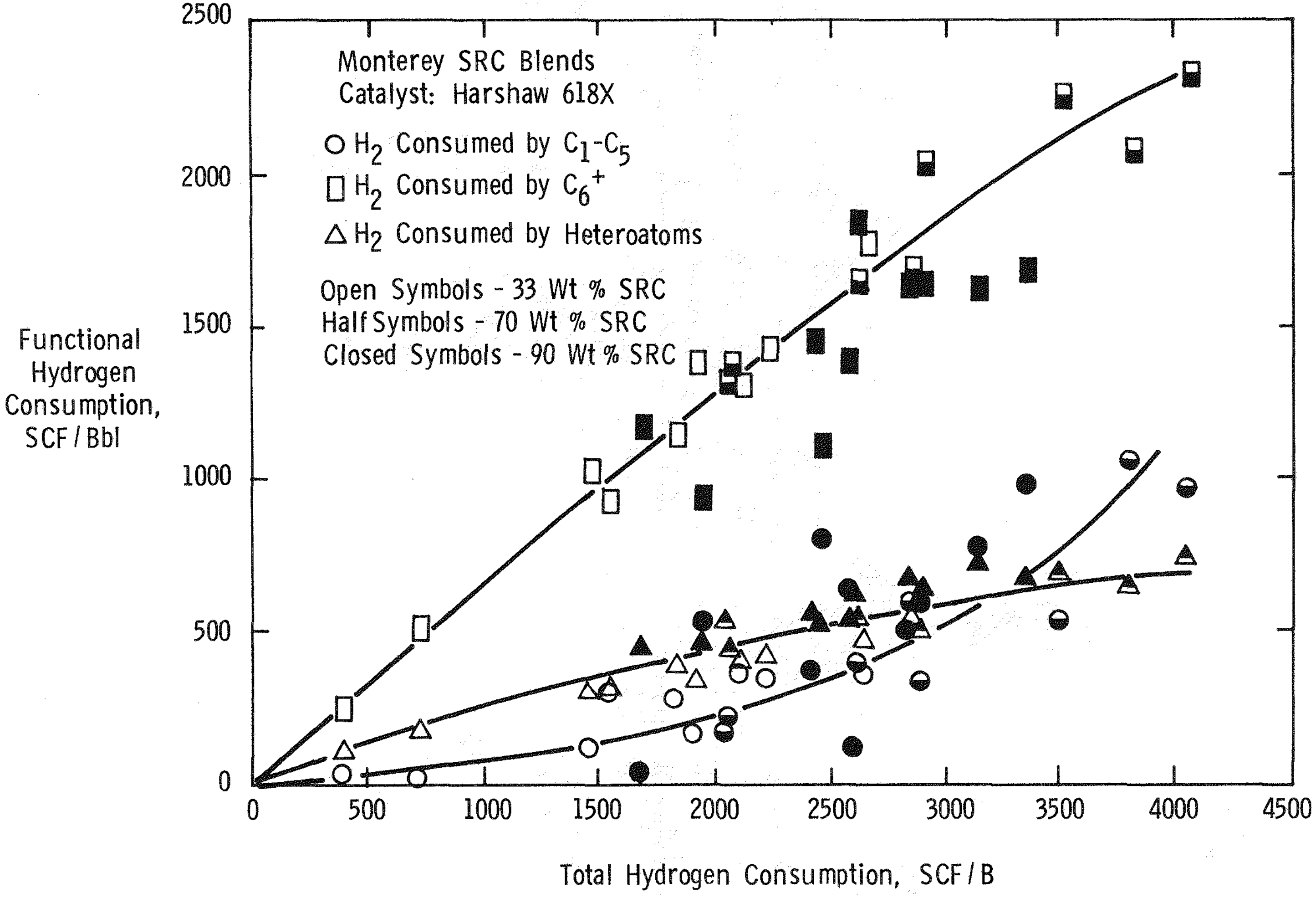


Figure 1-8

CCR CONVERSION FOR MONTEREY SRC BLENDS AT EQUIVALENT OVERALL LHSVs

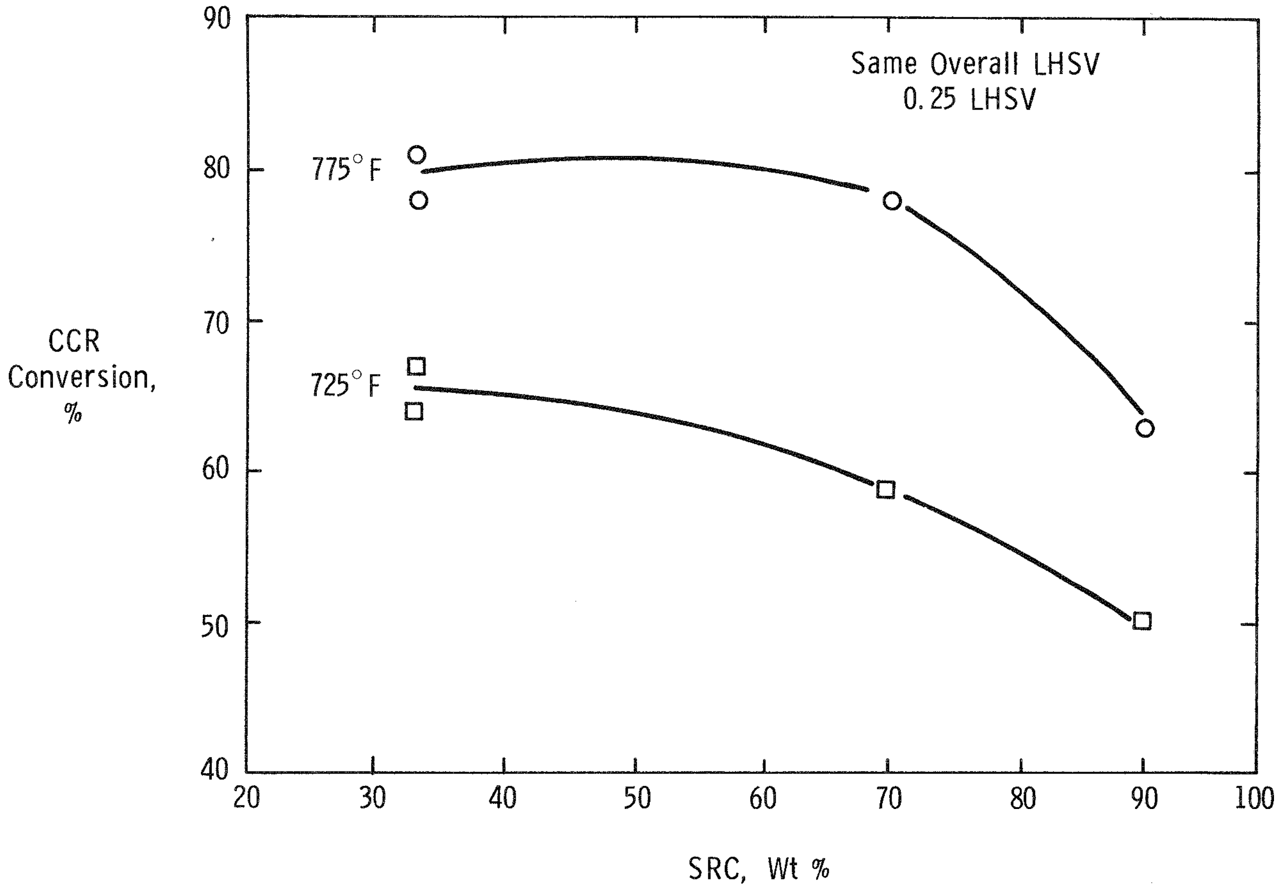
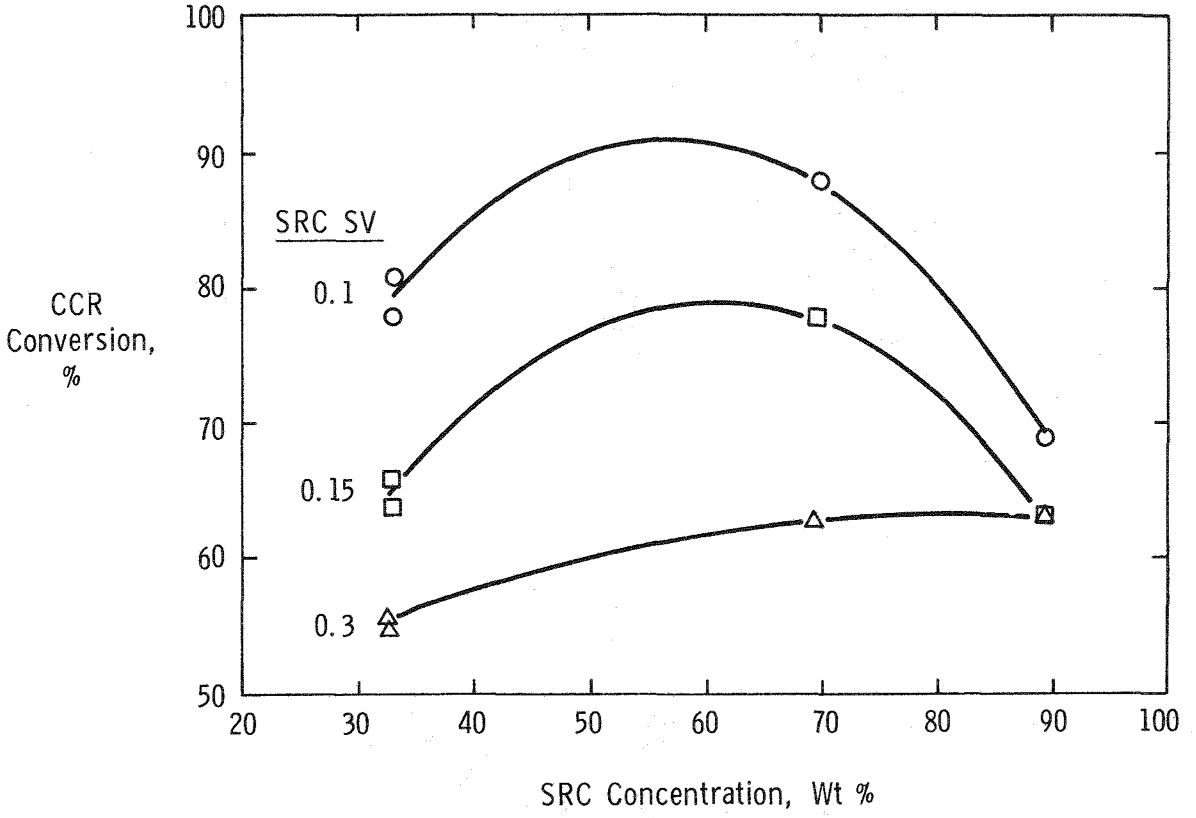


Figure 1-9

CCR CONVERSION FOR MONTEREY SRC BLENDS
AT EQUIVALENT LHSVs ON SRC



from the Wyodak and Monterey samples were 20-40% higher in molecular weight than the corresponding fractions of the Burning Star sample. The compositions of 33% SRC blends from Burning Star, Monterey, and Wyodak SRC's are given in Table 1-6.

Table 1-7 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.

The CCR in the SRC blends can be attributed to the SRC fraction alone, and its reduction is a good indication of SRC conversion. Since CCR is a measure of coke precursors, it is a useful parameter in hydroprocessing. Figure 1-10 shows that the CCR can be correlated with the asphaltenes and resins in the SRC blends for the three SRC's. 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 higher coking tendencies.

The molecular size (or molecular weight) of SRC becomes less important when hydroprocessed over large pore catalysts. In Table 1-8, a comparison is made for the hydroprocessing of 70% Burning Star and Monterey SRC's over a large pore catalyst, Harshaw 618X, at similar conditions. Although the percent removal of heteroatoms and CCR is similar, the 70% Monterey SRC consumes more hydrogen. In the 33% blends (Table 1-7), the total hydrogen consumption of Monterey SRC is less than that of Burning Star SRC.

SRC Conversion Sequence

In order to establish the sequence of SRC conversion, raw and hydrotreated SRC's were separated into IBP-650°F boiling range fraction, aromatic oils, resins, and asphaltenes by distillation and gradient elution chromatography (GEC). Elemental and GEC analyses are given in Table 1-9 for several products from a run in which 33 wt % Monterey SRC blends were hydroprocessed over a small pore CoMo/Al_2O_3 catalyst, HDS-1441A. 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 become 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

TABLE 1-6

COMPOSITION OF 33 *WT °/° SRC BLENDS*

	BURNING STAR	*MONTEREY*	*WYODAK*
PROPERTIES			
GRAVITY, API	-3.0	-2.7	-3.4
HYDROGEN, WT °/°	6.84	6.93	6.51
SULFUR, ''	0.47	0.57	0.30
NITROGEN, ''	1.03	1.00	0.99
OXYGEN, ''	3.91	4.10	3.70
CCR, ''	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 8-13)	24.78	26.09	26.12

* *CUT AT* 500°*F INSTEAD OF* 650°*F*

TABLE 1-7

COMPARISON OF HYDROPROCESSING 33 *WT* °/° *SRC BLENDS OVER HDS*-1441*A*

	BURNING STAR		*MONTEREY*		*WYODAK*	
	199-441	199-447	199-578	199-582	146-773	146-779
OPERATING CONDITIONS						
TEMPERATURE, °*F*	731	729	729	727	731	729
PRESSURE, PSIG	2000	2000	2000	2000	2000	2000
LHSV, VO/HR/VCAT	0.98	0.21	0.99	0.22	0.98	0.18
H2 CONSUMPTIONS, SCF/B	1986	2822	1047	2497	1138	3076
LIQUID PRODUCT PROPERTIES						
GRAVITY, API	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, ''	0.58	0.22	0.74	0.34	0.66	0.27
OXYGEN, ''	0.70	0.20	1.60	1.00	1.60	0.30
SULFUR, ''	0.11	0.06	0.17	0.09	0.05	0.02
CCR, ''	8.64	4.30	12.88	6.29	14.65	8.88
HETEROATOM REMOVAL, °/°						
SULFUR	73	85	71	85	83	95
OXYGEN	83	95	62	77	58	92
NITROGEN	46	79	28	67	35	74

TABLE 1-8

COMPARISON OF HYDROPROCESSING 70 WT °/° BURNING STAR AND MONTEREY REGULAR SRC'S OVER HARSHAW 618X

	70 °/° BURNING STAR SRC		70 °/° MONTEREY SRC	
	CHARGE	146-882	CHARGE	146-858
OPERATING CONDITIONS				
TEMPERATURE, °F	-	773	-	781
PRESSURE, PSIG	-	2000	-	2000
LHSV, VO/HR/VCAT	-	0.43	-	0.41
H2 CONSUMPTIONS, SCF/B	-	1750	-	2600
LIQUID PRODUCT PROPERTIES				
GRAVITY, API	-16.4	-3.3	-13.4	5.2
HYDROGEN, WT °/°	6.25	7.79	6.15	8.67
SULFUR, "	0.53	0.09	0.92	0.12
NITROGEN, "	1.66	0.94	1.65	0.79
OXYGEN, "	3.40	0.90	5.10	1.60
CCR, "	45.00	23.00	38.20	16.90
HETEROATOM REMOVAL, °/°				
SULFUR	-	83	-	88
OXYGEN	-	75	-	71
NITROGEN	-	46	-	55
CCR	-	51	-	59

TABLE 1-9

ELEMENTAL AND GEC ANALYSES OF HYDROPROCESSED 33°/° MONTEREY SRC BLENDS
CATALYST: HDS-1441A
PROGRAM: 2359   UNIT: 199

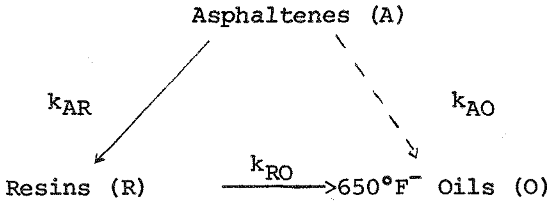
	CHARGE	MB-574	MB-575	MB-576	MB-577	MB-578	MB-580	MB-581	MB-582	MB-584
OPERATING CONDITIONS										
TEMPERATURE, °F	-	672	723	780	672	729	775	675	727	777
LHSV, VFF/HR/VCAT	-	2000	2000	2000	2000	2000	2000	2000	2000	2000
PRESSURE, PSIG	-	0.99	0.96	0.82	0.57	0.99	0.48	0.24	0.22	0.10
H2 CONSUMPTIONS, SCF/B	-	726	1047	1183	1085	1377	1946	1289	2490	3924
TLP YIELDS, WT PCT										
650°F-	51.21	62.38	59.32	59.74	62.82	55.85	72.59	55.34	67.78	89.78
SATURATES (CUT 1)	0.26	0.23	0.48	0.74	0.28	0.65	0.48	0.63	0.60	1.12
AROM. OILS (CUT 2-4)	9.51	8.48	17.11	22.54	9.40	17.40	13.70	17.33	15.40	7.63
RESINS/ASPH. (CUT 5-7)	12.93	15.60	14.57	12.75	14.77	15.01	9.35	14.78	10.63	0.81
POLAR ASPH. (CUT 8-13)	26.09	13.31	8.52	4.23	12.73	11.09	3.88	11.92	5.59	0.66
TOTAL	100	100	100	100	100	100	100	100	100	100
ELEMENTAL ANALYSES(A)										
IBP-650°F										
HYDROGEN, WT PCT	7.66	8.57	9.14	8.83	8.75	9.95	9.61	9.34	10.86	11.14
OXYGEN, WT PCT	3.8	2.6	2.2	1.2	1.9	1.0	0.3	2.0	0.9	0.3
NITROGEN, WT PCT	0.63	0.42	0.35	0.26	0.36	0.27	0.15	0.22	0.09	0.07
SULFUR, WT PCT	0.250	0.085	-	0.024	0.042	0.016	0.011	0.034	0.011	0.011
650°F+ BTMS										
HYDROGEN, WT PCT	6.16	6.50	6.60	6.89	6.73	6.60	7.14	7.12	7.50	8.47
OXYGEN, WT PCT	4.4	4.0	2.0	1.2	2.7	2.4	1.4	1.8	1.2	0.6
NITROGEN, WT PCT	1.39	1.65	1.38	1.05	1.57	1.33	0.98	1.21	0.87	0.20
SULFUR, WT PCT	0.91	0.47	-	1.26	0.46	0.37	0.13	0.25	0.24	0.03

(A) FOR EACH SAMPLE THE ELEMENTS WERE DETERMINED DIRECTLY FOR ONE DISTILLATE CUT AND CALCULATED BY DIFFERENCE FOR THE OTHER CUT.

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 conversion selectivity for the Monterey SRC is shown in Figure 1-11. The hydrogen content of the liquid product is used as an index of hydroprocessing severity. Figure 1-11 shows that polar and non-eluted 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 ("650°F$^-$ oils"). 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 rate constant for the conversion of asphaltenes directly to 650°F$^-$ oils, k_{AO}, is very close to zero. During the run (12 days on-stream), some deactivation was observed. To account for the deactivation, the rate constants were assumed to decay according to the equation:

$$k_{ij} = k^{o}_{ij} \exp\{-t/\tau\} \qquad (1\text{-}1)$$

where: t = days on-stream
τ = catalyst decay constant, days.

Integration of the first order equations yields the following expressions for the asphaltene and resin concentrations:

Figure 1-10

CCR AS A FUNCTION OF ASPHALTENES PLUS RESINS FOR HYDROPROCESSED SRCs

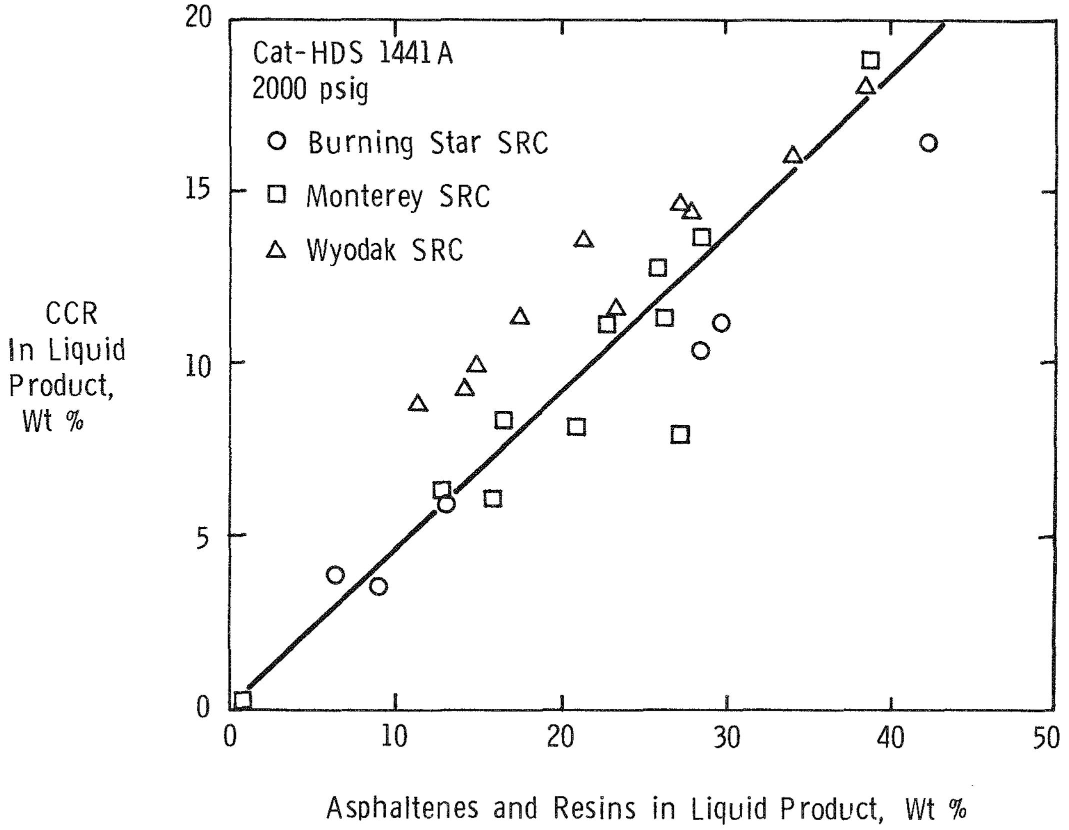
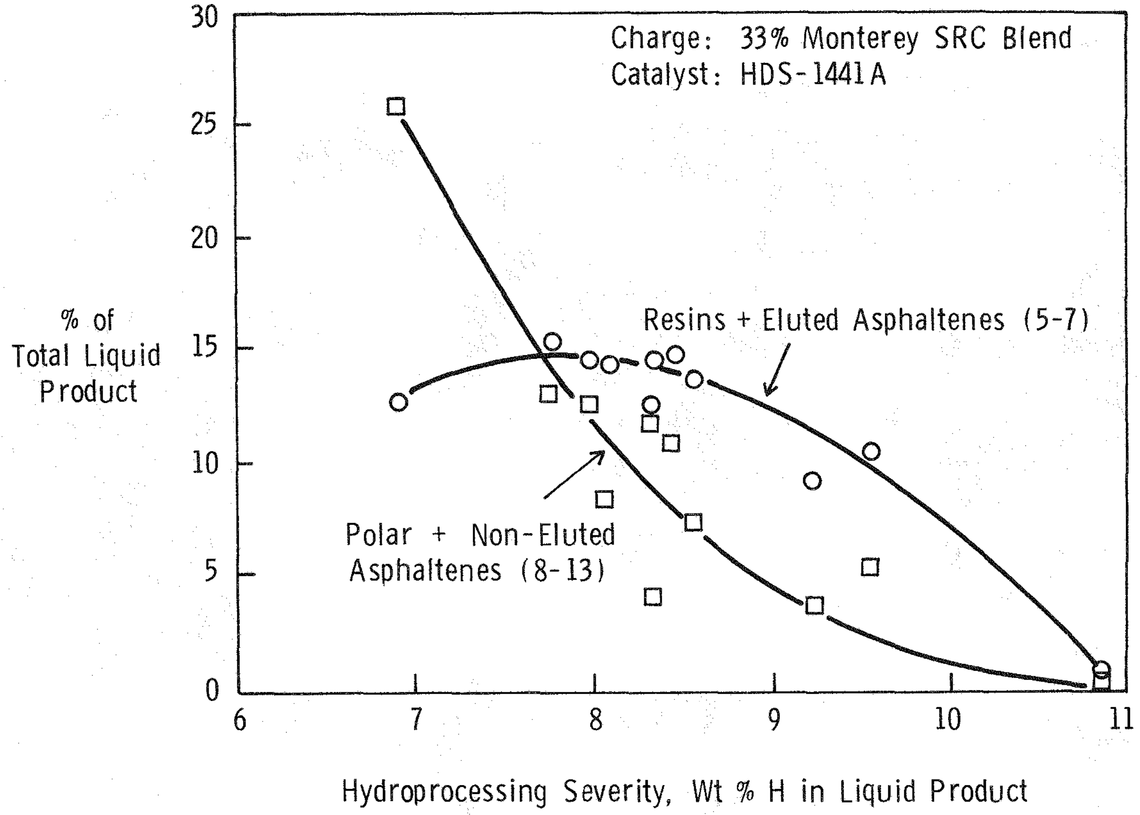


Figure 1-11

RESINS AND ASPHALTENES CONVERSION FOR 33% MONTEREY SRC BLEND



$$A = A_o \, e^{-k_{AR}/LHSV} \tag{1-2}$$

$$R = \frac{k_{AR}A_o}{k_{RO}-k_{AR}} \, e^{-k_{AR}/LHSV} + R_o - \frac{k_{AR}A_o}{k_{RO}-k_{AR}} \, e^{-k_{RO}/LHSV} \tag{1-3}$$

where the k's are a function of time as defined by equation (1-1).

The model fits the data well over the entire range of severity. The constants obtained from this fit are given in Table 1-10 below:

Table 1-10

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 (τ), DAYS
A ---> R	7.55×10^6	36,527	6.1
R ---> O	9.69×10^5	31,493	

$$^*k_{ij} = k^o_{ij} \, e^{-t/\tau} \, e^{-E_{ij}/RT}$$

Regular vs. SCT SRC

Coal can be solubilized at very short residence times in the SRC process. The short contact time (3-4 minutes) runs show considerably less hydrogen consumption and gas make than the regular SRC (40 minutes residence time). However, SCT SRC is higher in heteroatoms and polar asphaltenes (See Tables 1-4 and 1-11). Consequently, the viscosity of raw and hydrotreated SCT SRC's is significantly higher than that of regular SRC. Both SCT and regular SRC's show similar reactivity for heteroatom removal and GEC class conversion at equivalent conditions. Table 1-11 gives a direct comparison between upgrading 33% regular and SCT SRC's over Harshaw 618X. 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 hydrogen consumption is 700-800 scf/B greater for SCT SRC at similar process severities.

TABLE 1-11

COMPARISON OF HYDROPROCESSING REGULAR AND SCT SRC'S
LIQUID PRODUCT PROPERTIES AND GEC ANALYSES
CHARGE: 33 *WT °/° REGULAR AND SCT MONTEREY SRC BLENDS*
CATALYST: HARSHAW 618*X*

	REGULAR SRC		*SCT SRC*	
	CHARGE	*PRODUCT*	*CHARGE*	*PRODUCT*
OPERATING CONDITIONS				
TEMPERATURE, °F	-	777	-	782
LHSV, VFF/HR/VCAT	-	2000	-	2000
PRESSURE, PSIG	-	0.50	-	0.57
TLP PROPERTIES				
GRAVITY, API	-4.9	12.2	-5.9	12.1
HYDROGEN, WT PCT	7.0	9.2	6.5	10.0
OXYGEN, WT PCT	3.70	1.00	4.50	0.80
NITROGEN, WT PCT	1.12	0.32	1.14	0.30
SULFUR, WT PCT	0.58	0.04	1.01	0.07
CCR, WT PCT	17.1	6.2	16.8	5.4
K.V. (100°*C*), *CS*	13.4	1.9	416.0	2.2
GEC ANALYSIS, WT°/°				
650°*F-*	48.3	60.7	49.0	66.2
SATURATES (*CUT* 1)	0.4	1.4	0.3	2.1
AROM. OILS (*CUT* 2-4)	10.9	24.9	6.0	19.7
RESINS/ASPH. (*CUT* 5-7)	15.9	9.9	8.4	8.2
POLAR ASPH. (*CUT* 8-13)	24.5	3.1	36.3	3.8

The similarity between SCT and regular SRC is also shown in the functional conversion mechanism based on GEC analyses. Figures 1-12 and 1-13 show the change in GEC composition as a function of hydroprocessing severity. 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.

Kerr McGee CSD SRC

The processability of a coal liquid can, in principle, be improved by selectively removing the most hard-to-convert material. The Kerr-McGee Critical Solvent Deashing (K-M CSD) Process is a combination deashing/fractionation operation now being tested on solvent refined coal. Compared to a conventionally filtered Indiana V SRC, the K-M CSD SRC was lower in CCR content as well as lower in polar and non-eluted asphaltenes, as shown by GEC analyses. Moreover, the Kerr-McGee SRC was significantly more reactive for desulfurization, denitrogenation, and deoxygenation. In Table 1-12 the process conditions are shown for hydroprocessing K-M CSD SRC and conventionally filtered SRC to the same level of desulfurization.

Table 1-12

REACTIVITY COMPARISON OF TWO INDIANA V REGULAR SRC'S:
KERR-McGEE CSD SRC AND FILTERED SRC
(CATALYST: HARSHAW 618X)
T = 775°F; P = 2000 psig

	K-M CSD SRC	Filtered SRC
LHSV	1.71	0.58
% S Removal	73.6	70.3
% N Removal	28.9	23.1

The Kerr-McGee SRC can be desulfurized at three times the space velocity required for conventionally filtered SRC. This reduction in capital costs should significantly improve the economics of upgrading SRC to low sulfur boiler fuel.

1.4 CATALYST EVALUATION FOR UPGRADING HIGH SRC CONCENTRATION LIQUIDS

Fresh Catalyst Activity

A series of catalysts was evaluated for the upgrading of SRC/process recycle

Figure 1-12

RESINS AND ELUTED ASPHALTENES IN MONTEREY SRC BLENDS AS A FUNCTION OF HYDROPROCESSING SEVERITY

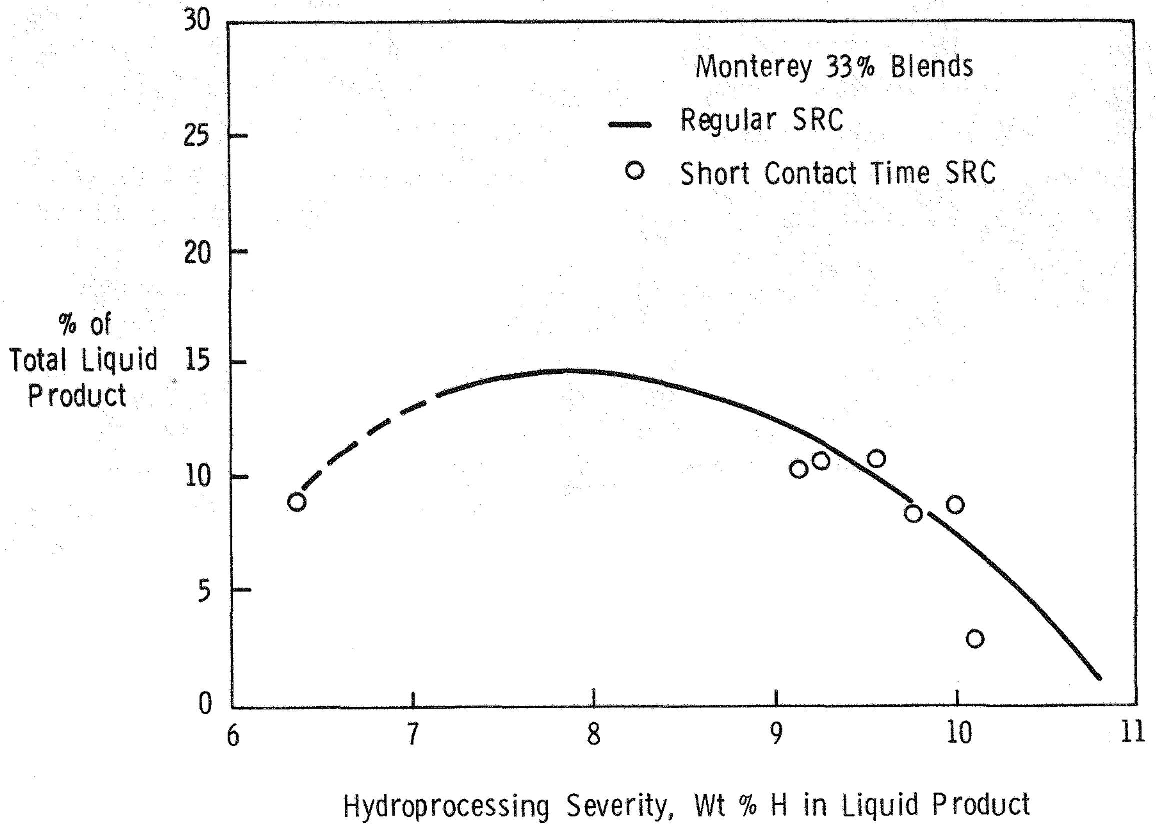
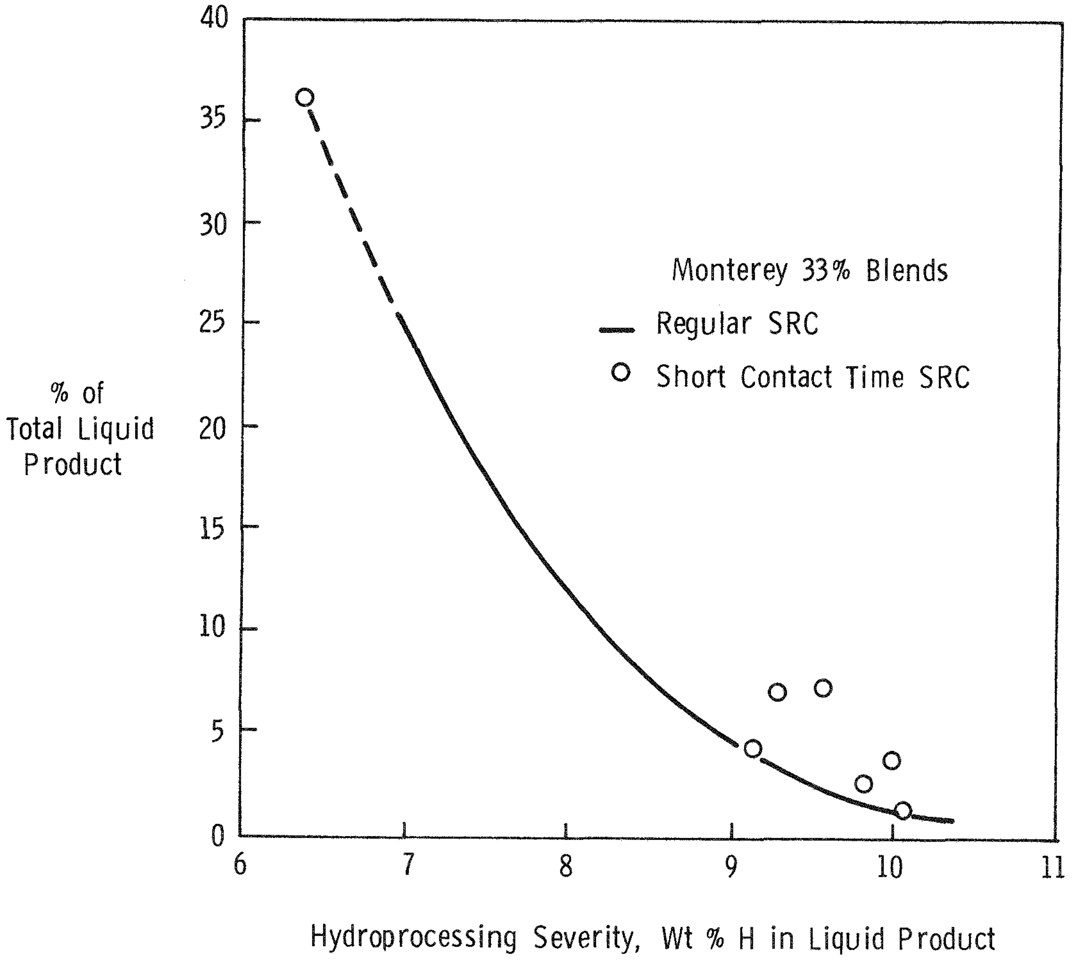


Figure 1-13

POLAR AND NON-ELUTED ASPHALTENES IN MONTEREY SRC BLENDS AS A FUNCTION OF HYDROPROCESSING SEVERITY



solvents blends to make low sulfur boiler fuel. In the catalytic hydrodesulfurization of SRC, hydrogen cost is a major economic factor. Consequently, a good catalyst must have efficient hydrogen utilization as well as have good desulfurization activity.

Nine catalysts were evaluated in our pilot unit, including three Mobil proprietary catalysts (HCL-1, HCL-2, HCL-3), two developmental catalysts (Amocat 1A and Amocat 1B), and four commercially available catalysts (American Cyanamid's HDN-1197 and HDS-1443 and Harshaw's 618X and HT-500). Harshaw 618X, a commercial large pore catalyst, previously found to be active for upgrading SRC/recycle solvents blends, was used as a reference catalyst. Charge stocks used in the catalyst evaluation were 50/50 blends of W. Kentucky SCT SRC and process recycle solvent. All catalysts had previously been found to be active either for hydrotreating petroleum feedstocks or for coal hydroliquefaction processes.

The nine catalysts have been divided into two groups in order to simplify the activity comparisons. Group A is made up of the more active desulfurization catalysts and includes HCL-2, HCL-3, HDS-1443, and Amocat 1A. Group B includes HCL-1, HT-500, 618X, HDN-1197, and Amocat 1B. For the activity comparisons, the sulfur removal and CCR reduction are plotted versus reactor temperature at liquid hourly space velocities of 0.5. Consequently, catalyst activity can be compared on the basis of temperature requirements for achieving specific liquid product sulfur or CCR content. The results are shown in Figures 1-14 and 1-15 for desulfurization and Figures 1-16 and 1-17 for CCR reduction, respectively. The results show:

- Mobil HCL-2 is the most active catalyst for desulfurization. It is 35°F more active than Harshaw 618X and 7°F more active than HDS-1443, the second most active catalyst.
- Mobil HCL-3 is the most active catalyst for CCR reduction. It is 40°F more active than Harshaw 618X and 10°F more active than HDS-1443.

In Figures 1-18 and 1-19, the total hydrogen consumption is plotted as a function of sulfur concentration in the total liquid products for Group A and Group B catalysts, respectively. Mobil HCL-2 and Amocat 1A are low hydrogen consumption catalysts, compared to Harshaw 618X; each uses 30-40% less hydrogen than Harshaw 618X catalyst to achieve a specific sulfur level in the liquid products. Amocat 1B (Mo/Al_2O_3), having low desulfurization activity, uses 50% more hydrogen than Harshaw 618X. HDS-1443 also seems to be low hydrogen consumption catalyst. The hydrogen consumption with Mobil HCL-1 is intermediate to Mobil HCL-3 and Harshaw 618X. Other catalysts, such as HT-500, Mobil HCL-3 and HDN-1197, showed similar hydrogen consumption/desulfurization selectivity as Harshaw 618X.

Catalyst Aging Tests

In order to establish process design conditions for the upgrading of SRC to low sulfur boiler fuel, constant temperature aging runs were made with 70% Indiana V regular SRC (over Mobil HCL-2 catalyst) and with 50% W. Kentucky SCT SRC (over Amocat 1A catalyst). Based on these runs, reactor operating conditions were estimated for both fixed bed and ebullated bed reactors. Typical fixed bed operating conditions are shown in Figure 1-20 for producing 0.4 wt % S in the 900°F$^+$ fraction (0.3 wt % S in the TLP) from Indiana V SRC. The cycle length, a function of both LHSV and the limit of reactor temperature, is 30 days at 0.3 LHSV and a reactor limit of 850°F.

For the operation of an ebullated bed reactor, the reactor temperature is kept essentially constant; catalyst activity is maintained by the continuous addition of fresh catalyst along with removal of similar amounts of aged catalyst. For processing 70% Indiana V regular SRC in an ebullated bed reactor (800°F; 0.5 SRC WHSV; 2000 psig), it is estimated that a catalyst make-up rate of up to 8 pounds per ton of SRC would be needed to produce a 900°F$^+$ fuel of 0.4 wt % S. This catalyst requirement can be significantly reduced by the use of multiple reactors, enabling the process to approach plug flow kinetics. Moreover, the translation of fixed bed data to ebullated bed operations may yield somewhat conservative estimates of catalyst make-up rates. Confirmation runs in an ebullated bed reactor are required to establish the actual catalyst make-up rates.

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2. P. W. Pillsbury, P. P. Singh, A. Cohn, T. R. Stein, and P. R. Mulik, Paper 79-GT-137, presented at the Gas Turbine Conference and Exhibit and Solar Energy Conference, San Diego, California, March 11-15, 1979.

Figure 1-14

COMPARISON OF DESULFURIZATION ACTIVITIES: GROUP A CATALYSTS

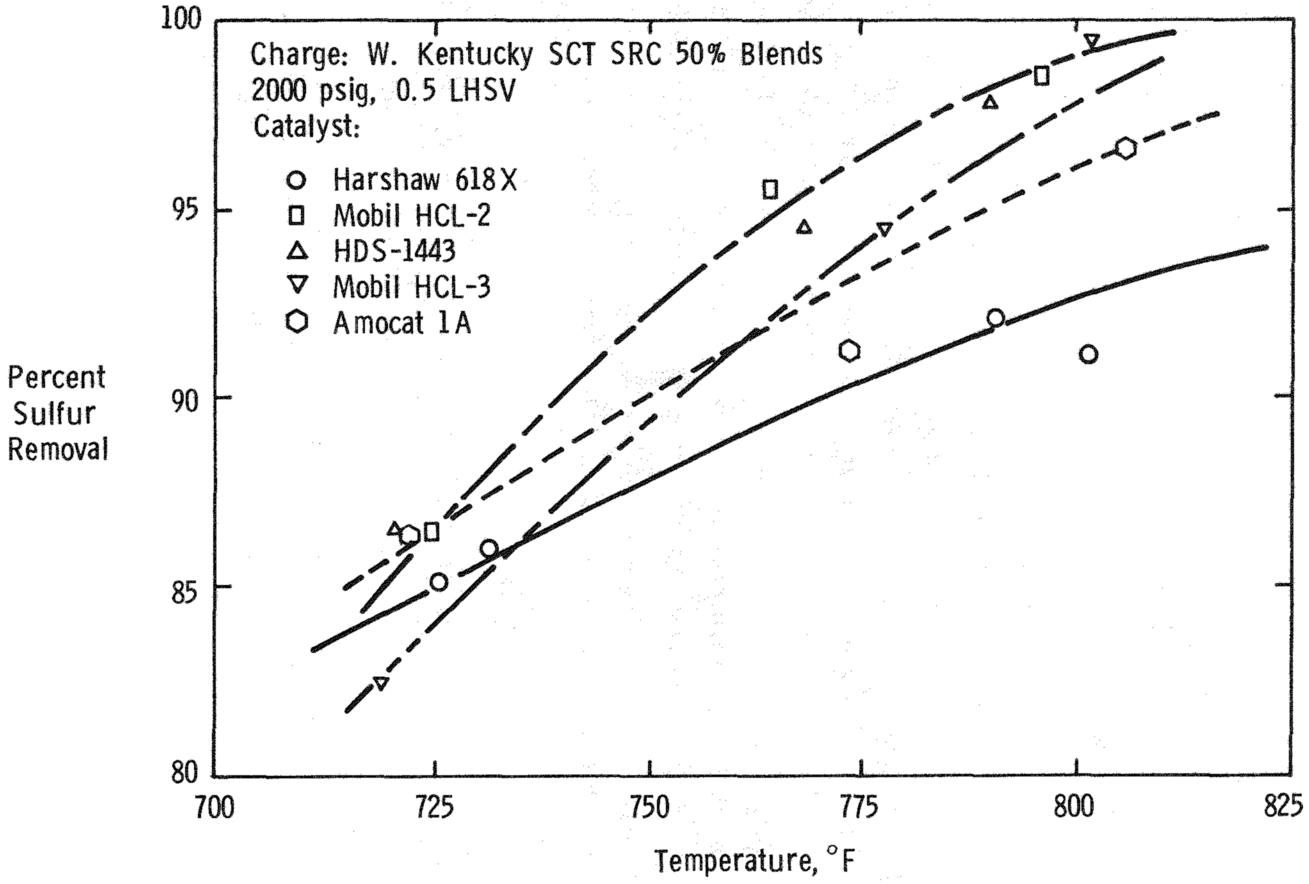


Figure 1-15

COMPARISON OF DESULFURIZATION ACTIVITIES: GROUP B CATALYSTS

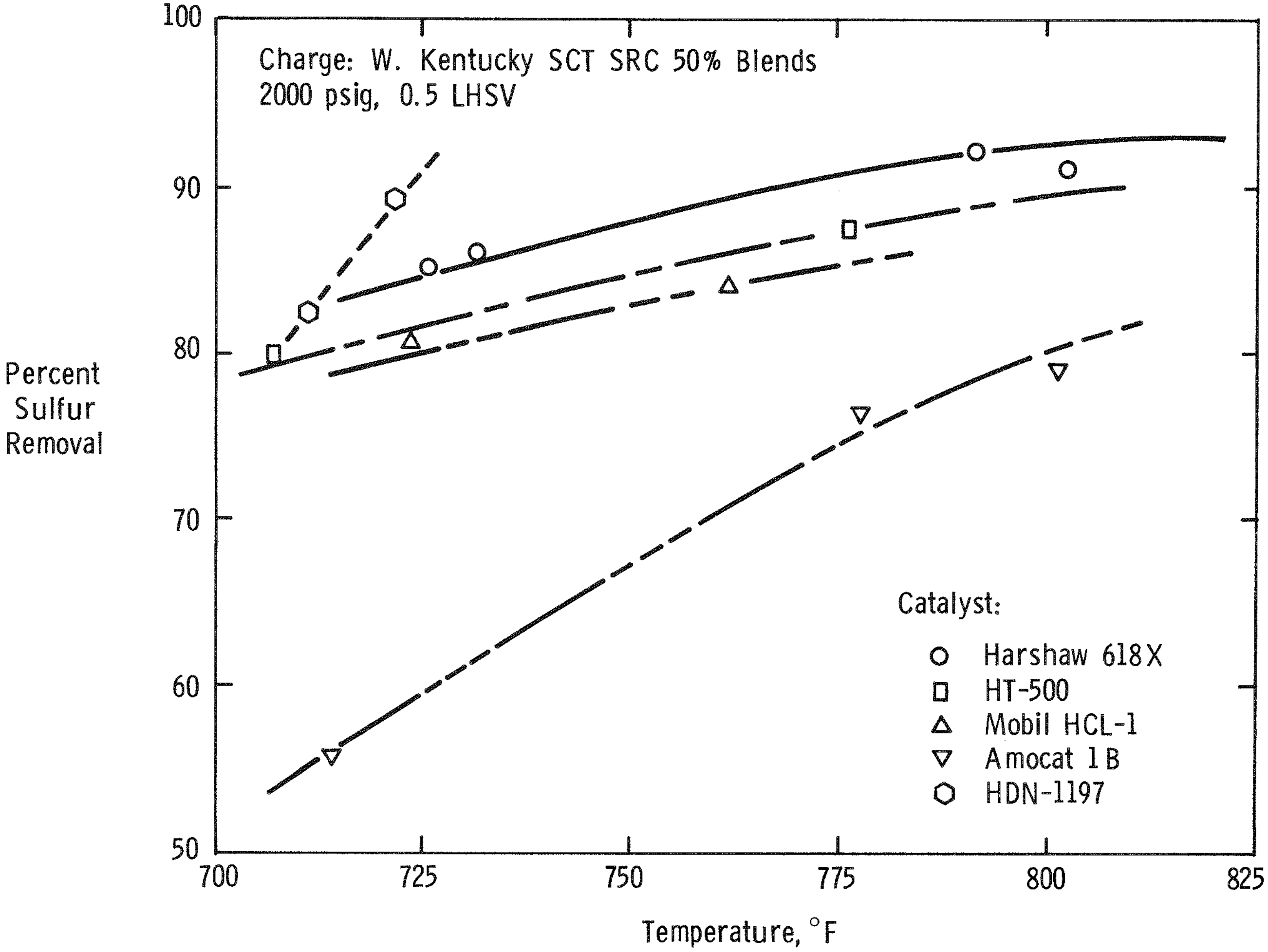


Figure 1-16

COMPARISON OF CCR REDUCTION ACTIVITIES: GROUP A CATALYSTS

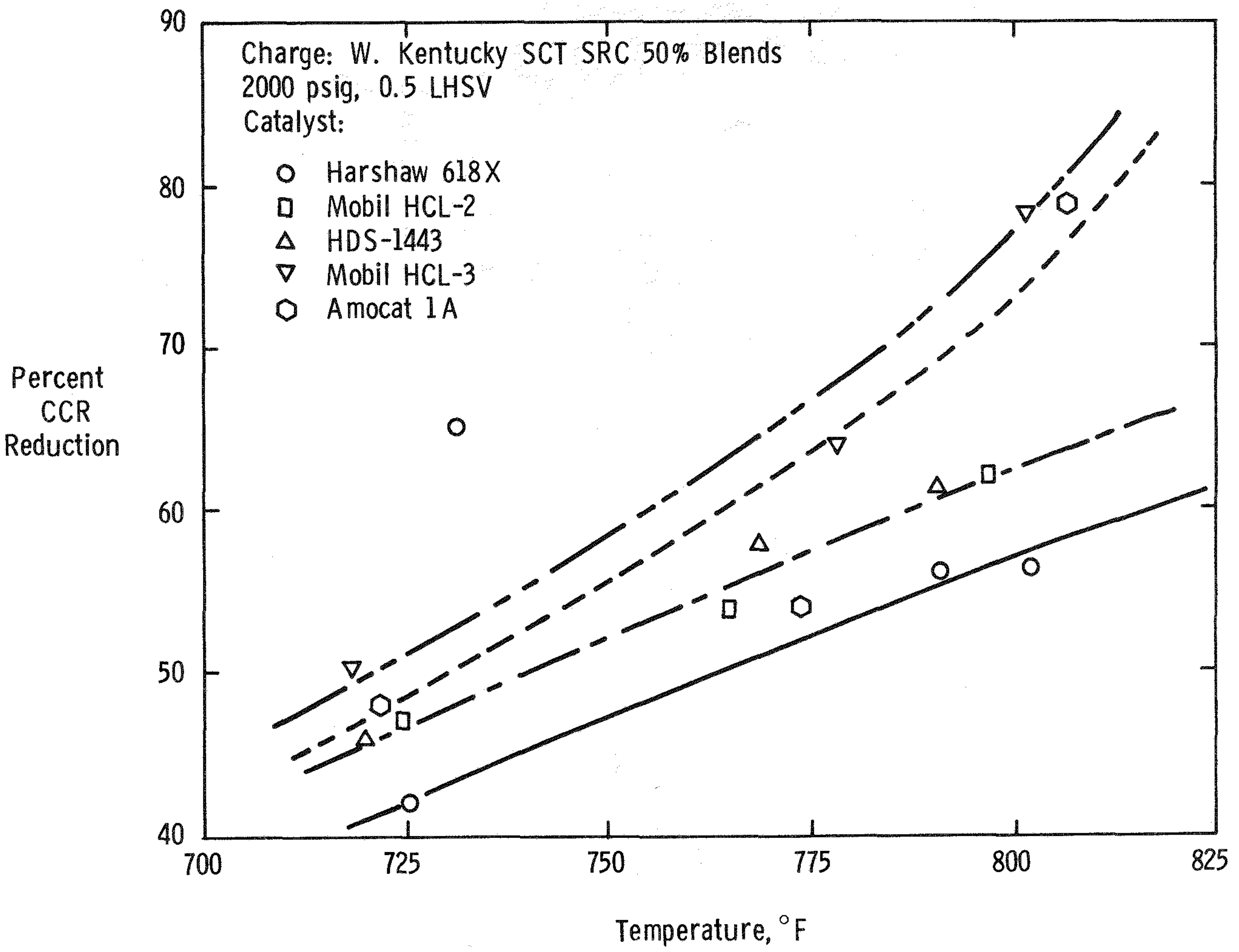


Figure 1-17

COMPARISON OF CCR REDUCTION ACTIVITIES: GROUP B CATALYSTS

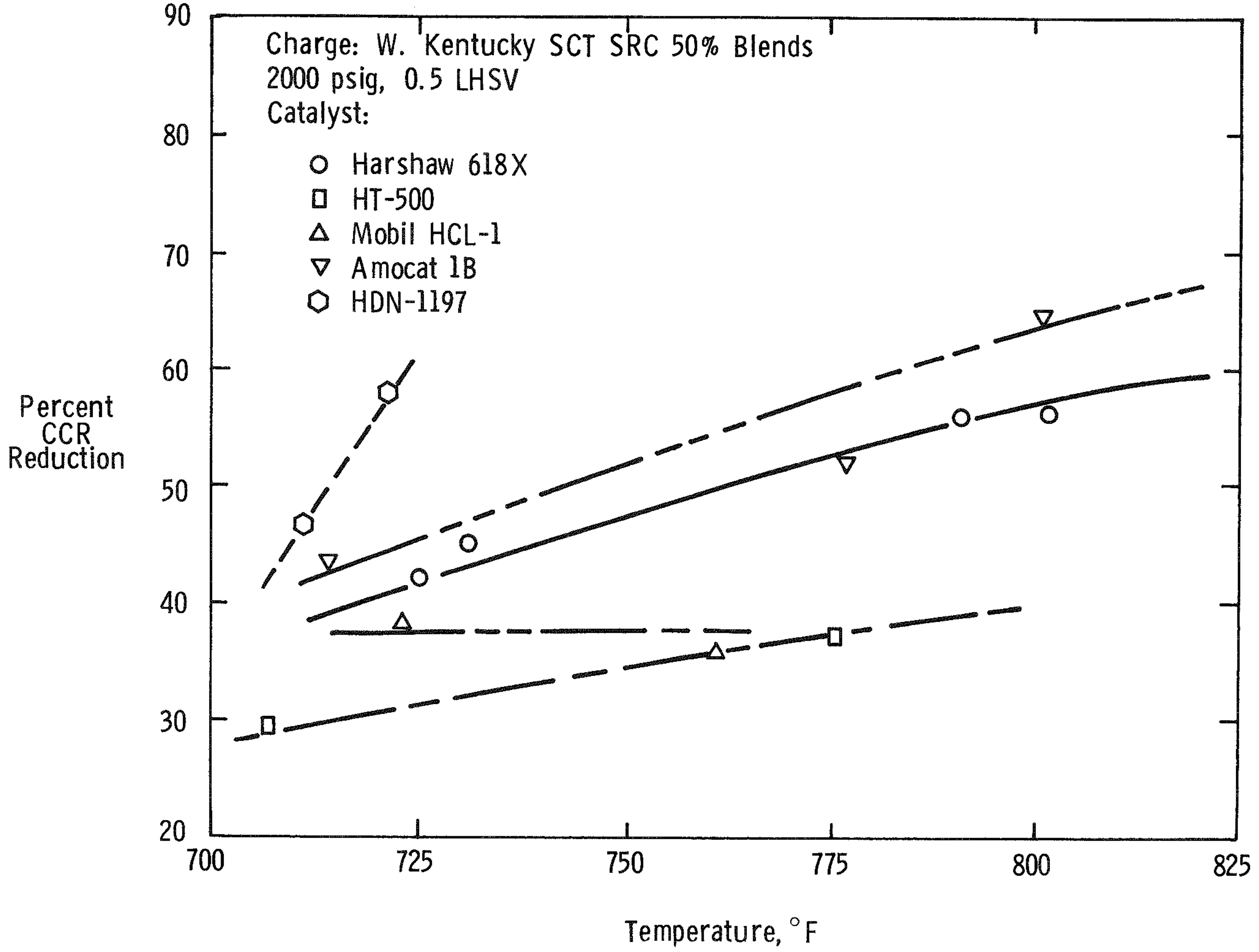


Figure 1-18

TOTAL HYDROGEN CONSUMPTION AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP A CATALYSTS

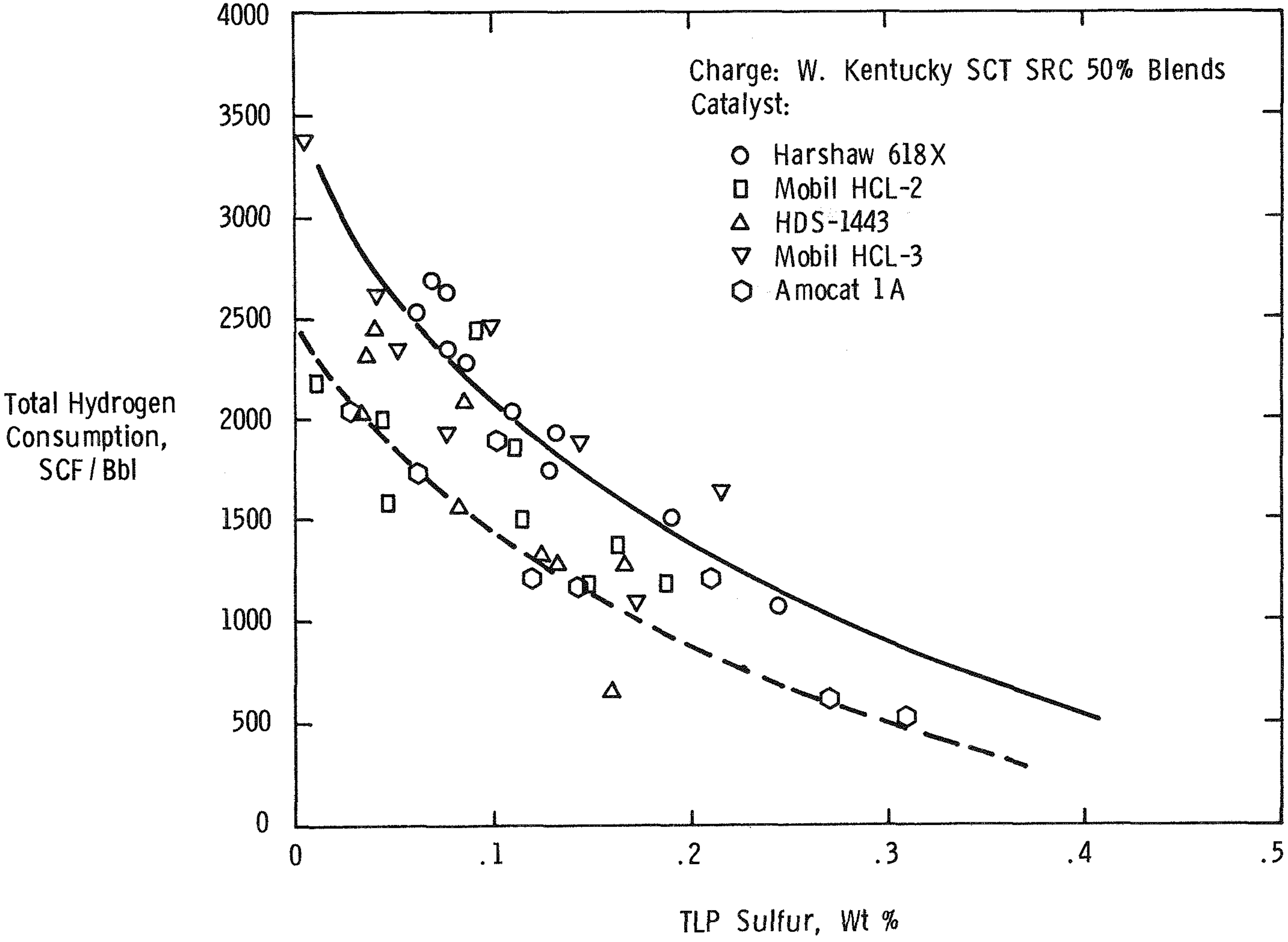


Figure 1-19

TOTAL HYDROGEN CONSUMPTION AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP B CATALYSTS

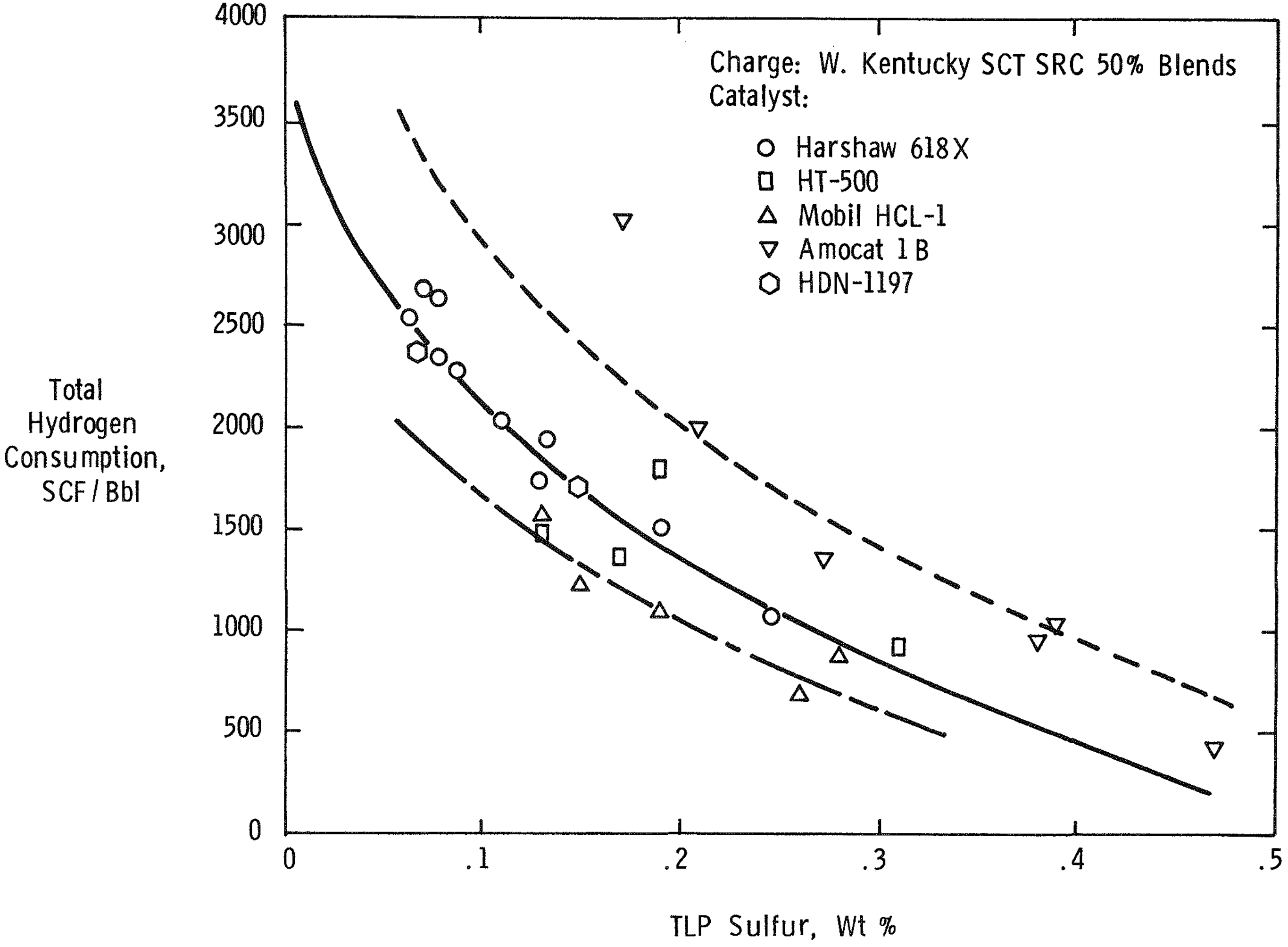
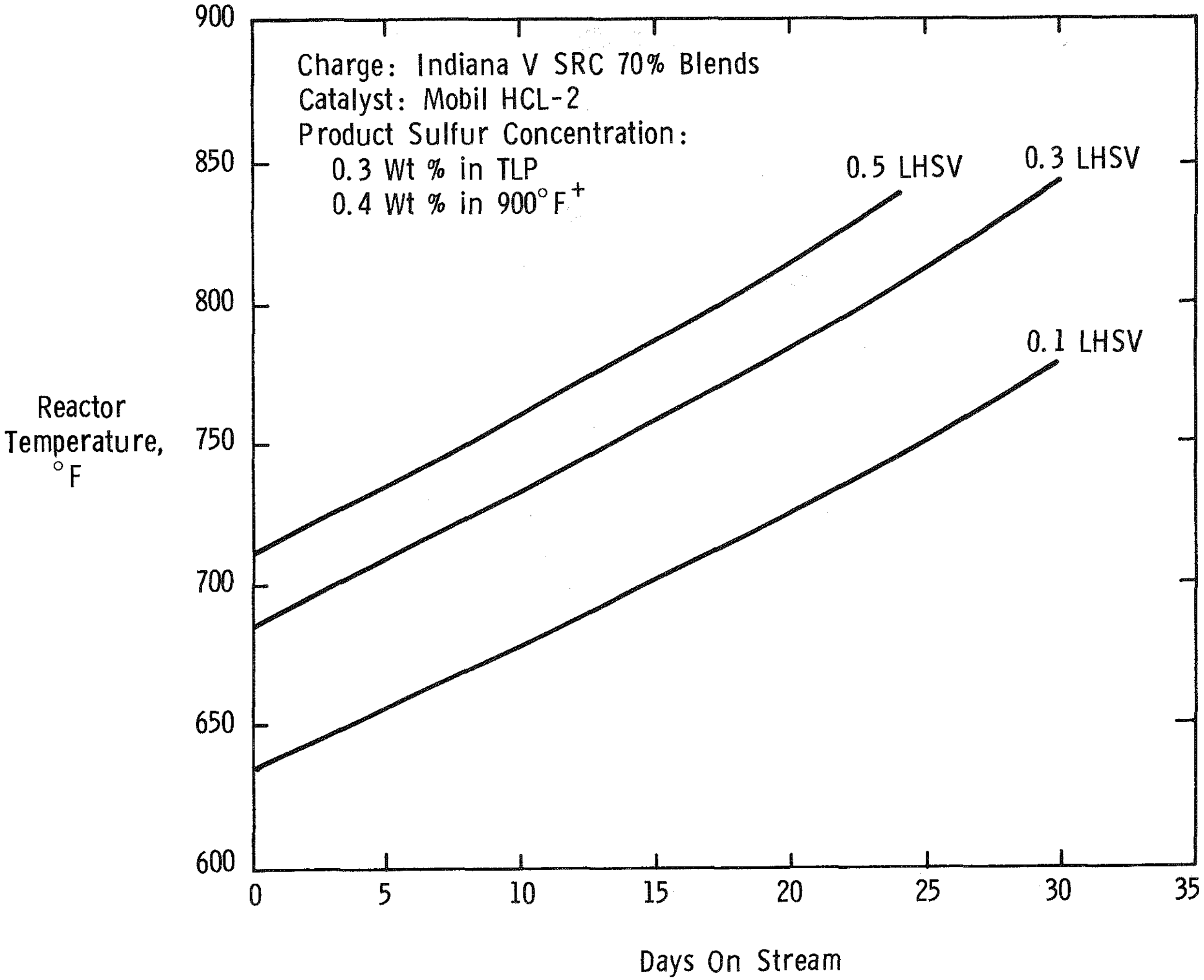


Figure 1-20

DESIGN VARIABLES FOR FIXED BED REACTOR



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 combustion turbine fuels. Presently, combustion 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, combustion 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, combustion 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 combustion turbine fuels. Results from this study were reported in the Phase I Report under EPRI Project 361-1. It was found that products from current coal liquefaction processes require considerable upgrading to meet current specifications for combustion 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 combustion turbines. Based on C^{13}-NMR and Gradient Elution Chromatography analyses, the higher boiling 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 higher boiling 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 combustion 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 combustion turbine fuel. The objective of this project was 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 was 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. The objective of the work carried out under a one-year extension of RP 361-2 was to evaluate a series of catalysts for the hydroprocessing of heavy coal liquefaction fractions and apply the most promising to the upgrading of these high boiling materials to low sulfur boiler fuels.

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 that these heavy coal liquids 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.

During the second year a major area of research was the processing of distillate coal liquids to high quality power generation fuels. The distillates were obtained from the H-Coal Process Development Unit and from the Wilsonville, Alabama SRC pilot plant. High heteroatom removal and low catalyst aging rates were achieved, but hydrogen consumption was as high as 2000 scf/B. The raw and hydrotreated H-Coal distillate products, along with SRC recycle solvent, were evaluated by Westinghouse Research in their small scale combustor. The raw H-Coal distillate, although significantly better than the SRC recycle solvent, was inferior to the No. 2 petroleum fuel oil used for reference. The hydrotreated H-Coal distillate was nearly equivalent in combustor performance to No. 2 fuel oil.

A second area of study was the upgrading of regular and short contact time (SCT) SRC to low sulfur boiler fuels. Blends of SRC in process derived recycle solvent containing as much as 90% SRC were processed in our unit, specifically modified to handle these viscous stocks. A kinetic model was developed linking the rates of conversion and heteroatom removal to the process conditions.

This report covers the work completed during the third and final year of this contract, February 1978 through July 1979. A series of catalysts was evaluated in our fixed bed pilot unit for upgrading SCT SRC to low sulfur fuel oil. Short contact time SRC, being more viscous than regular SRC, could be processed at SRC concentrations only up to 50 wt %. The objective of this work was to identify high desulfurization activity catalysts which efficiently utilize hydrogen. Six commercial and three proprietary catalysts were tested. The commercial catalysts, with various promoters and support pore size distributions, were selected because of their known uses in petroleum desulfurization or coal hydroliquefaction. Catalyst aging studies were carried out with 70% regular SRC as well as 50% SCT SRC. Design variables of both fixed bed and ebullated bed reactors were predicted from a kinetic aging model.

Elemental, physical, and chemical characterization was made for a wide range of SRC and H-Coal products. The composition of short contact time SRC recycle solvent, along with solvent generated from the hydroprocessing of SCT SRC/recycle solvent blends, were studied in order to compare their potential use in short contact time processes. Alkali metal contamination in H-Coal distillates was also investigated, and the feasibility of several separation methods was examined. A calorimetric analysis was made of several coal-derived liquids, and a heating value correlation was subsequently developed based on elemental composition.

Section 3

HYDROPROCESSING OF HEAVY COAL LIQUIDS

3.1 CATALYST EVALUATION

During this year a series of catalysts was evaluated for the upgrading of SRC/ process recycle solvent blends to make low sulfur boiler fuel. In the catalytic hydrodesulfurization of SRC, hydrogen cost is a major economic factor. Consequently, a good catalyst must have efficient hydrogen utilization as well as have good desulfurization activity. It was previously reported (1,2) that Harshaw 618X, a commercial large pore Ni-Mo/Al_2O_3 catalyst, was active for upgrading SRC/recycle solvent blends. This catalyst was used as a reference catalyst against which others in this study were compared.

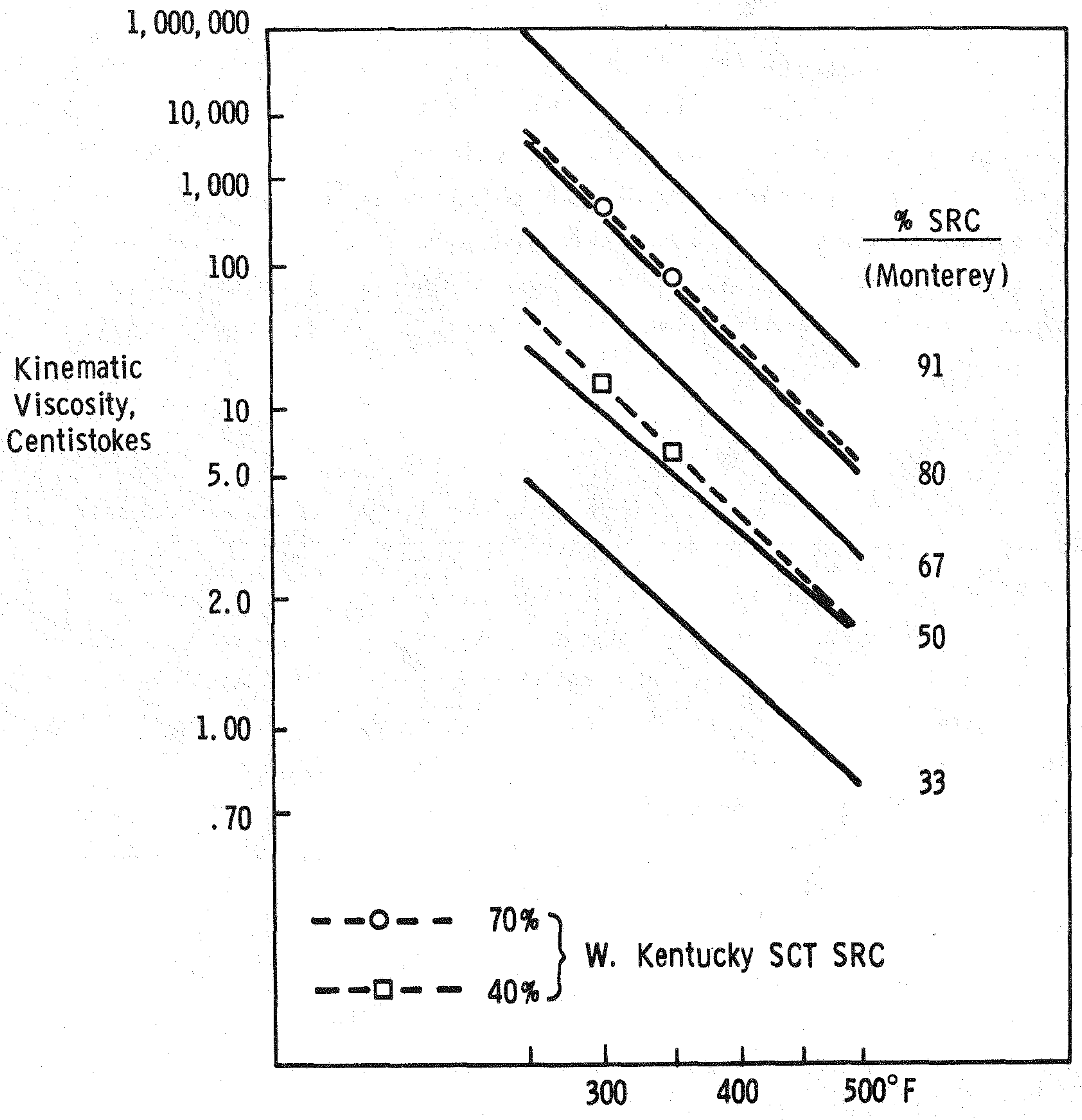
Experimental

Nine catalysts, including three Mobil proprietary catalysts and six commercially available catalysts, were evaluated in a fixed bed, downflow pilot unit. All catalysts had previously been found to be active either for hydrotreating petroleum stocks or for coal hydroliquefaction processes. The charge stocks used in the catalyst evaluations were 50% W. Kentucky short-contact-time (SCT) SRC blends with SCT process recycle solvent. The selection of 50% blending was based on the fluidity requirements. W. Kentucky SCT SRC blends gave considerably higher viscosity than the Monterey regular SRC blends, as shown in Figure 3-1. To assure smooth operation in Mobil's pilot unit, the kinematic viscosity of the charge stocks should be less than 100 centistokes at 350-410°F. Consequently, a 50% blend was selected for the charge stocks.

A detailed description of the pilot unit was previously reported in the 1978 Annual Report (2). Some unit modifications were made to improve mechanical operation. A description of these modifications is given in Appendix B. All lines and valves were heat traced so that temperatures could be controlled at 350-410°F. Plugging in the lines or valves occurred due to SRC coking if the temperature exceeded 450°F. All catalysts were extrudates and were presulfided with a 10% H_2S/H_2 mixture. The operating conditions were as follows:

Figure 3-1

COMPARISON OF KINEMATIC VISCOSITY OF 40 AND 70 WT % SHORT CONTACT TIME (SCT) W. KENTUCKY SRC BLENDS WITH REGULAR MONTEREY SRC BLENDS



Pressure, psig	2000
Temperature, °F	710-810
Space velocity, $V_{FF}/Hr/V_{cat}$	0.3-2.0
H_2 circulation, scf/B	5000

Results and Discussions

The material balances of the catalyst evaluations are given in Appendix A. Properties of W. Kentucky SCT SRC and recycle solvent are given in Table 3-1. Properties of non-proprietary catalysts evaluated in this study are given in Table 3-2. Harshaw 618X, the base case catalyst, is a large pore Ni-Mo/Al_2O_3 catalyst. HT-500, also a Ni-Mo/Al_2O_3 catalyst, has an intermediate pore size. HDS-1443A has a bimodal pore size distribution and is a Ni-Mo version of HDS-1442A, used in the H-Coal® process. HDN-1197 is a high loading Ni-Mo/Al_2O_3 normally used for denitrogenation. Amocat 1A and 1B are developmental catalysts which have been evaluated as coal liquefaction catalysts (3). Three Mobil catalysts (HCL-1, -2, and -3) were also evaluated; because of their proprietary nature, properties have been excluded from this report.

The nine catalysts have been divided into two groups in order to simplify the activity comparisons. Group A is made up of the more active desulfurization catalysts and includes Mobil HCL-2, Mobil HCL-3, American Cyanamid HDS-1443, and Amocat 1A. Group B includes Mobil HCL-1, Harshaw 618X, American Cyanamid HDN-1197, and Amocat 1B. For the activity comparisons, the total product heteroatom removals (and CCR reduction) are plotted versus reactor temperature at liquid hourly space velocities of 0.5 and 1.0. As shown in Appendix C, the ratio of 650°F^+ S to total liquid product S is approximately a constant for hydroprocessed W. Kentucky SCT SRC. Therefore, the TLP sulfur is a good indicator of catalyst activity for SRC desulfurization. Consequently, catalyst activity can be compared on the basis of temperature requirements for achieving specific liquid product heteroatom (or CCR) contents. The results are shown in Figures 3-2 through 3-17. An index of these figures is as follows:

	Group A		Group B	
LHSV	0.5	1.0	0.5	1.0
Desulfurization	Fig. 3-2	Fig. 3-3	Fig. 3-10	Fig. 3-11
Denitrogenation	Fig. 3-4	Fig. 3-5	Fig. 3-12	Fig. 3-13
Deoxygenation	Fig. 3-6	Fig. 3-7	Fig. 3-14	Fig. 3-15
CCR Reduction	Fig. 3-8	Fig. 3-9	Fig. 3-16	Fig. 3-17

The results show:

- Mobil HCL-2 is the most active catalyst for desulfurization of the total product. It is 35°F more active than Harshaw 618X and 7°F more active than HDS-1443, the second most active catalyst. The order of desulfurization activities is:

Table 3-1

PROPERTIES OF W.KENTUCKY SCT SRC AND RECYCLE SOLVENT

	W. Kentucky SCT SRC (76D3653)	Recycle Solvent* (78D839)
Gravity, °API	NA	4.1
Hydrogen, wt %	6.03	7.72
Sulfur, wt %	0.97	0.49
Nitrogen, wt %	1.99	0.73
Oxygen, wt %	5.3	2.7
CCR, wt %	44.98	-
Distillation, °F (D-2887)		
IBP	NA	433
10%	NA	476
30%	NA	514
50%	NA	567
70%	NA	614
90%	NA	689
EP	NA	808

---

* 475°F+ cut from received SCT Process Recycle Solvent

NA: Not Available

Table 3-2

FRESH CATALYST PROPERTIES

	Harshaw	Harshaw	Cyanamid	Cyanamid	Amoco*	Amoco
	618X	HT-500	HDN-1197	HDS-1443A	1B	1A
Mobil ID	7243	8343	9366	9377	9392	9547
Compositions, wt %						
Ni	2.7	2.4	3.7	2.9	0	
CoO						2.9
MoO_3	14.8	14.0	21.7	15.5	14.9	19.7
Physical Properties						
Surface Area, m^2/g**	140	193	130	306	167	154
Pore Volume, cc/g***	0.60	0.5111	0.379	0.764	0.67	0.662
Pore Diameter, Å	172	104	117	100	160	172
Particle Diameter, inch	1/16	1/16	1/20	1/16	1/16	1/16
Pore Size Dist., cc/g						
0- 30Å	0.025	0.004	0.059	0.164	0.027	0.065
30- 80Å	0.037	0.125	0.123	0.355		0.046
80-100Å	0.036	0.252	0.112	0.020	0.063	0.063
100-200Å	0.464	0.123	0.035	0.033	0.419	0.385
200Å+	0.038	0.007	0.050	0.192	0.153	0.103

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* Amoco's Analyses

** BET method

*** Hg penetration

Figure 3-2

COMPARISON OF DESULFURIZATION ACTIVITIES: GROUP A CATALYSTS

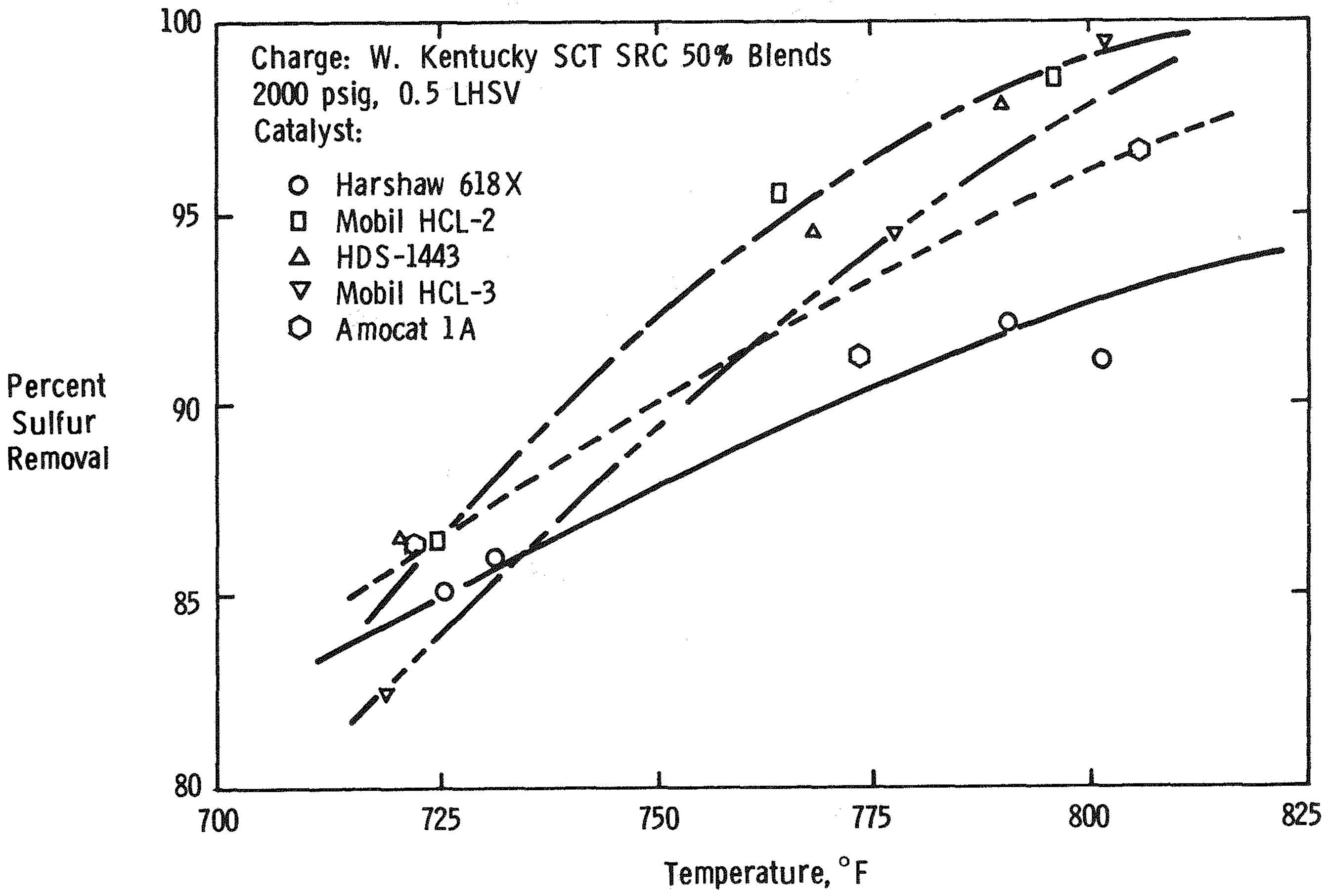


Figure 3-3

COMPARISON OF DESULFURIZATION ACTIVITIES: GROUP A CATALYSTS

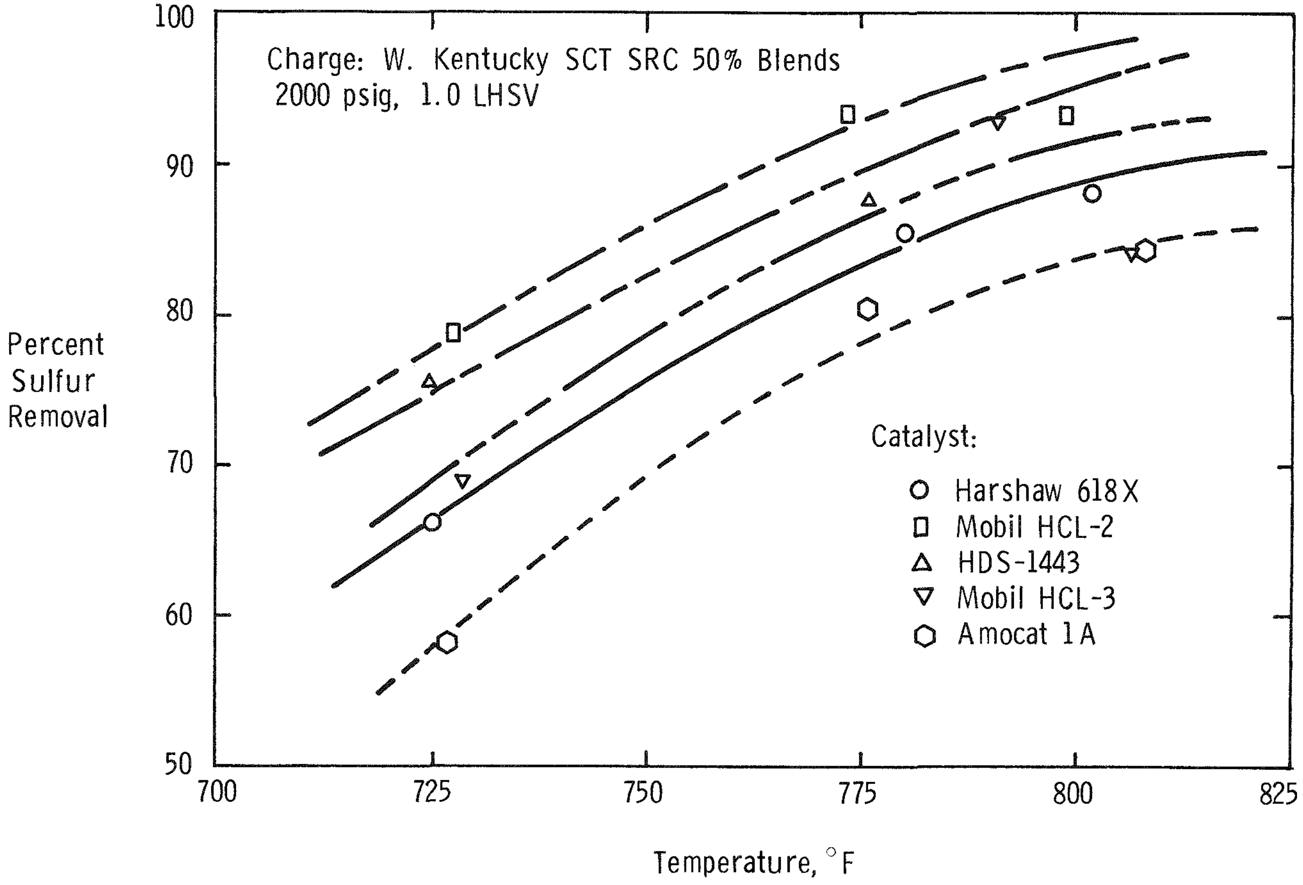


Figure 3-4

COMPARISON OF DENITROGENATION ACTIVITIES: GROUP A CATALYSTS

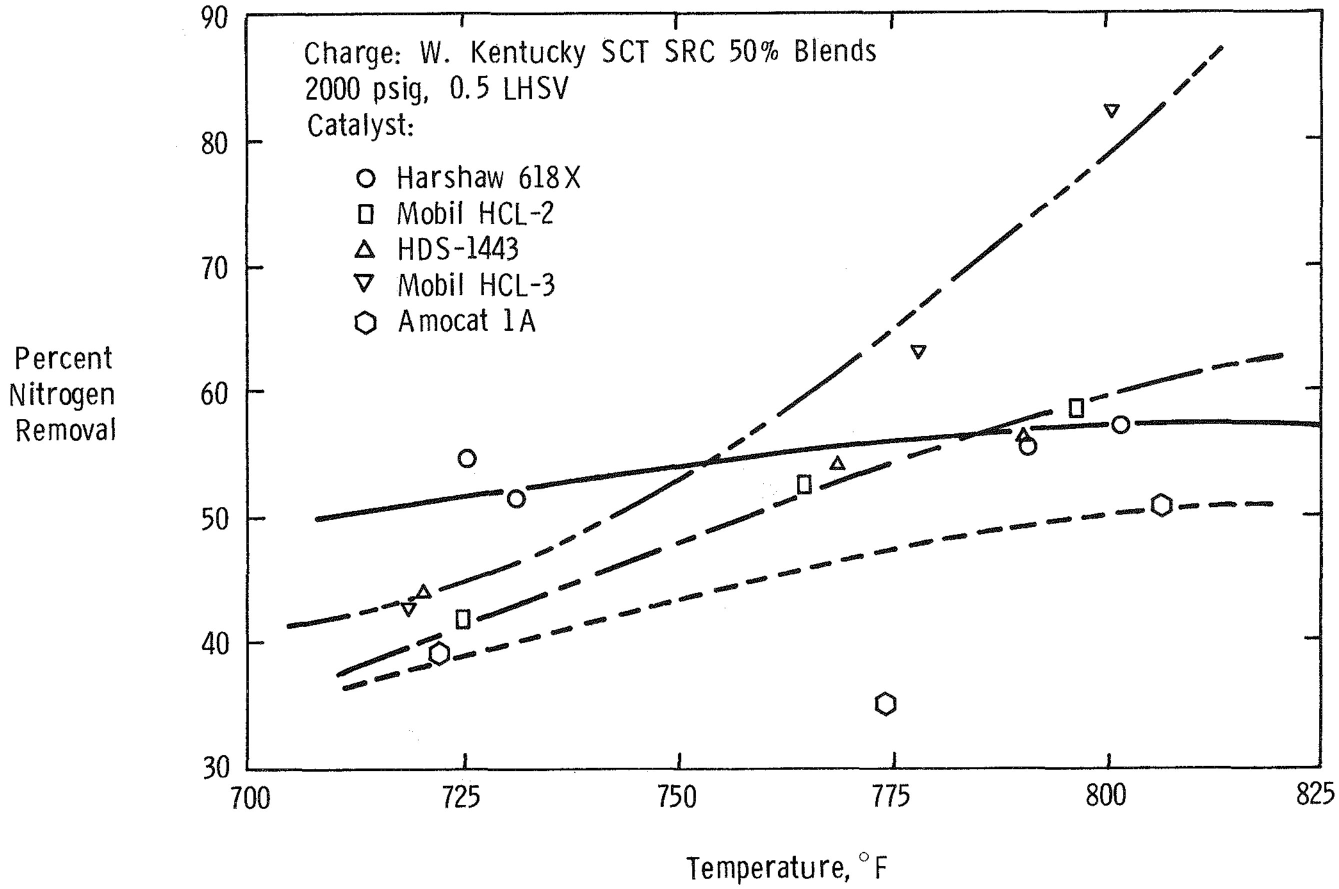


Figure 3-5

COMPARISON OF DENITROGENATION ACTIVITIES: GROUP A CATALYSTS

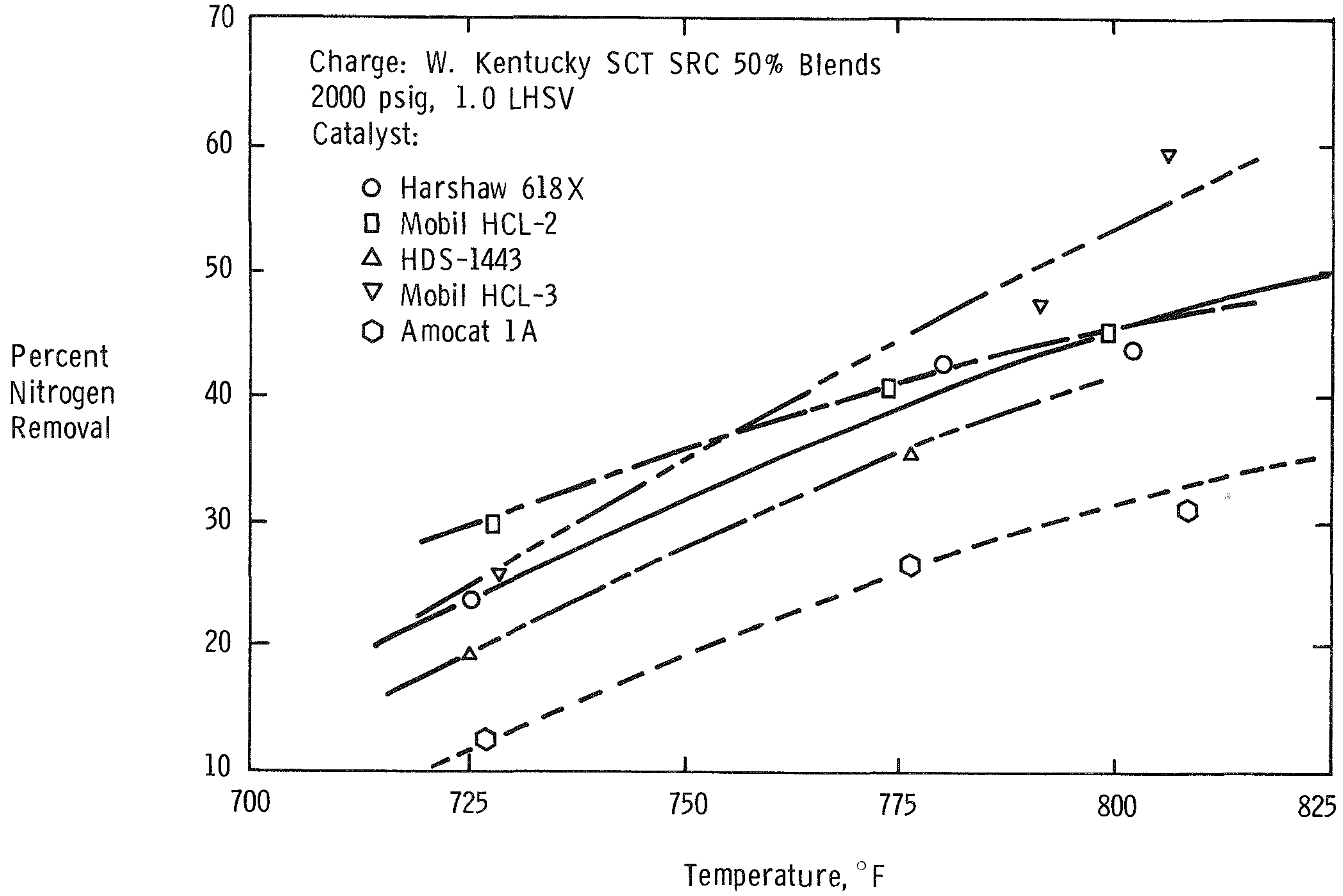


Figure 3-6

COMPARISON OF DEOXYGENATION ACTIVITIES: GROUP A CATALYSTS

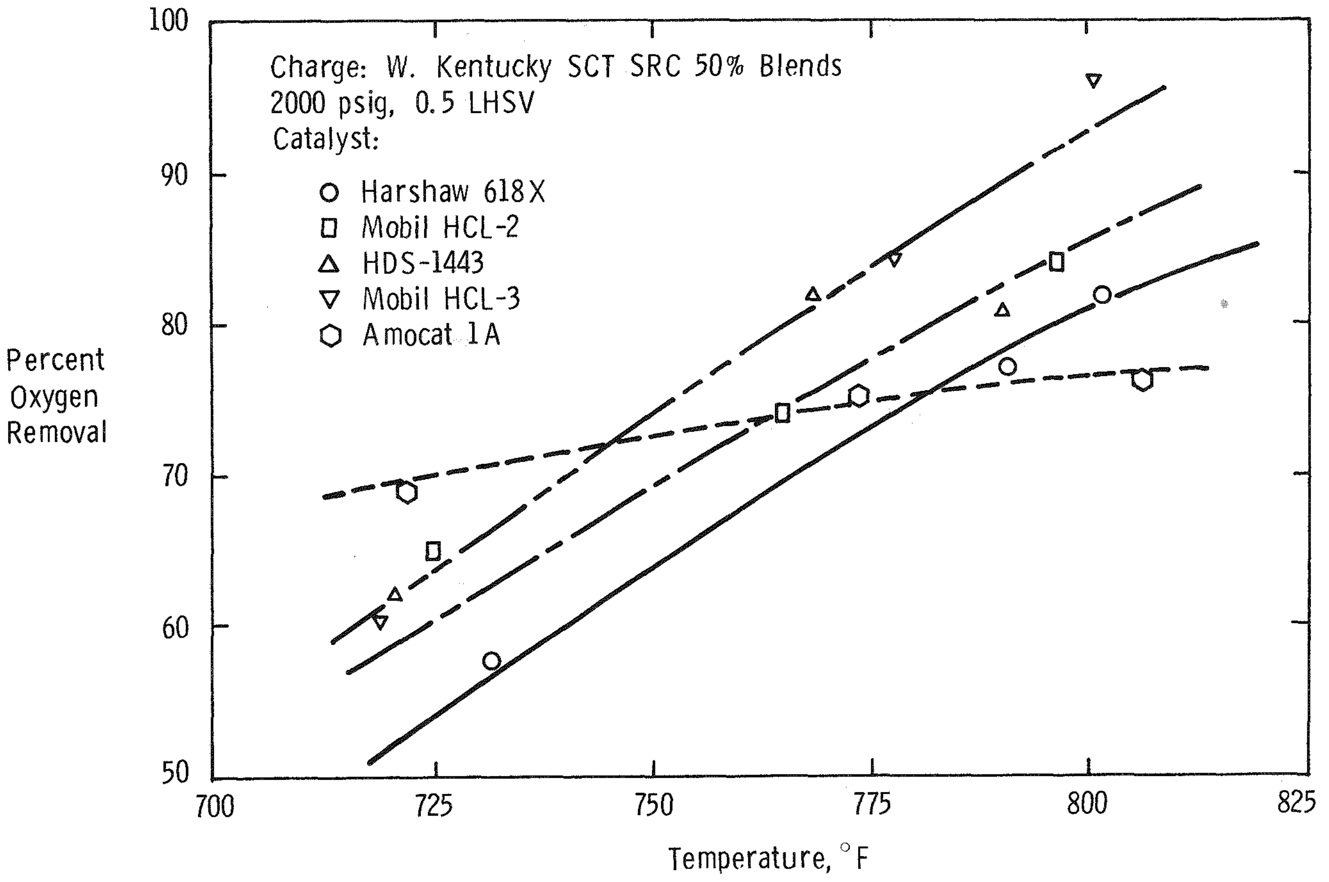


Figure 3-7

COMPARISON OF DEOXYGENATION ACTIVITIES: GROUP A CATALYSTS

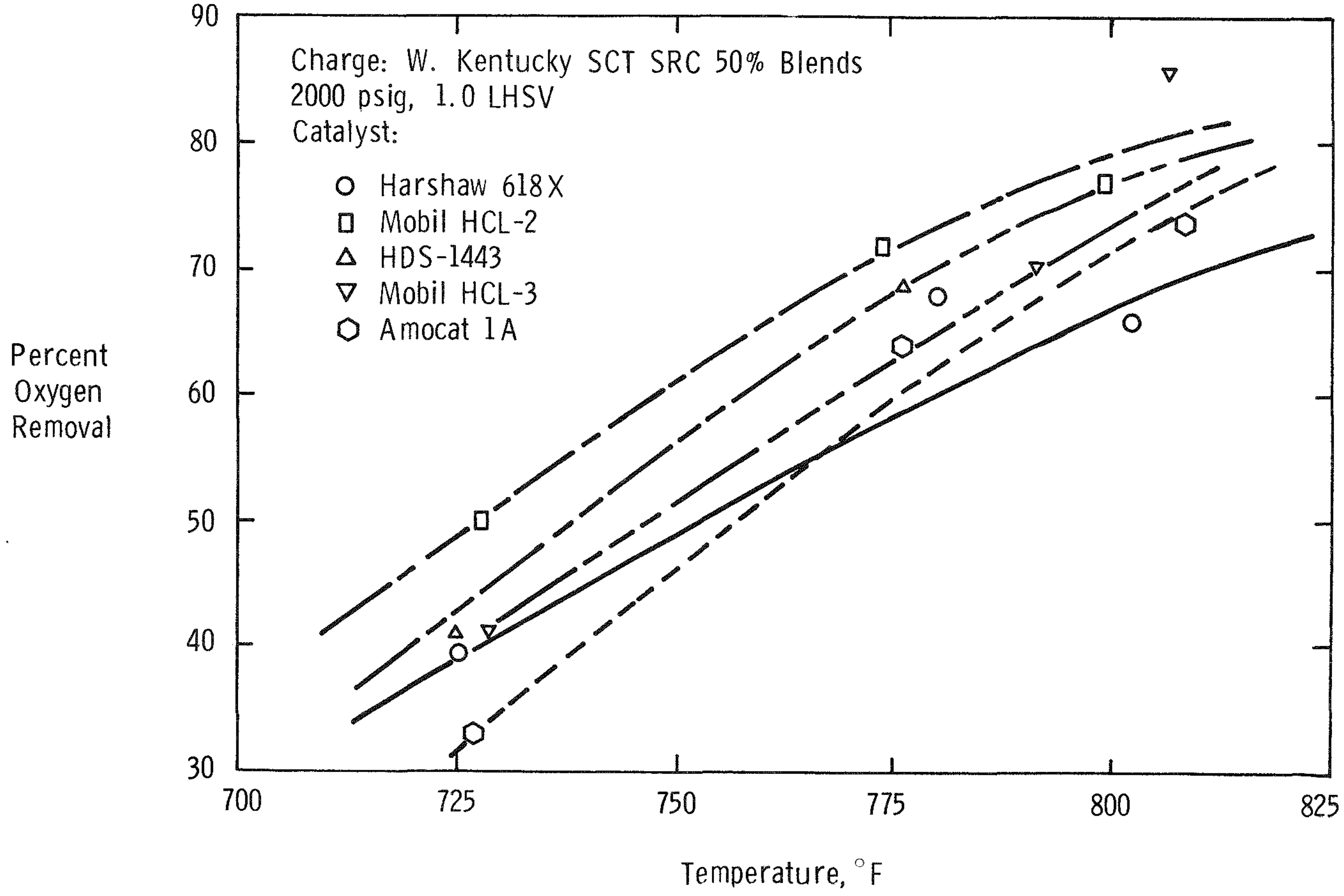


Figure 3-8

COMPARISON OF CCR REDUCTION ACTIVITIES: GROUP A CATALYSTS

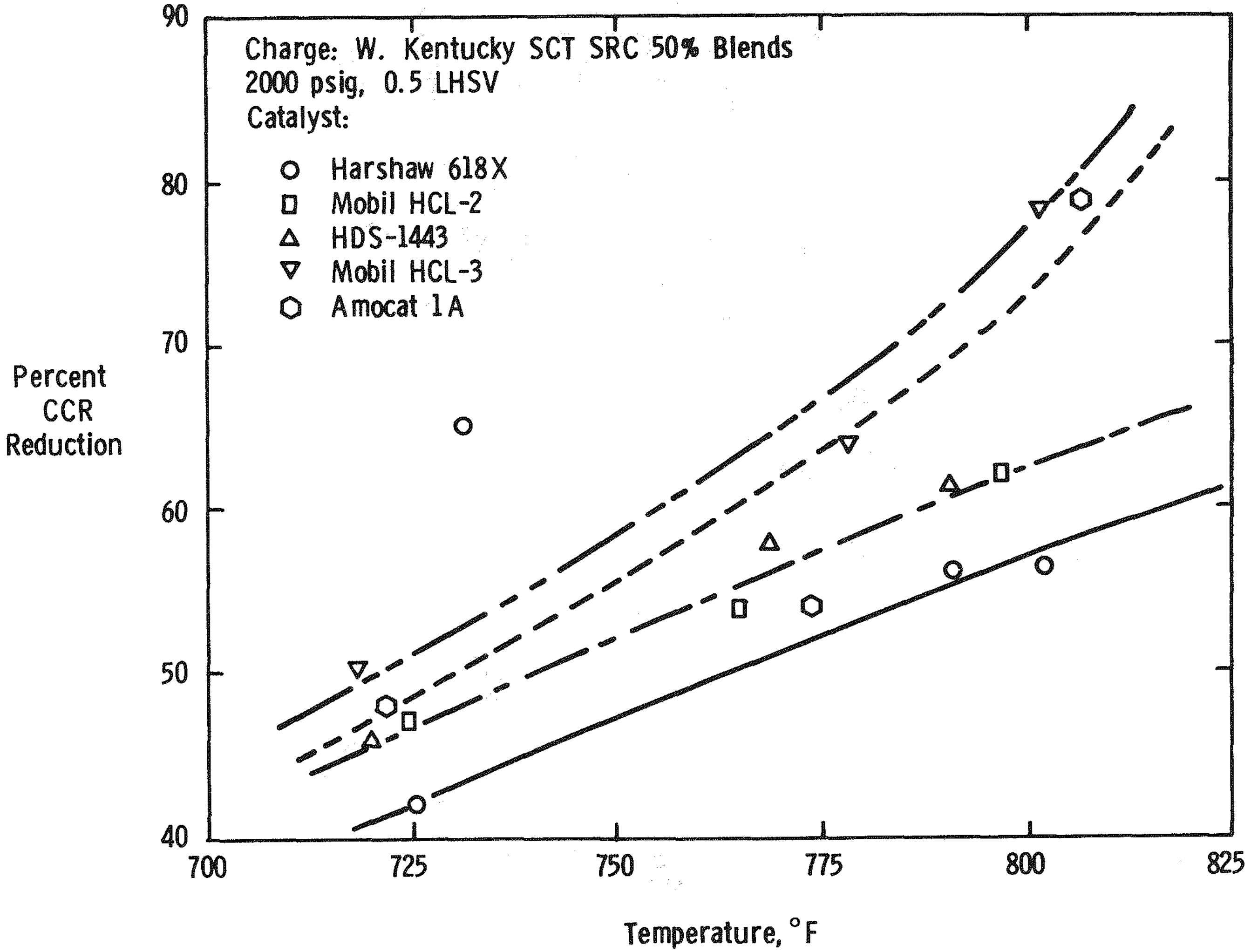


Figure 3-9

COMPARISON OF CCR REDUCTION ACTIVITIES: GROUP A CATALYSTS

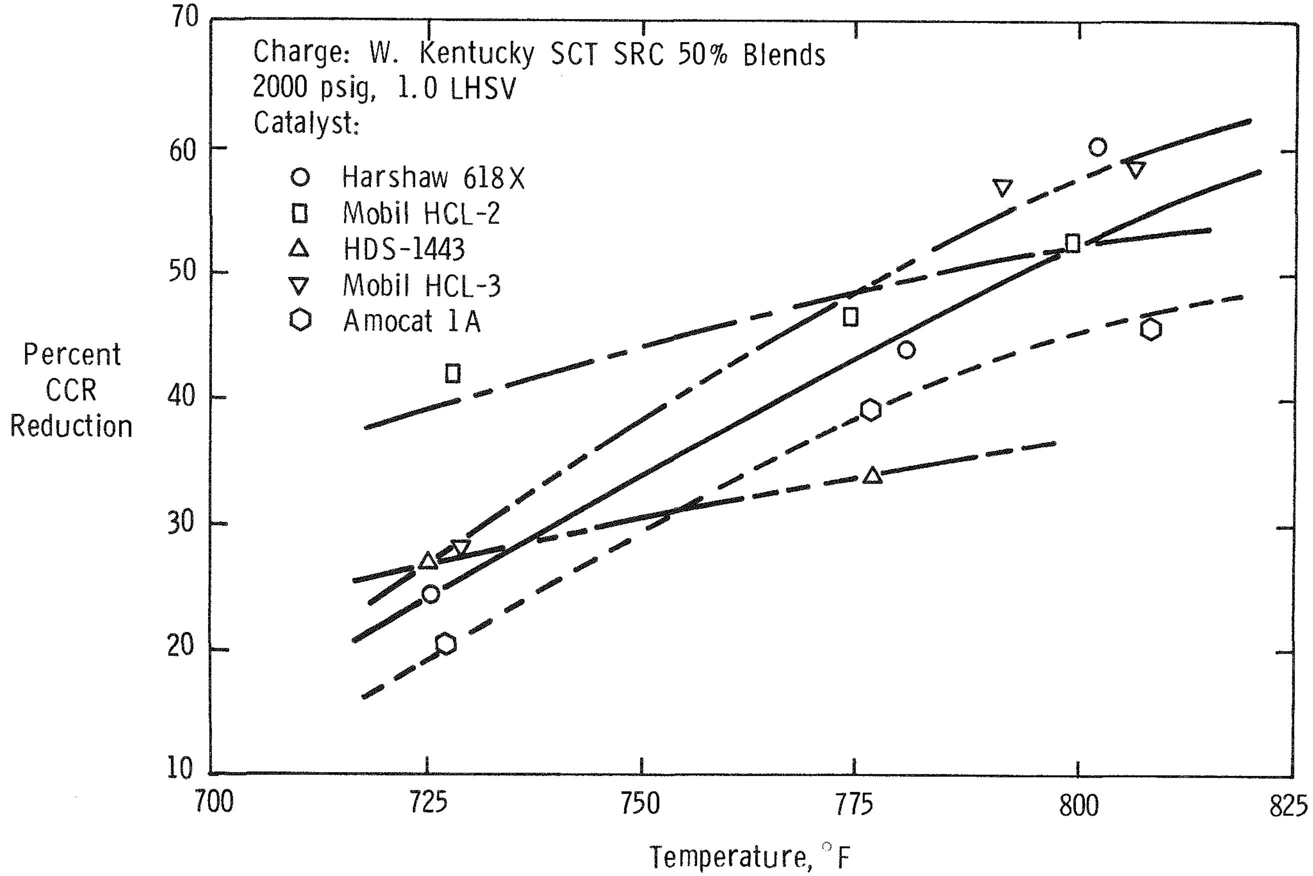


Figure 3-10

COMPARISON OF DESULFURIZATION ACTIVITIES: GROUP B CATALYSTS

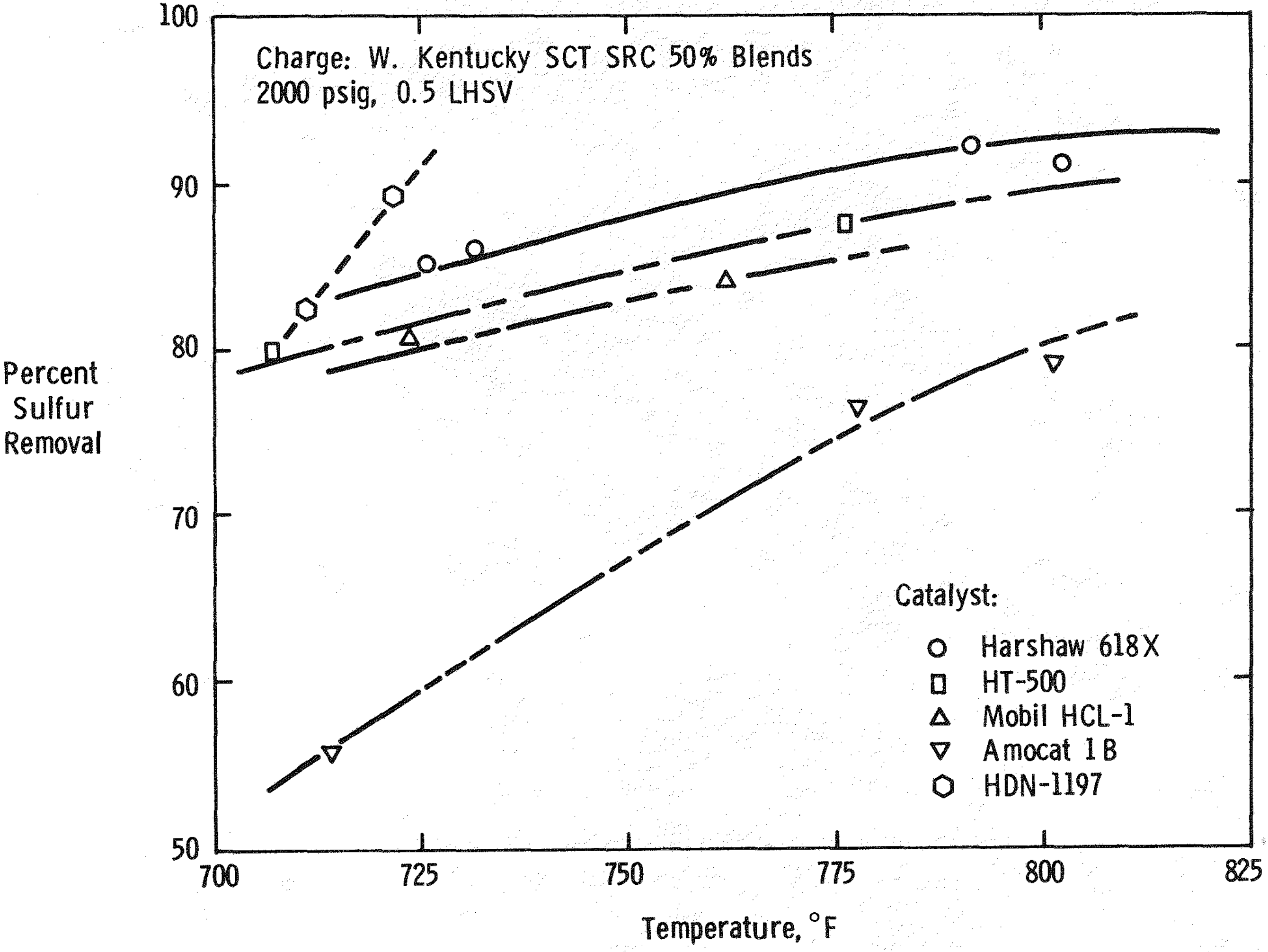


Figure 3-11

COMPARISON OF DESULFURIZATION ACTIVITIES: GROUP B CATALYST

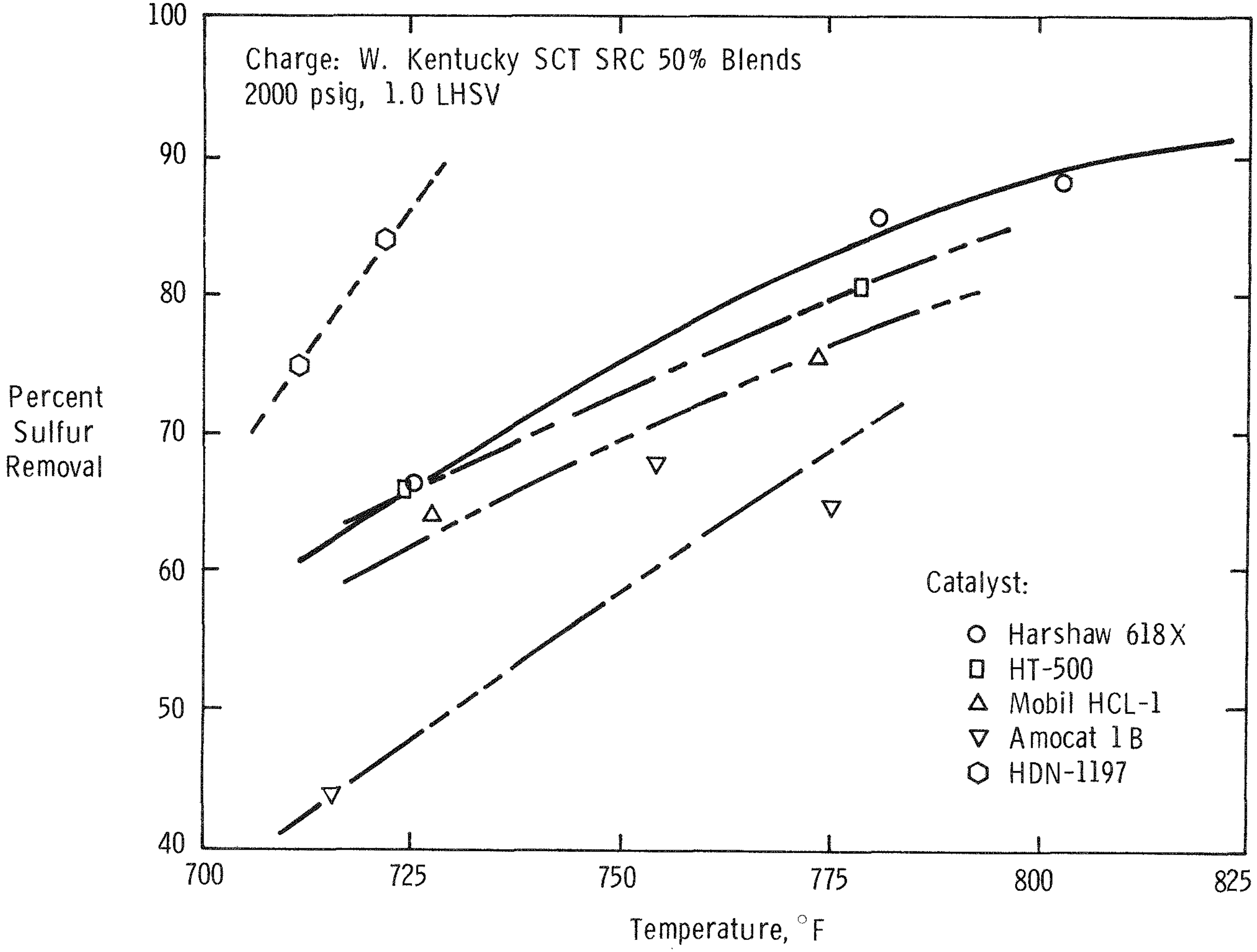


Figure 3-12

COMPARISON OF DENITROGENATION ACTIVITIES: GROUP B CATALYST

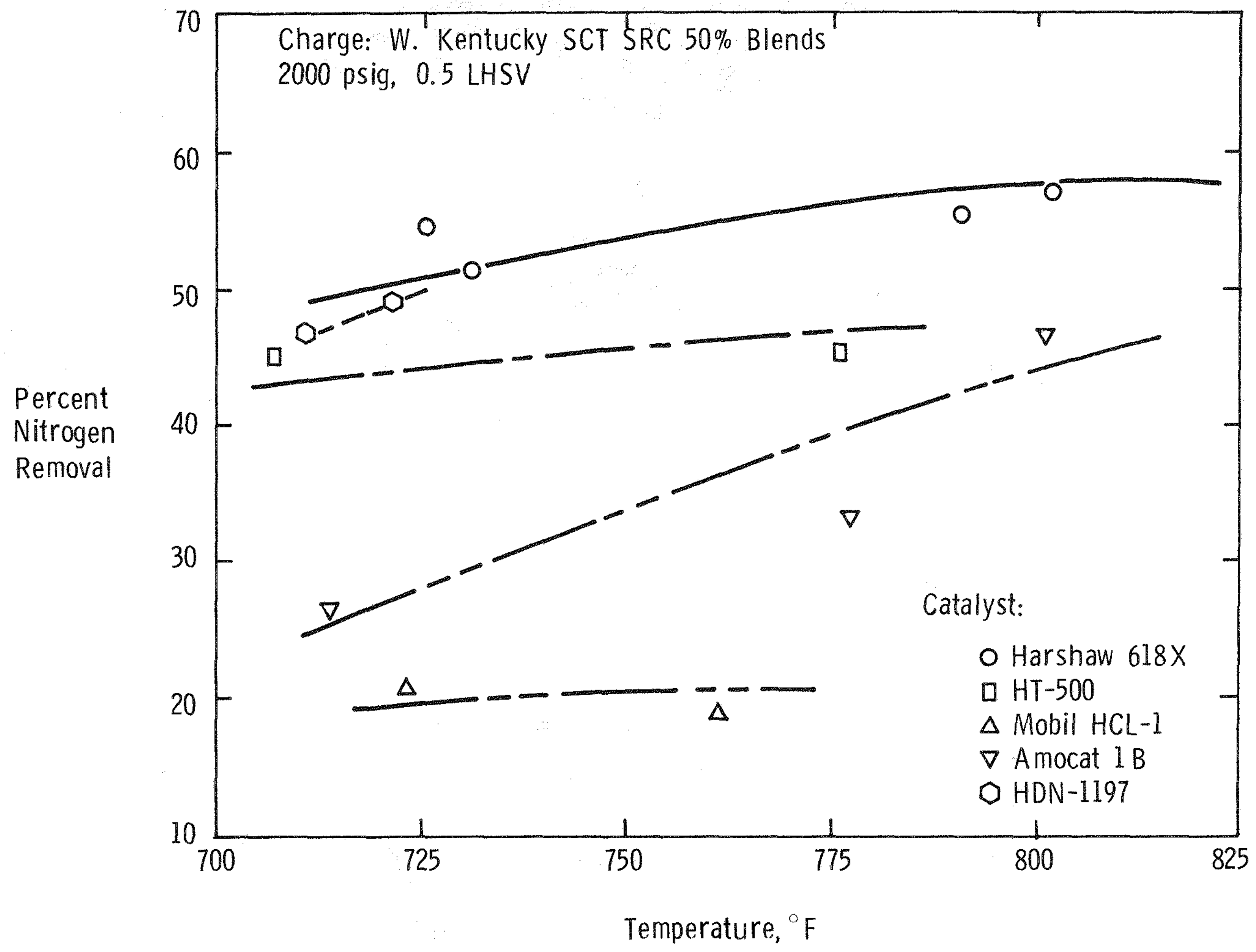


Figure 3-13

COMPARISON OF DENITROGENATION ACTIVITIES: GROUP B CATALYSTS

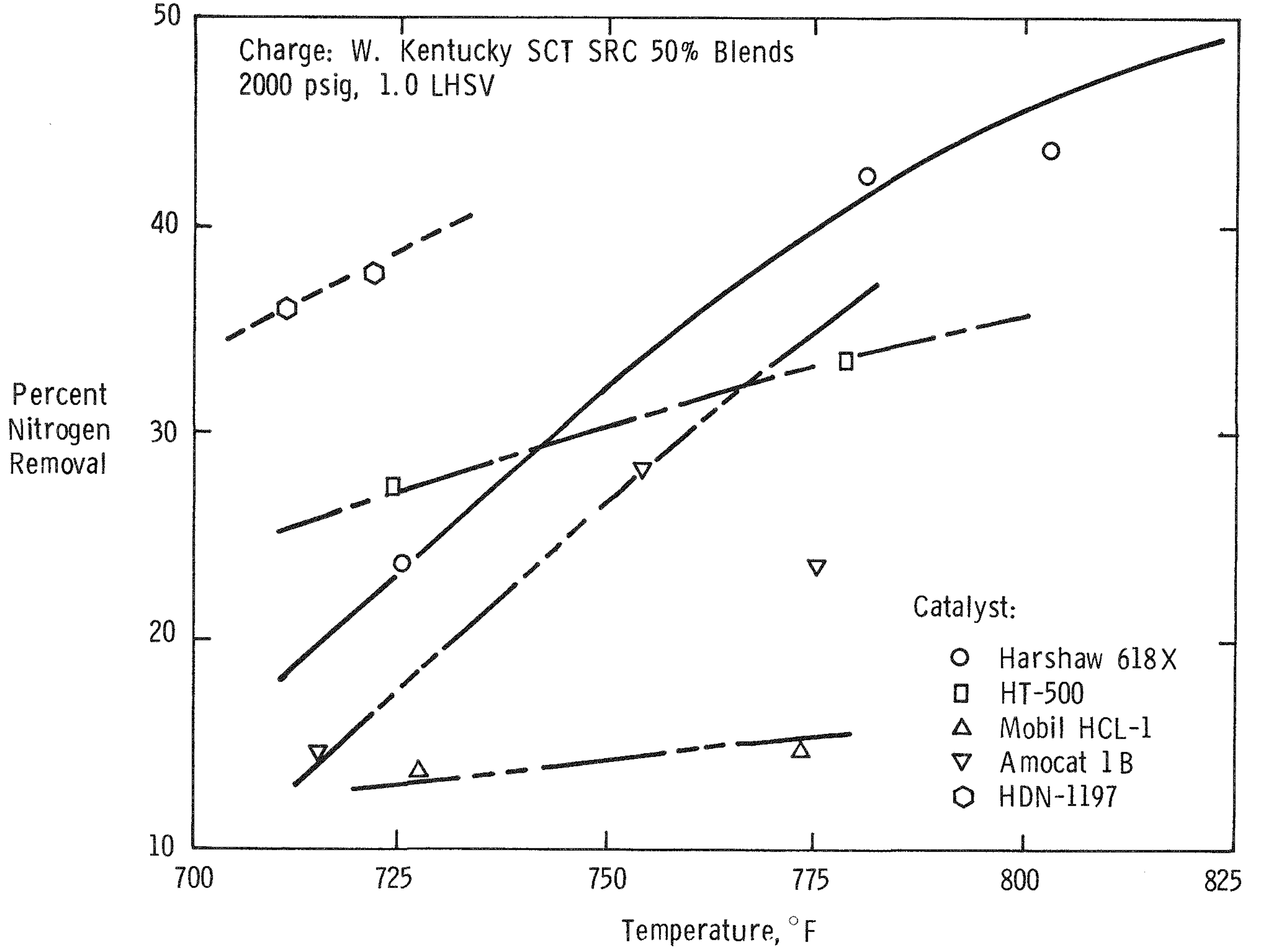


Figure 3-14

COMPARISON OF DEOXYGENATION ACTIVITIES: GROUP B CATALYSTS

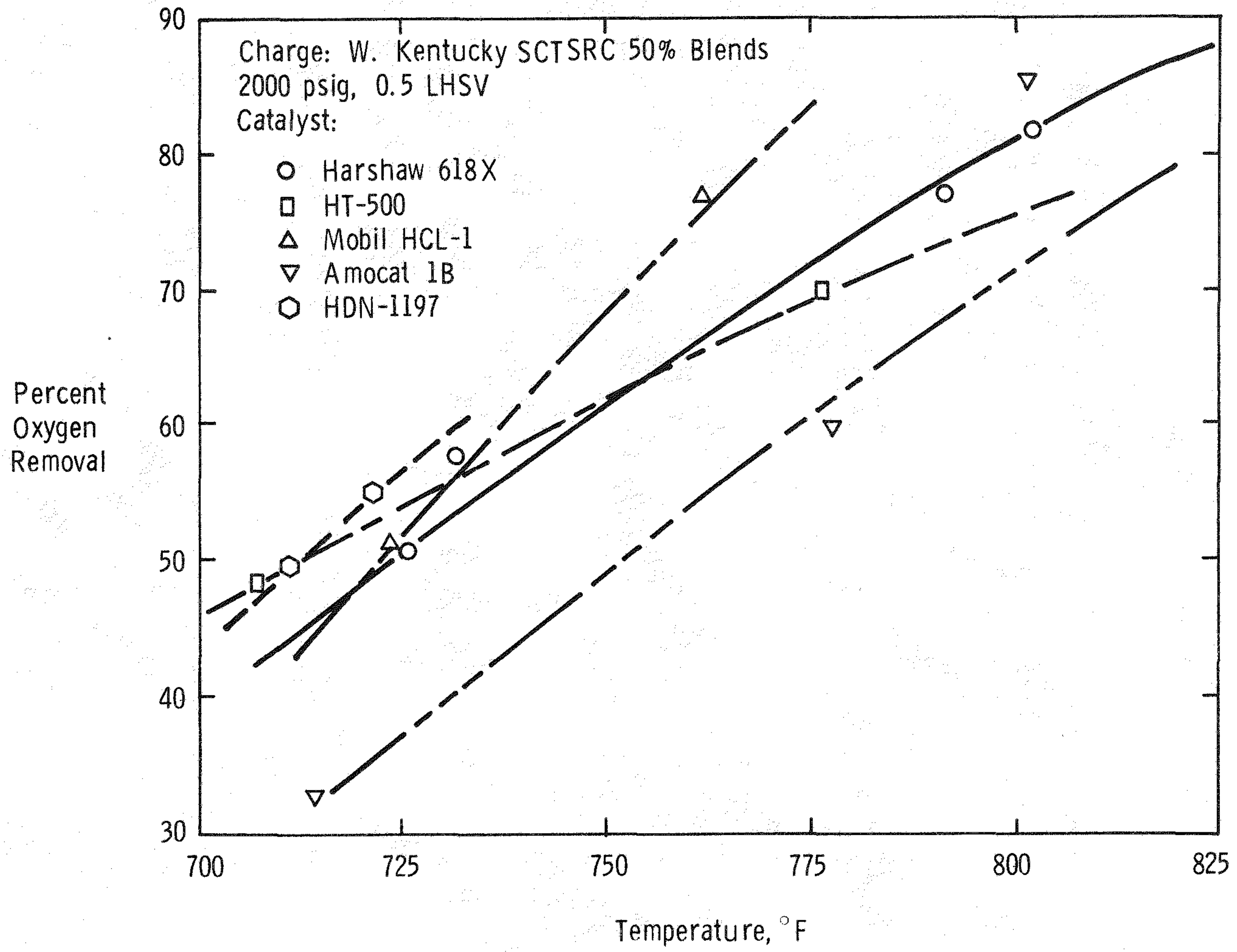


Figure 3-15

COMPARISON OF DEOXYGENATION ACTIVITIES: GROUP B CATALYSTS

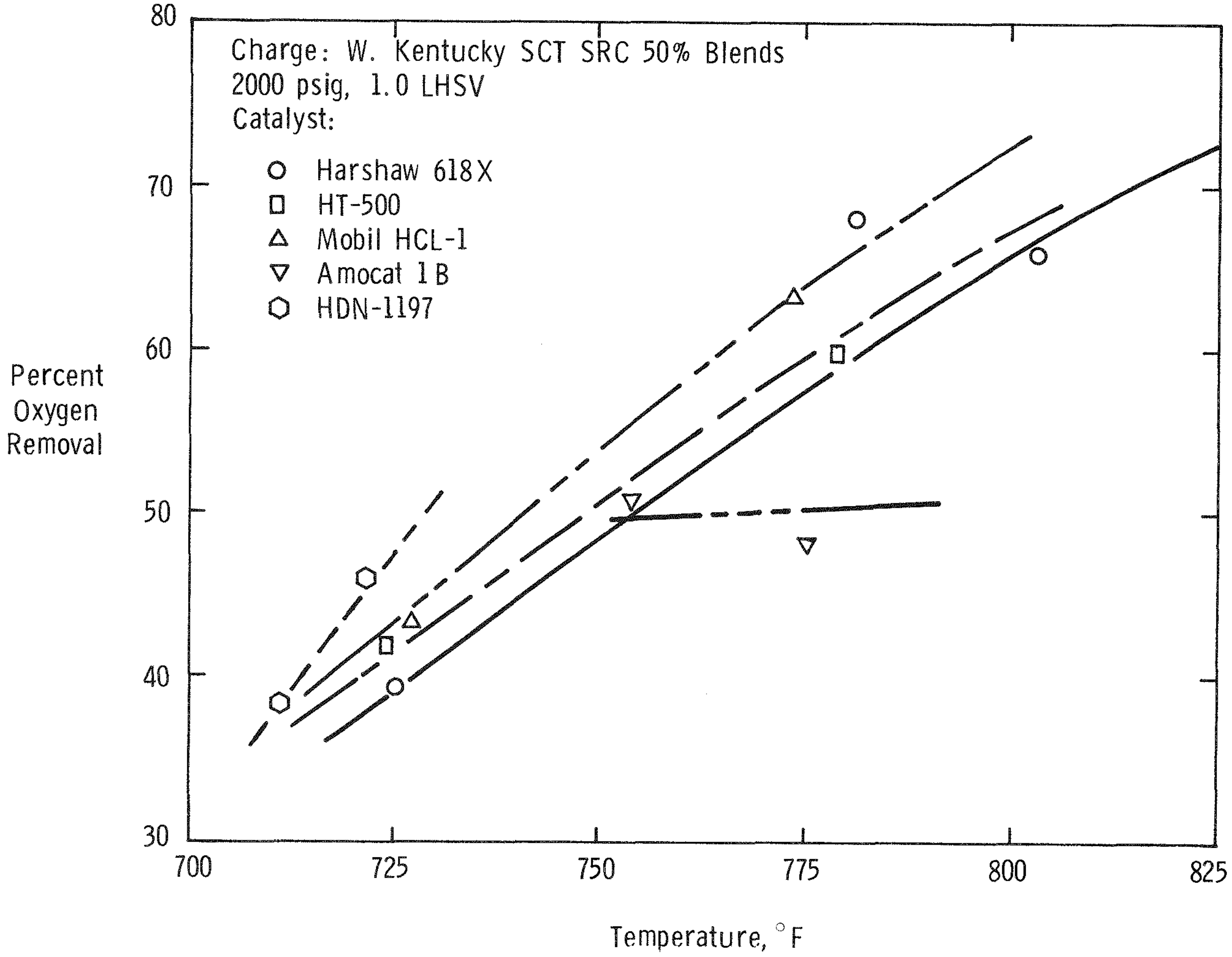


Figure 3-16

COMPARISON OF CCR REDUCTION ACTIVITIES: GROUP B CATALYSTS

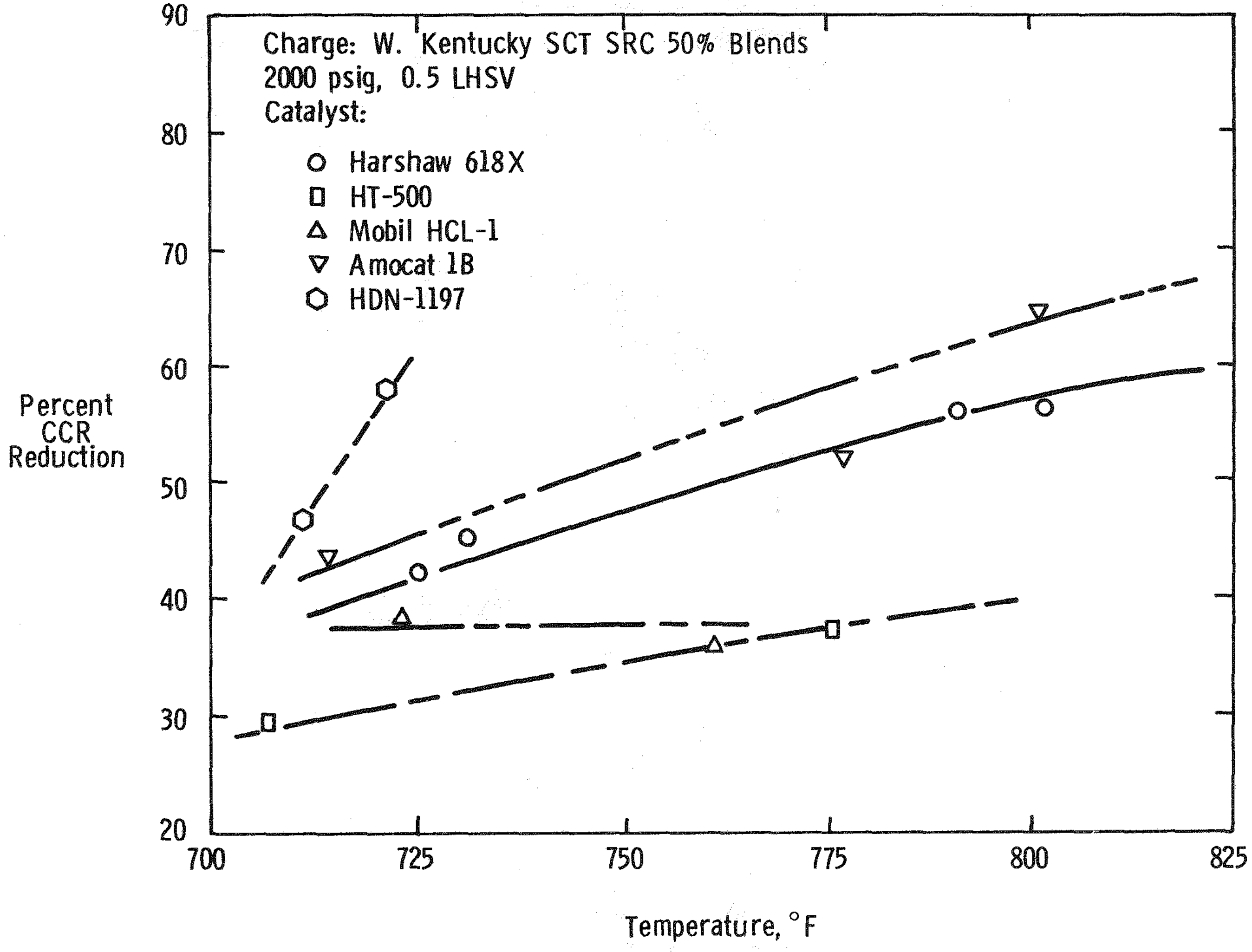
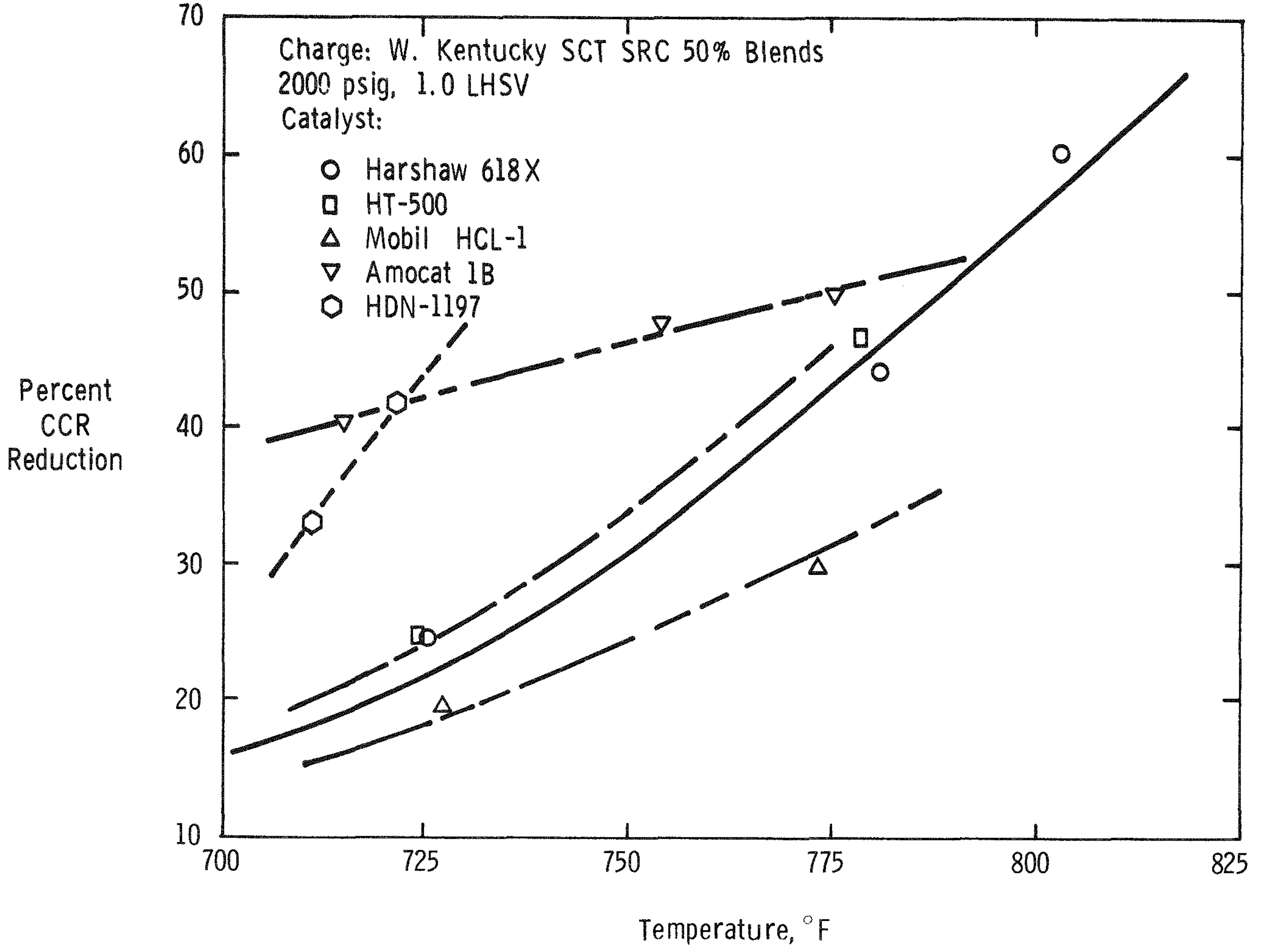


Figure 3-17

COMPARISON OF CCR REDUCTION ACTIVITIES: GROUP B CATALYSTS



  - Mobil HCL-2 > HDS-1433 > Mobil HCL-3 ≃ Amocat 1A > HDN-1197 ≃ Harshaw 618X > HT-500 > Mobil HCL-1 > Amocat 1B.

- Mobil HCL-3 is the most active catalyst for total product denitrogenation. It is 10°F more active than Harshaw 618X and 5°F more active than Mobil HCL-2. The order of denitrogenation activities is:
  - Mobil HCL-3 > Mobil HCL-2 ≃ HDN-1197 > Harshaw 618X ≃ HDS-1443 > HT-500 > Amocat 1B > Amocat 1A > Mobil HCL-1.

- Mobil HCL-2 is the most active catalyst for total product deoxygenation. It is 35°F more active than Harshaw 618X and 10°F more active than HDS-1443. The order of deoxygenation activities is:
  - Mobil HCL-2 > HDS-1443 > Mobil HCL-3 ≃ Amocat 1A ≃ HDN-1197 > Mobil HCL-1 ≃ HT-500 > Harshaw 618X > Amocat 1B.

- Mobil HCL-3 is the most active catalyst for CCR reduction. It is 40°F more active than Harshaw 618X and 10°F more active than HDS-1443. The order of CCR removal activities is:
  - Mobil HCL-3 > HDS-1443 ≃ HDN-1197 ≃ Amocat 1B > Mobil HCL-2 > Amocat 1A ≃ Harshaw 618X > HT-500 > Mobil HCL-1.

As discussed previously, one of the primary research objectives is to reduce hydrogen consumption without sacrificing desulfurization activity. In Figures 3-18 and 3-19, the total hydrogen consumption is plotted as a function of sulfur concentration in the total liquid products for group A and group B catalysts, respectively. As shown in Figures 3-18 and 3-19, Mobil HCL-2 and Amocat 1A are low hydrogen consumption catalysts, compared to Harshaw 618X; each uses 30-40% less hydrogen than Harshaw 618X catalyst to achieve a specific sulfur level in the total liquid products. Amocat 1B (Mo/Al_2O_3), having low desulfurization activity, uses 50% more hydrogen than Harshaw 618X as shown in Figure 3-19. HDS-1443 also seems to be a low hydrogen consumption catalyst. The hydrogen consumption with Mobil HCL-1 is intermediate to Mobil HCL-3 and Harshaw 618X. Other catalysts, such as HT-500, Mobil HCL-3 and HDN-1197, showed similar hydrogen consumption/desulfurization selectivity as Harshaw 618X.

Generally, the hydrogen consumption is determined by the catalyst's hydrogenation activity for the liquid (i.e., C_6+) products; 50-80% of the total hydrogen consumption is utilized for hydrogenation of C_6+ products. The selectivity of C_6+ liquids hydrogenation versus sulfur removal is shown in Figures 3-20 and 3-21. Similar to the total hydrogen consumption/sulfur removal, Mobil HCL-2 and Amocat 1A are low C_6+ hydrogenation activity catalysts. Mobil HCL-3, however, shows a

Figure 3-18

TOTAL HYDROGEN CONSUMPTION AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP A CATALYSTS

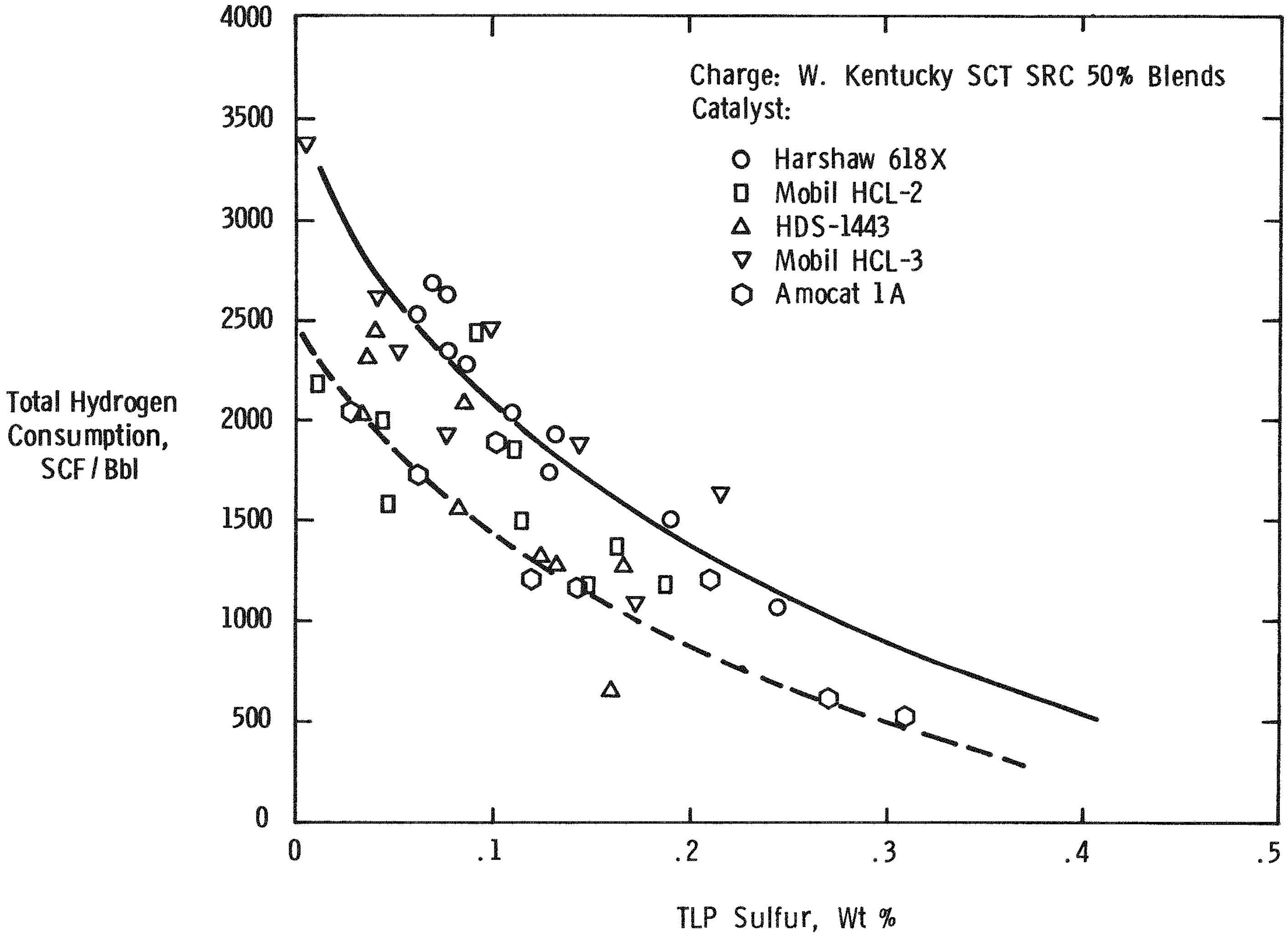


Figure 3-19

TOTAL HYDROGEN CONSUMPTION AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP B CATALYSTS

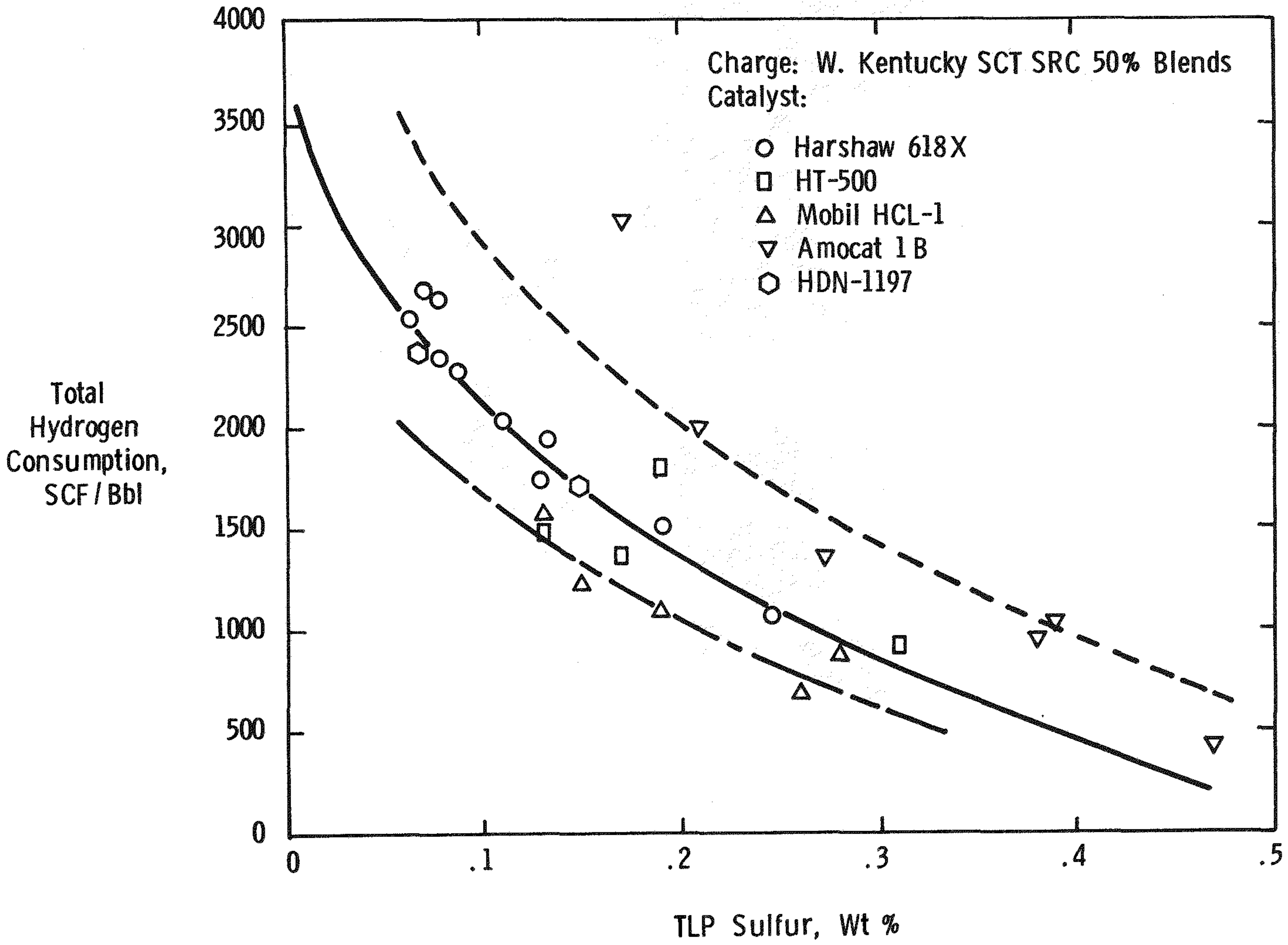


Figure 3-20

HYDROGEN CONSUMED BY C_6^+ AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP A CATALYSTS

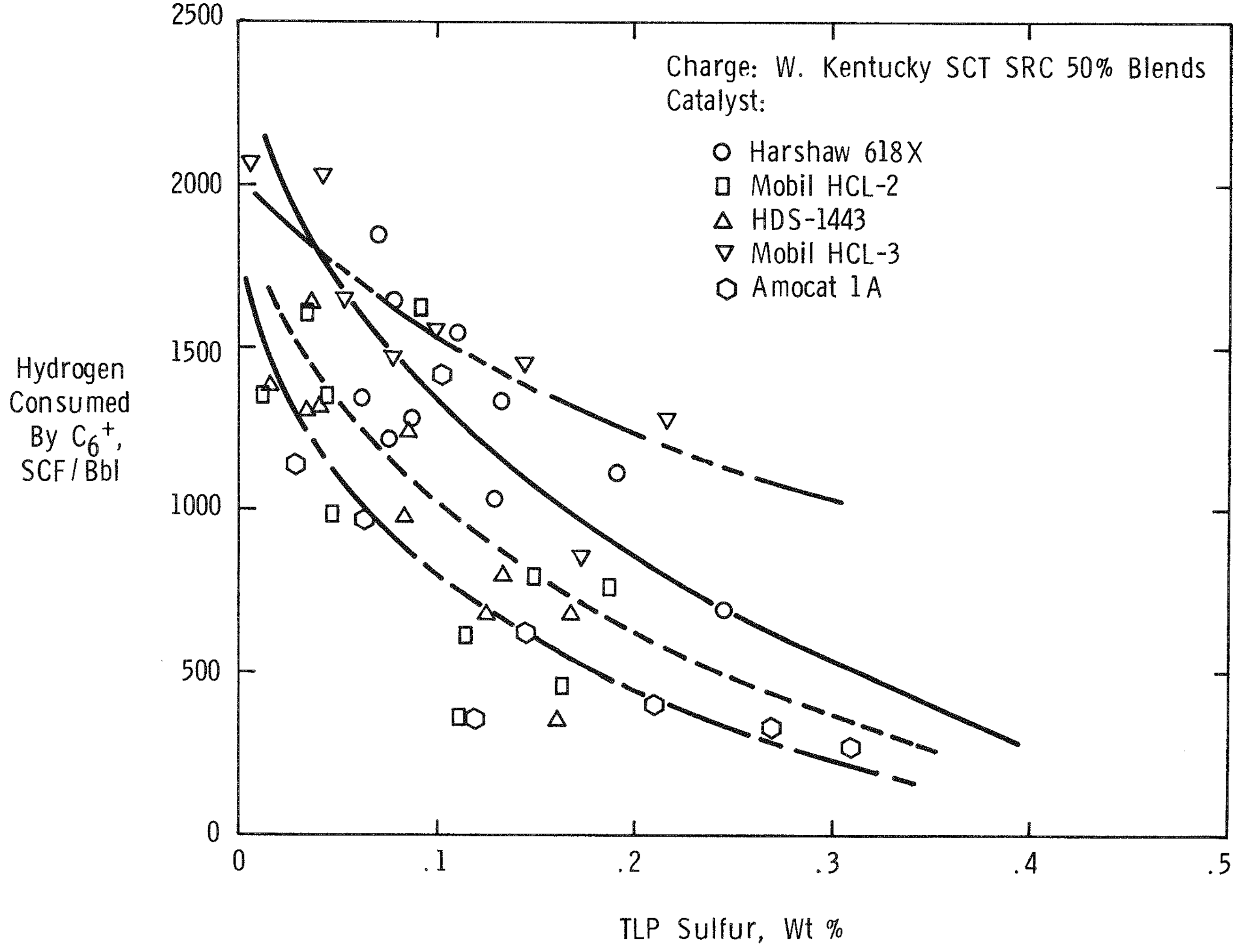
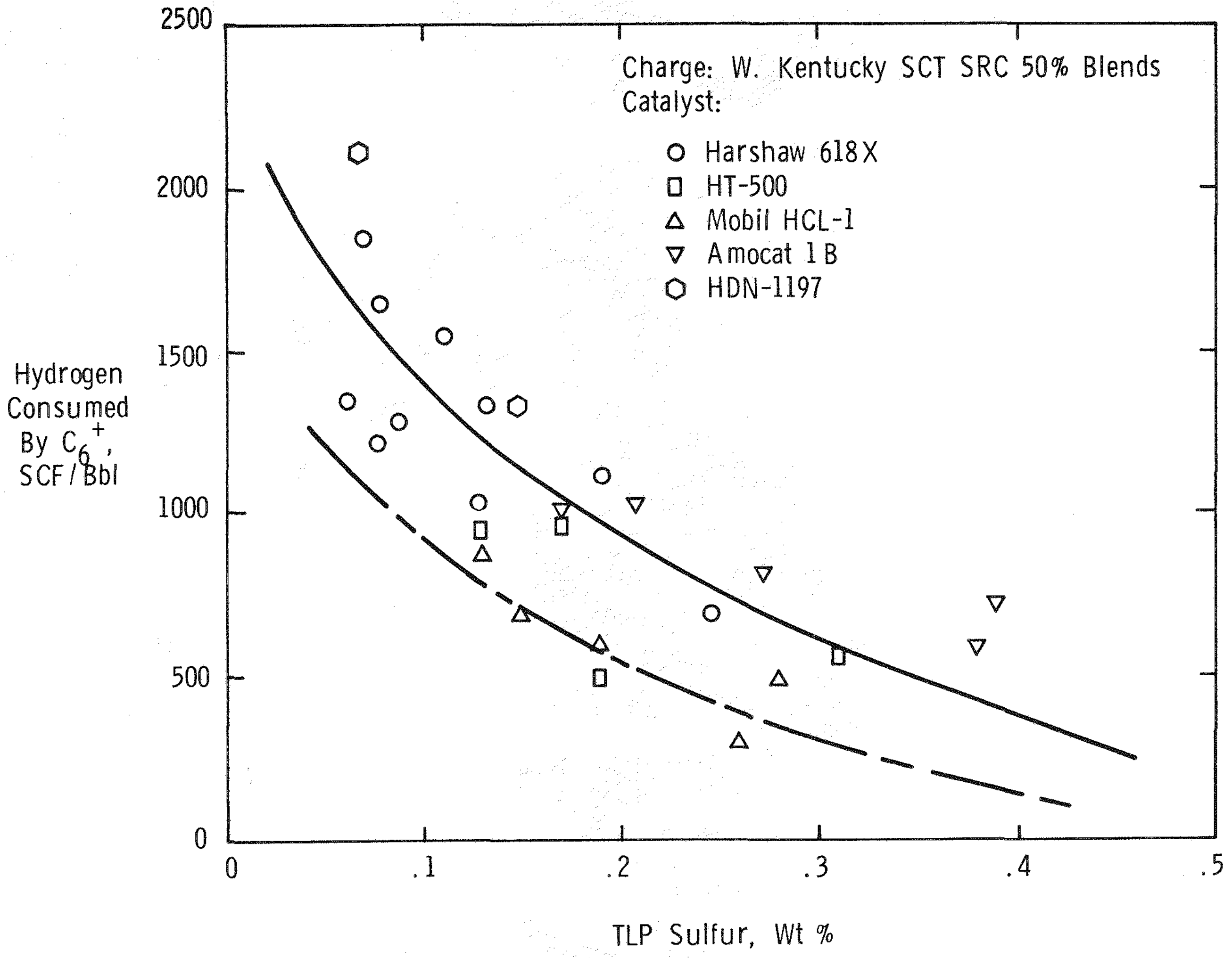


Figure 3-21

HYDROGEN CONSUMED BY C_6^+ AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP B CATALYSTS



significantly high hydrogenation activity.

In addition to hydrogenation of C_6+ liquids, hydrogen also is consumed for light material (C_1-C_5) formation and for heteroatom removal. The gas make, (i.e., C_1-C_5 formation) is strongly dependent on temperature. However, the heteroatom removal is primarily determined by the type of catalysts. The group A catalysts have similar heteroatom removal/desulfurization selectivities, as shown in Figure 3-22. Here, hydrogen consumption for the heteroatom removal is plotted versus sulfur concentration in the products. A similar plot is made for group B catalysts as shown in Figure 3-23. Amocat 1B shows a significantly higher hydrogen consumption for heteroatom removal compared to that of Harshaw 618X.

Based on the above discussions, the following conclusions can be drawn:

- For total product desulfurization, Co-Mo/Al_2O_3 is more active than Ni-Mo/Al_2O_3. As an example, Amocat 1A is more active than Harshaw 618X; Amocat 1B is the least active catalyst.
- Hydrogen consumption/desulfurization selectivity may be affected by the type of catalyst promoter, e.g., Mo/Al_2O_3 (Amocat 1B) > Ni-Mo/Al_2O_3 (Harshaw 618X) > Co-Mo/Al_2O_3 (Amocat 1A).
- The presence of large pores in the catalyst support (i.e., > 100Å) appears to improve desulfurization activity. The order of desulfurization activity of the three Ni-Mo/Al_2O_3 catalysts of of similar loading is: HDS-1443 > Harshaw 618X > HT-500 (see section 5 for molecular size distributions of raw and hydrotreated W. Kentucky SCT SRC).

Based on the activity evaluations, Mobil HCL-2, Amocat 1A and Cyanamid HDS-1443 were selected for constant temperature aging runs. Detailed discussions of the aging runs are given on section 4.

All catalysts except Cyanamid HDN-1197 gave homogeneous products. HDN-1197 yielded a product with two incompatible phases; analyses of these two phases are given in Table 3-3. The solid phase (precipitate) was consistently of a lower quality than the liquid phase (filtrate). HDN-1197 is a highly loaded Ni-Mo/Al_2O_3 catalyst with 75% of its pore volume less than 100Å.

Analyses of Used Catalysts

The analyses of four used catalysts tested with 50% W. Kentucky SCT SRC blends are given in Table 3-4. All catalysts were evaluated in a similar sequence of process conditions. The coke and iron depositions appear to be strongly dependent upon catalyst; Harshaw 618X and HDS-1443 are high, but Amocat 1A and 1B are low in coke

Figure 3-22

HYDROGEN CONSUMED BY HETEROATOM REMOVAL AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP A CATALYSTS

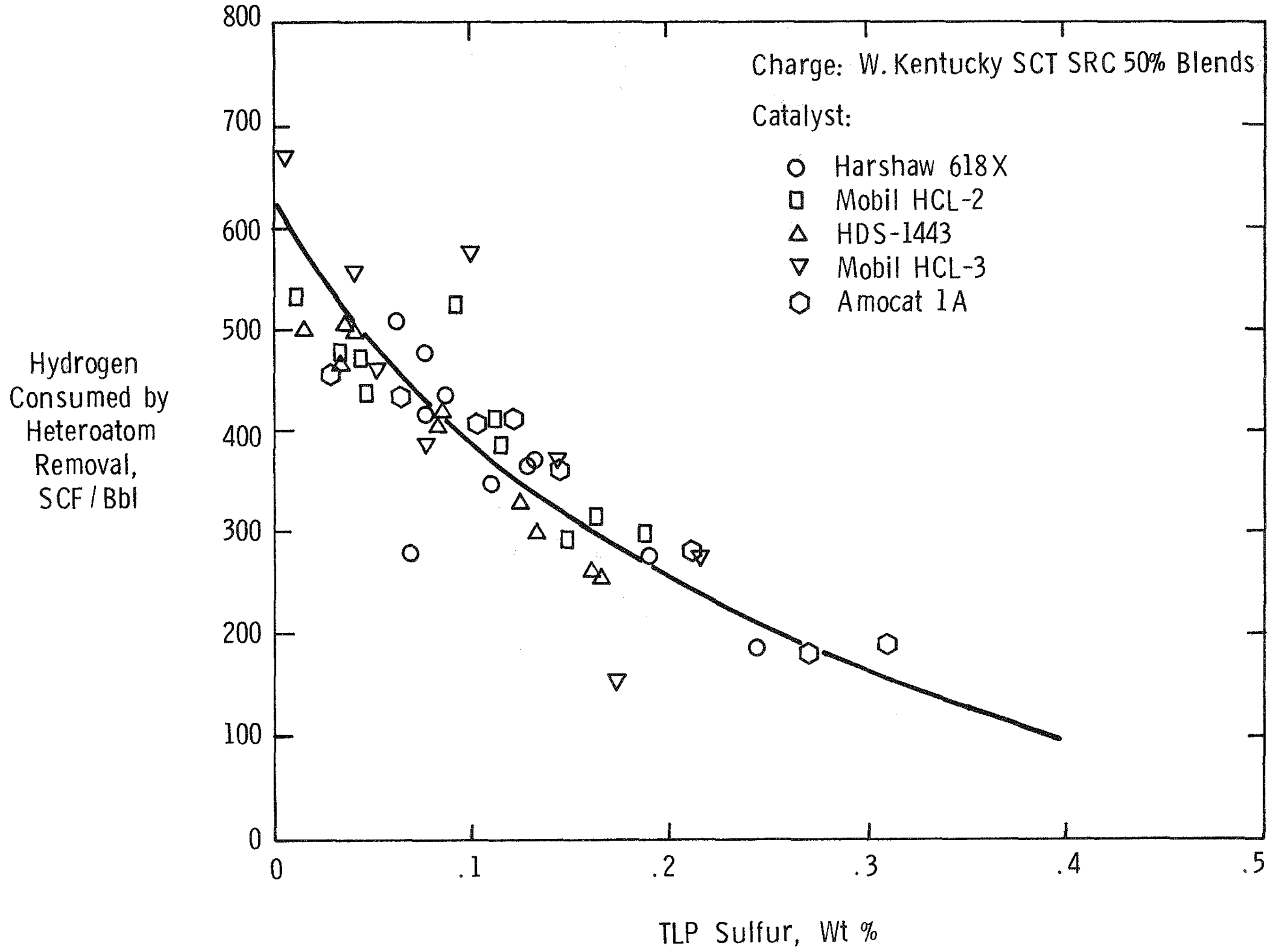


Figure 3-23

HYDROGEN CONSUMED BY HETEROATOM REMOVAL AS A FUNCTION OF SULFUR IN LIQUID PRODUCT: GROUP B CATALYSTS

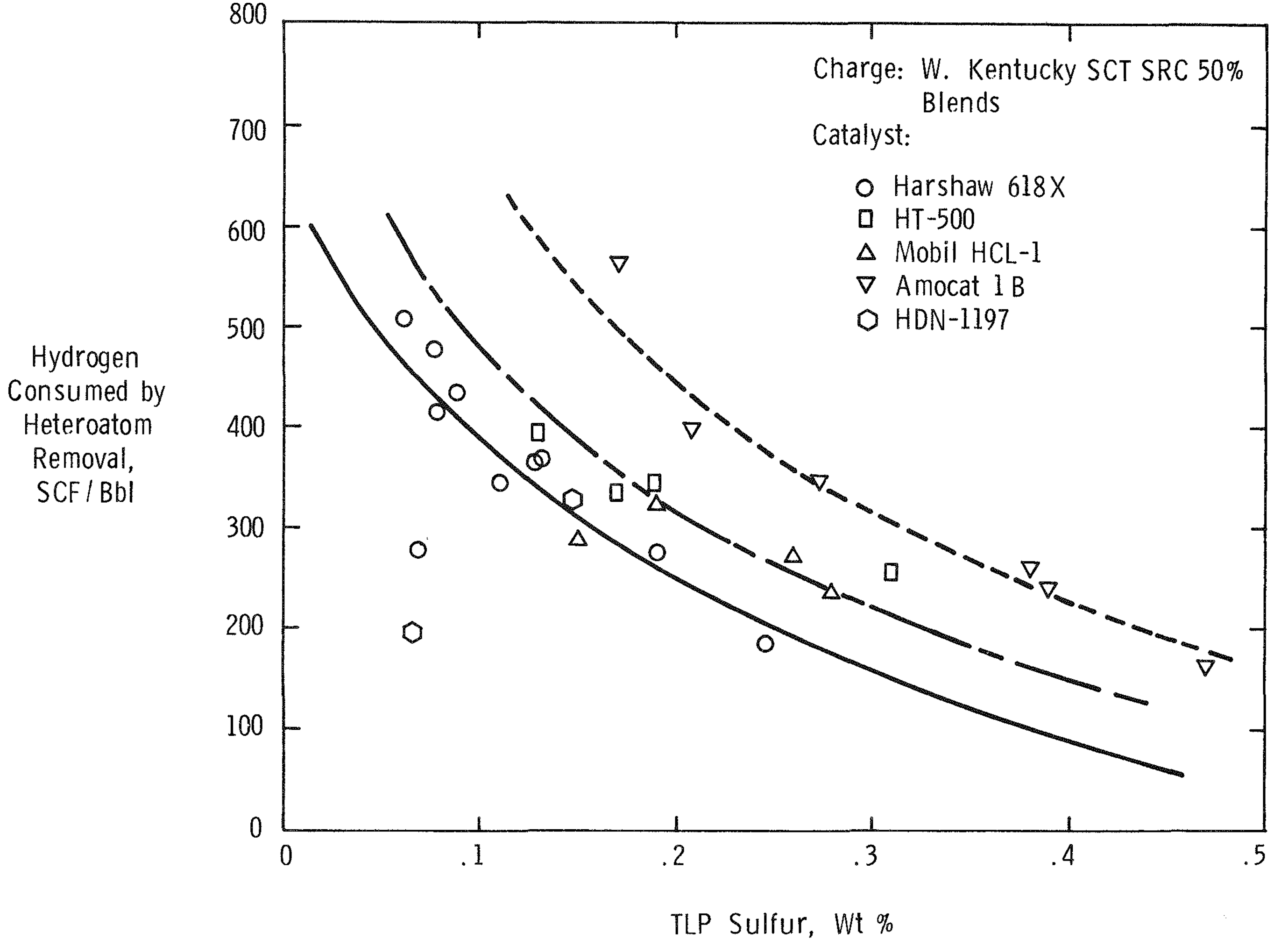


Table 3-3

ANALYSES OF FILTRATE AND PRECIPITATE OBTAINED FROM THE RUN WITH HDN-1197

	Filtrate	Precipitate
State at 77°F	Liquid	Solid
Elementary Analyses		
Gravity, °API	8.5	1.1
Hydrogen, wt %	9.32	8.21
Sulfur, wt %	NA	0.43
Nitrogen, wt %	0.46	0.82
Oxygen, wt %	1.63	2.90
CCR, wt %	9.32	19.6
Distillation, °F (D-2887)		
IBP	263	NA
10 vol %	451	NA
50 vol %	598	NA
70 vol %	780	NA
90 vol %	>1000	NA

Feed: Products taken from the line-out period of CT-146-2827-937 material balance.

NA: Not available.

TABLE 3-4

USED CATALYST PROPERTIES
AFTER HYDROPROCESSING OF W. KENTUCKY SCT SRC 50°/° BLENDS

	HARSHAW 618X	HT-500	HDS-1443	AMOCAT 1B	AMOCAT 1A	AMOCAT 1A	AMOCAT 1A
MOBIL'S ID, J-	7243	8343	9367	9392	9547	9547	9547
FRESH CATALYST							
SURFACE AREA,M2/G	140	193	335	167	154	154	154
PORE VOLUME, CC/G	0.602	0.511	0.750	0.670	0.662	0.662	0.662
PORE DIAMETER, Å	172	104	90	160	172	172	172
USED CATALYST (AS RECEIVED)							
REACTOR SECTION	WHOLE BED	WHOLE BED	WHOLE BED	WHOLE BED	TOP SECT.	MID SECT.	BTM SECT.
SURFACE AREA, M2/G	132	147	201	137	129	125	125
PORE VOLUME, CC/G	0.349	0.253	0.568	0.510	0.519	0.487	0.480
PORE DIAMETER, A	106	69	113	149	161	156	154
IRON, WT PCT	0.61	0.30	0.35	0.77	0.85	0.61	0.30
COKE, WT PCT	20.6	23.0	18.0	12.8	14.0	13.0	12.0

deposition. Since surface area measurements can include contributions by contaminants (particularly coke), these values have no clearcut meaning. Besides the coke deposition, metal deposition on the catalysts contributes to the catalyst deactivation.

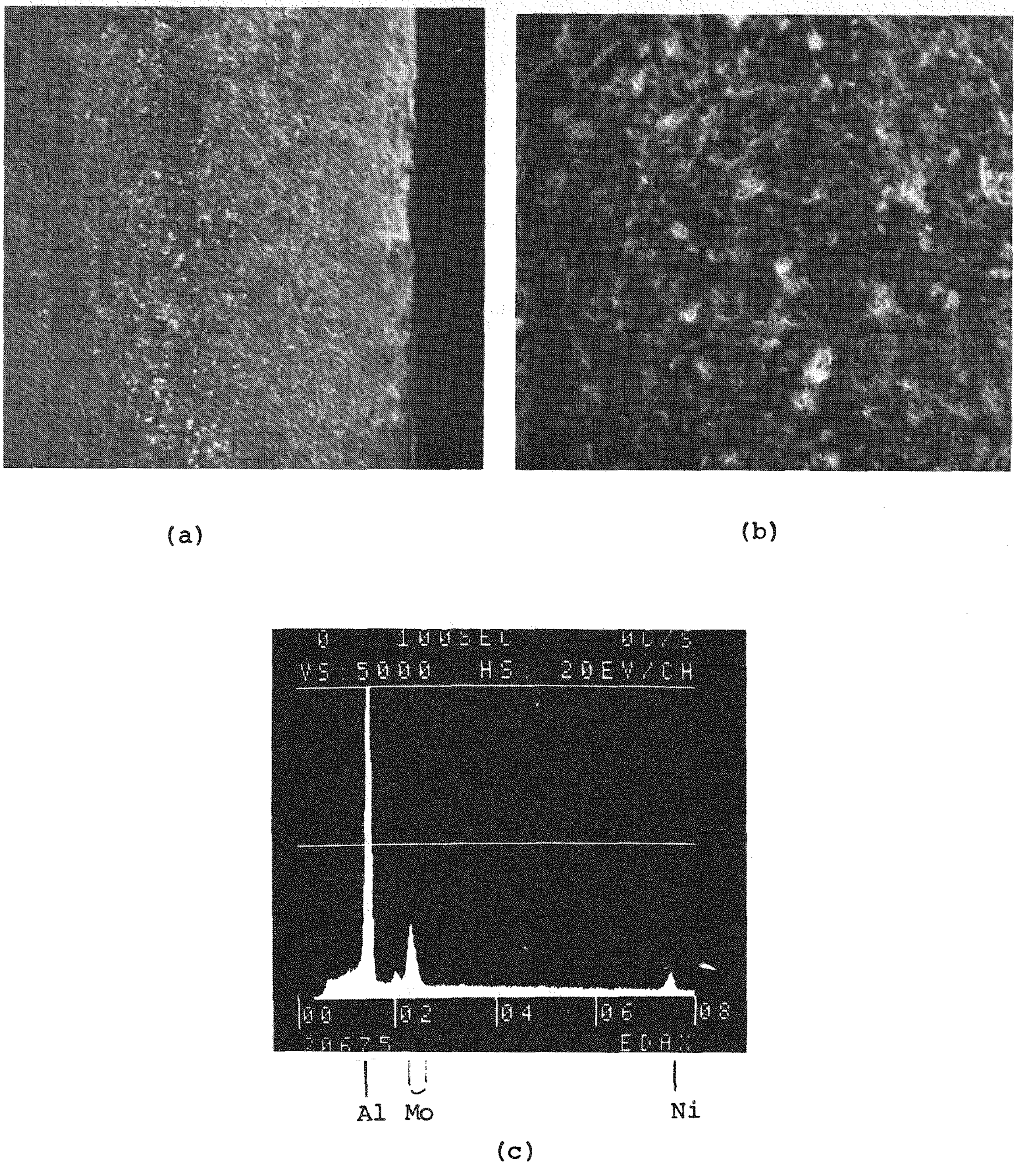
The deposition of metal contaminants on a used Harshaw 618X catalyst was analyzed with a scanning electron microscope (SEM). The SEM data and x-ray emission spectra for fresh and used catalysts at different locations in the catalyst particle are shown in Figures 3-24 through 3-27. The used catalyst, tested after our standard process, showed a large buildup of iron and titanium in a narrow band (< 2 microns) of the catalyst exterior. Other contaminants (e.g., K, Ca, Si) were detected at lower concentrations. As shown in Figure 3-25, strong intensities of deposited metals were clearly shown in the x-ray emission spectrum taken near the extrudate external surface. The intensities were substantially reduced in analyses made away from the exterior edge, as shown in Figure 3-26. No metal deposition was found in the center of the extrudate (Figure 3-27). These results indicate that the hydroprocessing of coal liquids is well suited to ebullated bed reactors; the motion of the fluidized catalysts may provide a continuous, partial regeneration by mildly abrading the metals-rich pellet exterior.

3.2 HYDROPROCESSING OF INDIANA V SRC

Table 3-5 shows the elemental and CCR analyses of two Indiana V regular SRC samples received from Wilsonville, Alabama. The second sample (high sulfur, Mobil ID 78D1323) was higher in sulfur, nitrogen, and CCR. As shown in Table 3-6, the GEC analyses of these two samples were quite different. The high sulfur sample was similar in GEC composition to a Monterey regular SRC previously analyzed (2). The "low sulfur" drum (Mobil ID 78D385) appeared to be inhomogeneous; high sulfur (0.9 wt %) samples were also found in this drum. Both samples were hydroprocessed in the pilot unit, and Table 3-7 compares some results for the hydroprocessing of the 70% blends from these two SRC's. At similar processing conditions, the products from the high sulfur sample are higher in sulfur and nitrogen and consume slightly more (50-90 scf/B) hydrogen. On heteroatom versus hydrogen selectivity plots (Figures 3-28 through 3-31), the second (high sulfur) sample shows consistently higher heteroatoms and CCR at a given hydrogen content.

When compared to results from a previous study (2) on a 70% Monterey blend, this higher sulfur Indiana V SRC sample gives products with 0.7 - 1.1 wt % lower hydrogen content and almost twice the CCR content (Table 3-8). Although the recycle solvents are somewhat different (see Table 3-9), the reason for these large differences in the

FIGURE 3-24



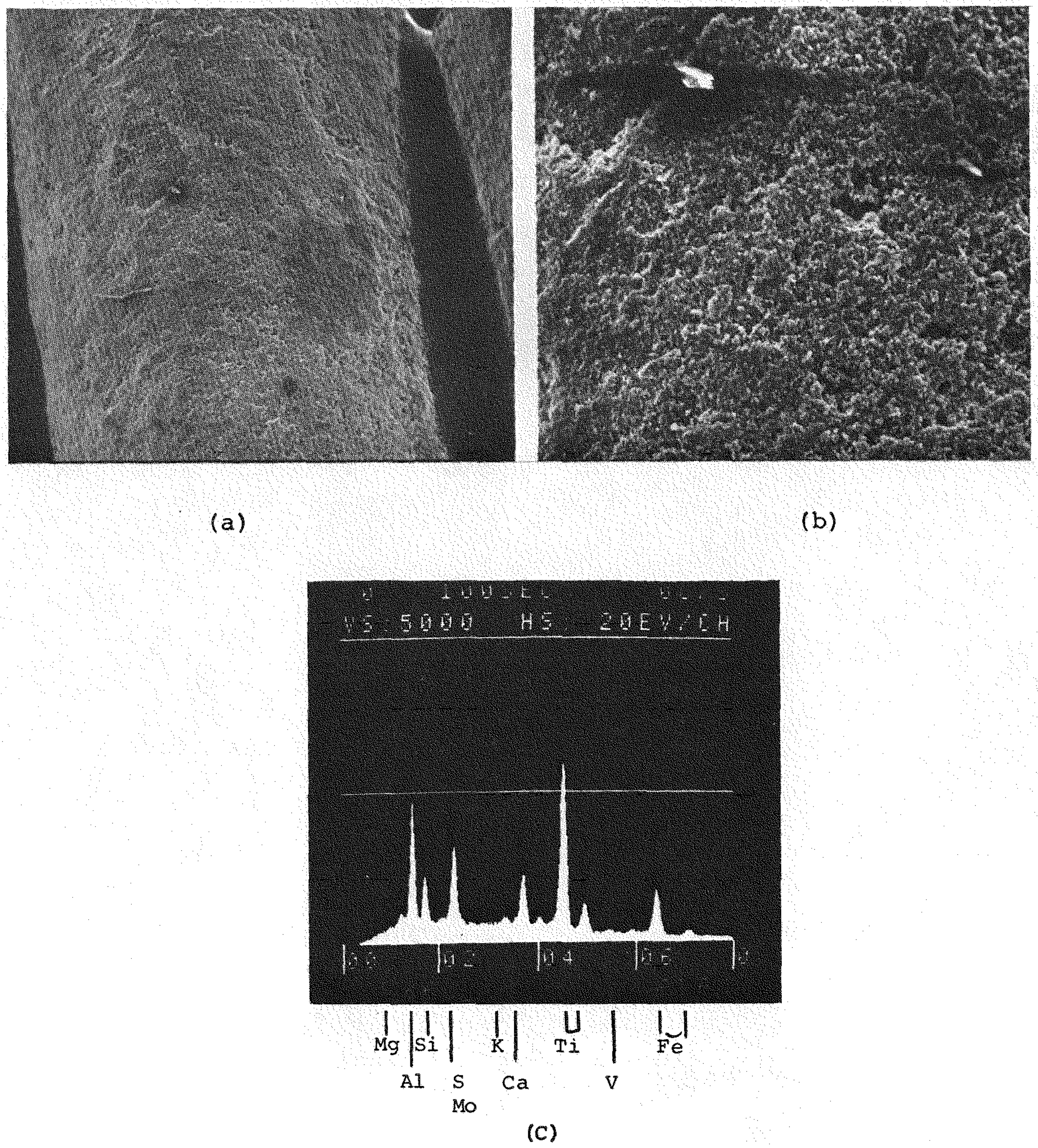
SEM Examination of Fresh Harshaw 618X

(a) X50: pellet external surface.

(b) X203: pellet external surface at center of (a).

(c) X-ray emission spectrum taken of external surface of (b), showing presence of Al, Mo and Ni.

FIGURE 3-25



(a) (b)

(C)

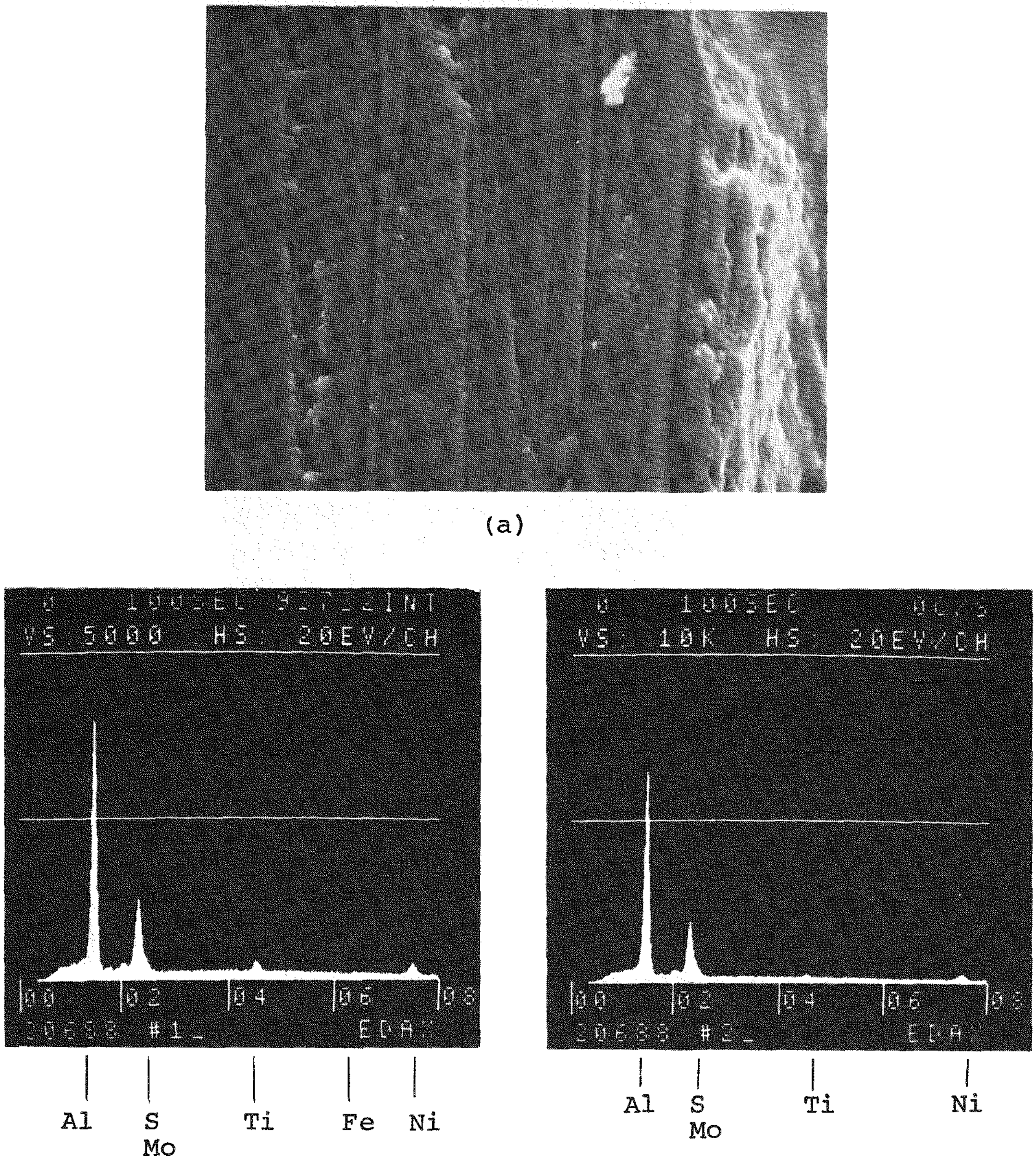
SEM Examination of Used Harshaw 618X After Hydroprocessing 50% W. Kentucky SCT SRC Blends

(a) X50: pellet external surface.

(b) X200: pellet external surface at center of (a).

(c) X-ray emission spectrum taken of external surface of (b), showing presence of Mg, Al, Mo, K, Ca, Ti, V and Fe.

FIGURE 3-26



(b)

(c)

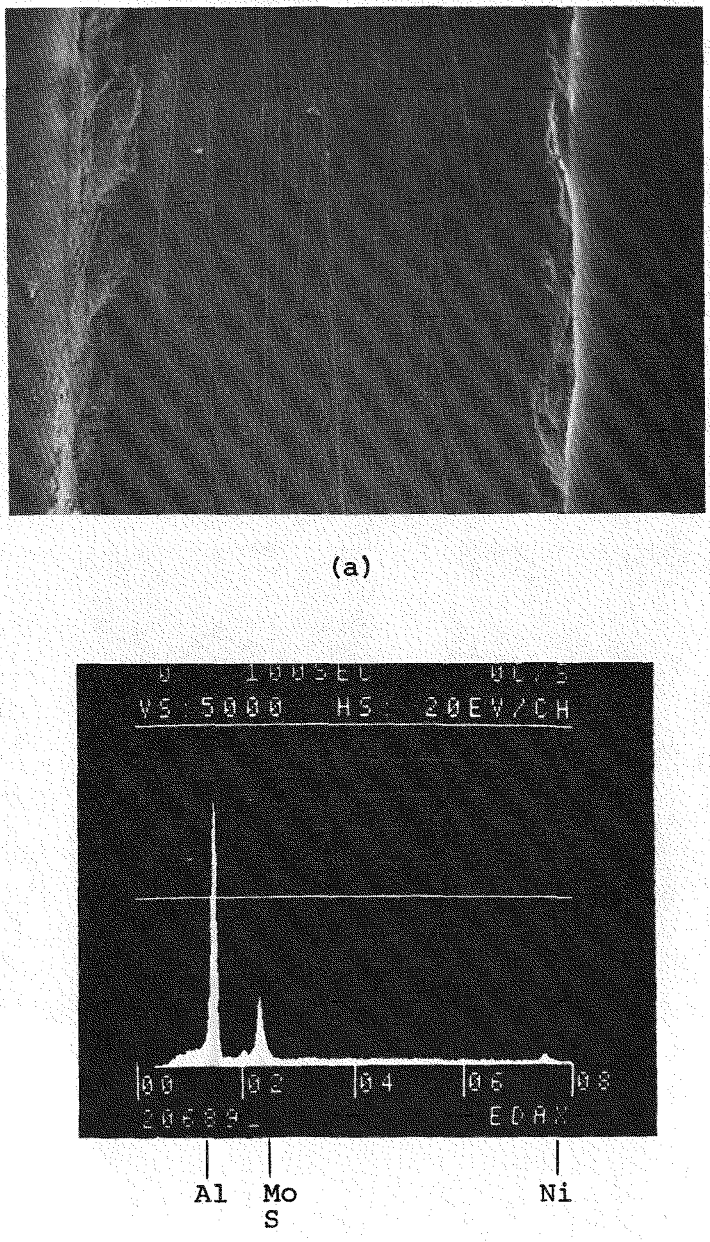
SEM Examination of Used Harshaw 618X After Hydroprocessing 50% W. Kentucky SCT SRC Blends

(a) X1000: pellet cross section of 80 μ width from edge.

(b) X-ray emission spectrum taken at 0-20 μ from the edge of the cross section showing presence of Al, Mo, Ti, Fe and Ni.

(c) X-ray emission spectrum taken at 60-80 μ from the edge of the cross section showing presence of Al, Mo, Ti and Ni.

FIGURE 3-27



(a)

(b)

SEM Examination of Used Harshaw 618X After Hydroprocessing 50% W. Kentucky SCT SRC Blends

(a) X50: pellet cross section

(b) X-ray emission spectrum taken at the center of the cross section showing presence of Al, Mo, and Ni.

Table 3-5

ANALYSES OF INDIANA V SRC

	Low Sulfur Sample (78D-385)	High Sulfur Sample (78D-1323)
Hydrogen, Wt %	5.32	5.80
Sulfur, Wt %	0.45	0.85
Oxygen, Wt %	3.8	3.3
Nitrogen, Wt %	1.3	1.97
CCR, Wt %	52.4	55.1

TABLE 3-6

GEC ANALYSES OF SRC

GEC Analyses, Wt %	Indiana V (Low Sulfur)	Indiana V (High Sulfur)	Monterey (Regular)	Monterey (SCT)
	(78D-385)	(78D-1323)	(75D-3018)	(77D-13)
Saturates (Cut 1)	0.23	0.04	0.02	0.04
Aromatic Oils (Cuts 2-4)	9.6	4.4	7.7	4.3
Resins/Asphaltenes (Cuts 5-7)	21.5	33.1	33.0	13.6
Polar Asphaltenes (Cuts 8-12)	40.4	52.7	54.1	50.5
Non-Eluted Asphaltenes (Cut 13)	28.3	9.7	5.2	31.9

TABLE 3-7

HYDROPROCESSING OF INDIANA V SRC
70% BLEND OVER HARSHAW 618X

	Charge Low-S	Charge High-S	Low-S	High-S	Low-S	High-S
OPERATING CONDITIONS						
Pressure, psig			2000			
Temperature, ^{O}F			725		800	
LHSV, Hr-1			0.87	0.81	0.50	0.48
LIQUID PRODUCT PROPERTIES						
Hydrogen, Wt %	5.92	6.44	6.49	7.2	7.86	7.85
Sulfur, Wt %	0.38	0.62	0.15	0.3	0.064	0.09
Oxygen, Wt %	3.60	3.51	2.7	3.0	1.0	1.36
Nitrogen, Wt %	1.43	1.78	1.0	1.7	0.93	1.16
CCR, Wt %	43.0	37.8	38.7	33.7	15.4	-
H_2 Cons., SCF/Bbl	-	-	650	709	1670	1860

Figure 3-28

SULFUR VS. HYDROGEN CONTENT FOR TWO INDIANA V SRC'S

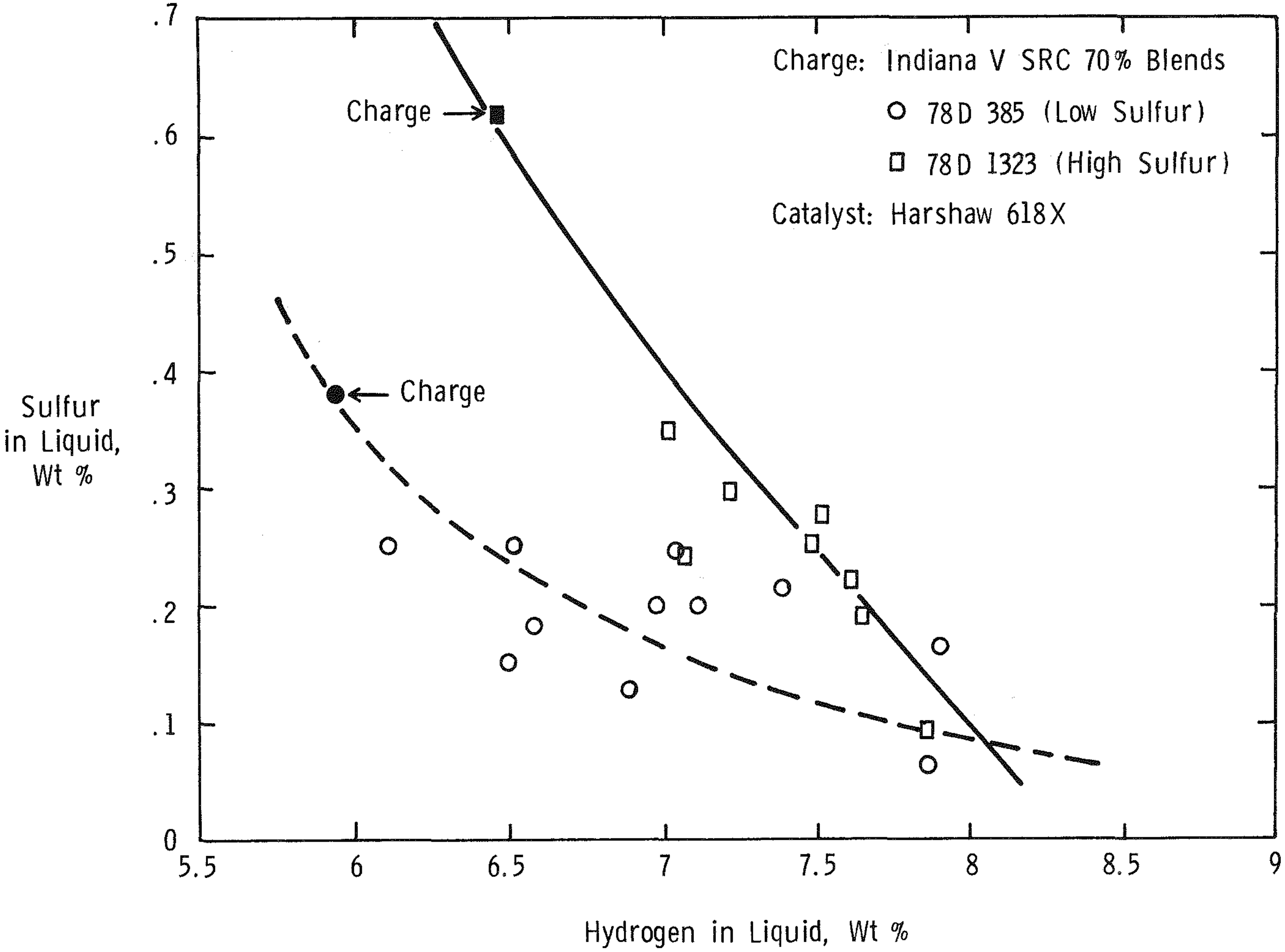


Figure 3-29

OXYGEN VS. HYDROGEN CONTENT FOR TWO INDIANA V SRC'S

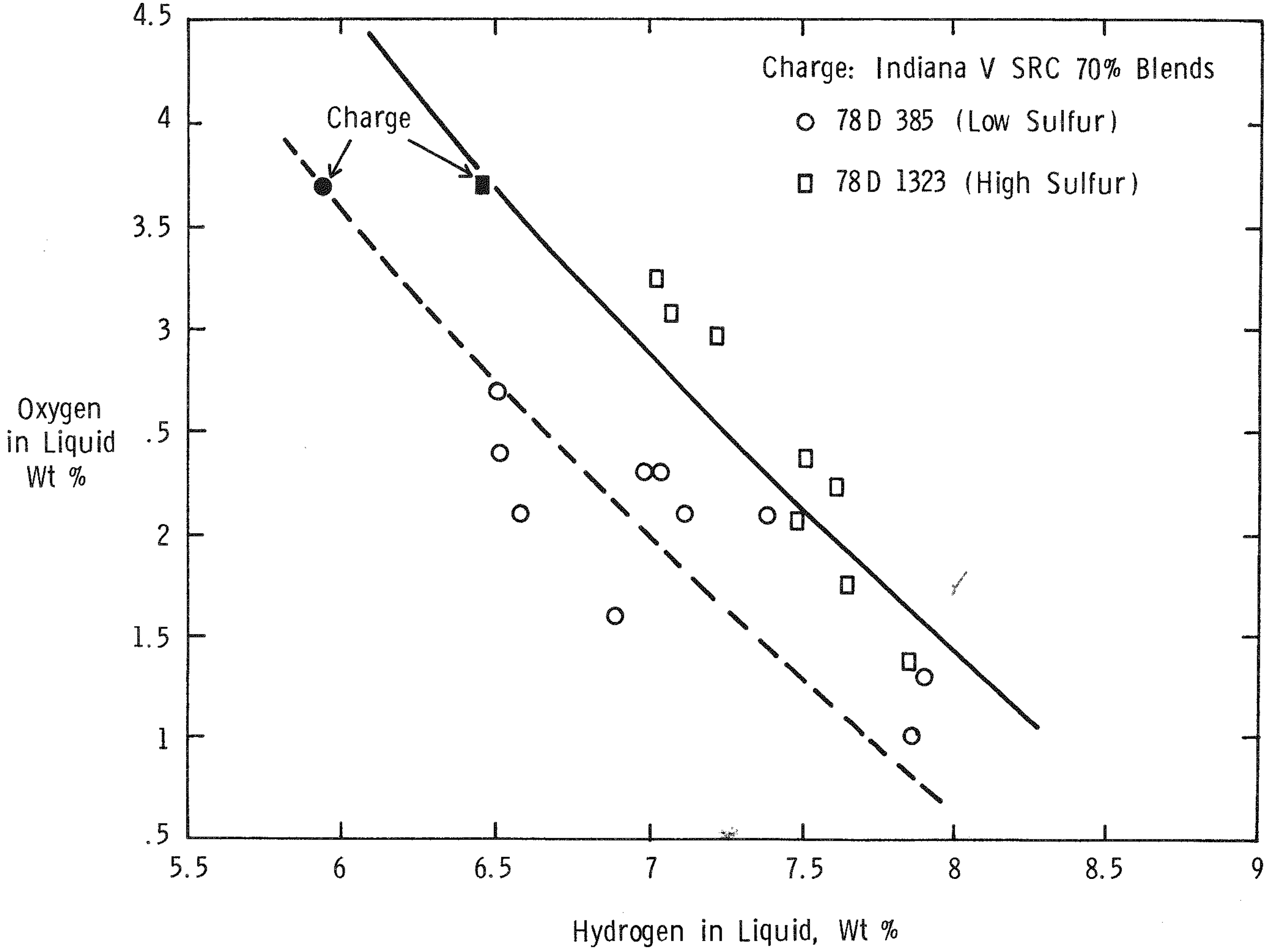


Figure 3-30

NITROGEN VS. HYDROGEN CONTENT FOR TWO INDIANA V SRC'S

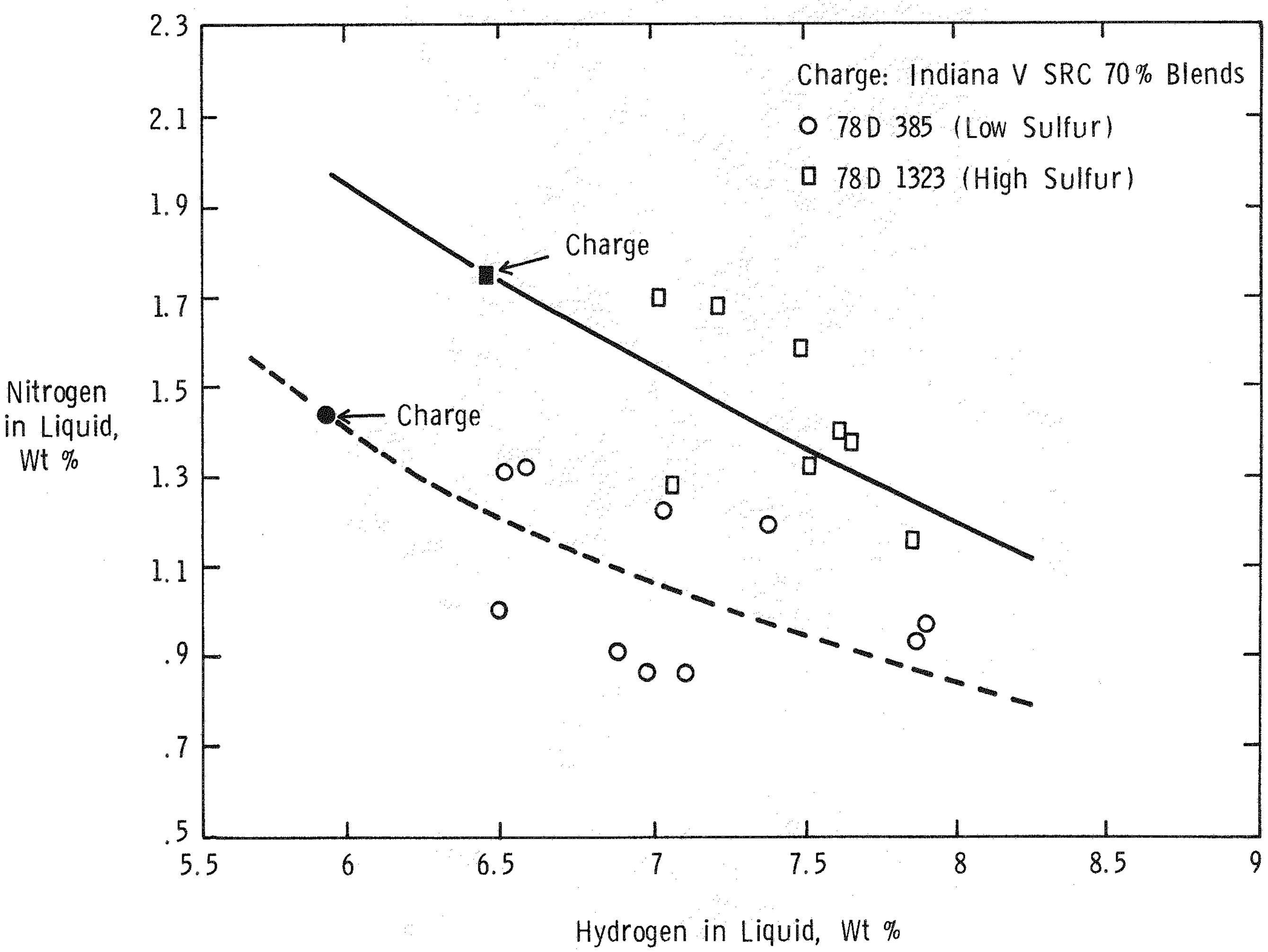


Figure 3-31

CCR VS. HYDROGEN CONTENT FOR TWO INDIANA V SRC'S

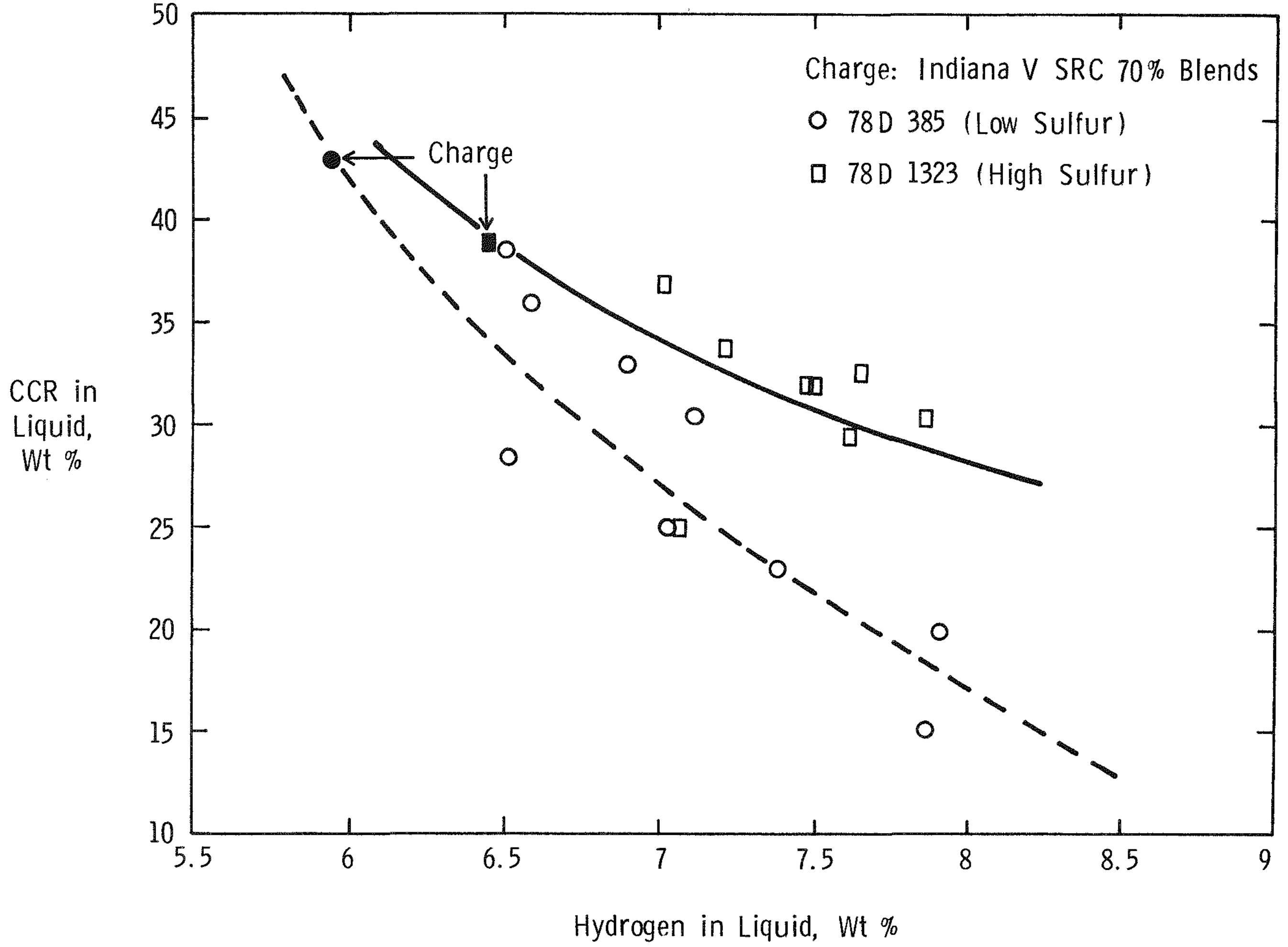


Table 3-8

HYDROPROCESSING OF INDIANA V AND MONTEREY SRC
70% BLEND OVER HARSHAW 618X

	Charge		Products			
	Indiana V	Monterey	Indiana V	Monterey	Indiana V	Monterey
Operating Conditions						
Pressure, psig			2000			
Temperaure, °F			726		775	
LHSV, HR^{-1}			0.41	0.44	0.48	0.42
Liquid Product Properties*						
Hydrogen, wt %	6.44	6.15	7.5	8.18	7.64	8.77
Sulfur, wt %	0.62	0.92	0.28	0.34	0.19	0.12
Oxygen, wt %	3.51	5.10	2.3	2.17	1.76	1.90
Nitrogen, wt %	1.78	1.65	1.3	0.95	1.3	0.85
CCR, wt %	37.81	38.17	31.0	18.4	32.5	14.2

* Solvents for two blends are different.

Table 3-9

PROPERTIES OF RECYCLE SOLVENTS USED DURING 1977 AND 1978

	77D391*	78D839**
Gravity, °API	3.8	4.1
Hydrogen, Wt %	7.42	7.72
Sulfur, Wt %	0.32	0.49
Nitrogen, Wt %	0.61	0.73
Oxygen, Wt %°	3.3	2.7
CCR, Wt %	1.1	-
Distillation, °F (D2887)		
IBP	354	433
10%	397	476
30%	441	514
50%	476	567
70%	527	614
90%	630	689
EP%	917	808

* Used with blends of Burning Star, Monterey, and Wyodak SRC's

** Used with some blends of W. Kentucky SCT and Indiana V regular SRC's products is not known. Figure 3-28 shows that a lower sulfur product can be obtained at a given hydrogen concentration with the new Indiana V sample. This means a lower hydrogen consumption would be necessary to achieve the same sulfur in the product with the Indiana V SRC.

The high desulfurization and low hydrogen consumption are shown in Table 3-10, in which elemental analyses of the 650°F$^-$ distillate and residual fuel (650°F$^+$) are given. Indiana V SRC (with 0.85 wt % sulfur) can be hydrotreated at 2000 psig, 775°F and 1.5 LHSV over Harshaw 618X to produce a 0.4 wt % sulfur residual fuel. The total hydrogen consumption is 700-900 scf/B.

Table 3-10

Analyses of Process-Generated Distillate and Residual Fuel
(Charge: 70% Indiana V Reg. SRC)

	Charge	MB-922	MB-925	MB-927	MB-928
Balance Conditions					
Catalyst		Harshaw 618X			
Pressure, psig		2000			
Temperature, °F		723	780	777	728
LHSV		0.81	1.81	0.58	0.48
H_2 Consumption, scf/B		709	588	1349	1331
TLP Properties					
Hydrogen, Wt %	6.43	7.20	6.93	7.64	7.18
Oxygen, Wt %	3.80	2.97	3.08	1.75	1.37
Nitrogen, Wt %	1.72	1.68	1.28	1.38	1.16
Sulfur, Wt %	0.620	0.299	0.242	0.191	0.094
Distillate (650°F$^-$) Properties					
Yield, Wt %	23.0	22.5	29.9	29.5	28.6
Oxygen, Wt %	2.88	1.97	1.93	1.30	1.13
Nitrogen, Wt %	0.594	0.521	0.579	0.528	0.481
Sulfur, Wt %	0.40	0.067	0.057	0.034	0.005
Residual Fuel (650°F$^+$) Properties					
Oxygen, Wt %	2.95	2.49	2.02	1.47	1.33
Sulfur, Wt %	0.90	0.40	0.36	0.20	0.13
Nitrogen Wt %	2.34	1.25	1.22	1.37	1.34

Section 4

CATALYST AGING STUDIES

Three hydrogen-efficient desulfurization catalysts identified in Section 3 - Mobil HCL-2, Amocat 1A, and Cyanamid HDS-1443 - were selected for catalyst aging studies. The aging studies were conducted in our fixed bed pilot unit at constant reactor temperature and liquid (SRC blends) flow rates so that the data could be transformed to simulate ebullated bed reactor operation. An aging model was developed to fit the data, and the model was subsequently used to simulate process conditions required to produce a low sulfur residual fuel (0.4 wt % in 900°F$^+$) from regular SRC or SCT SRC. In the process simulations both fixed bed and ebullated bed reactors were examined.

4.1 DERIVATION OF MODELS

Catalyst Deactivation Model

A simple model was used to determine the catalyst deactivation rate based on constant temperature rate data. The model assumes that catalyst deactivation is independent of reactants and products in the system and that the deactivation is first order with respect to its activity:

$$\frac{dk}{dt} = -\frac{k}{\tau} \qquad (4\text{-}1)$$

where k is the reactant rate constant of the catalyst, τ is the deactivation time constant, and t is the age of the catalyst.

Integration of Equation 4-1 with an initial rate constant, k_f, at t=0 yields:

$$k = k_f\, e^{-t/\tau} \qquad (4\text{-}2)$$

The model further assumes second order reaction for heteroatom removal and CCR reduction. Incorporating with catalyst deactivation, the rate equation for heteroatom removal (or CCR reduction) is:

$$-\frac{dC_A}{d(1/LHSV)} = k_o\, e^{-E/RT}\, e^{-t/\tau}\, C_A^2 \qquad (4\text{-}3)$$

where k_o is the fresh pre-exponential factor, LHSV is liquid hourly space velocity, T is reaction temperature, E is activation energy, and C_A is heteroatom (or CCR) concentration.

For a fixed bed reactor, the kinetic equation derived from Equation 4-3 is:

$$\frac{1}{C_A} - \frac{1}{C_{A_o}} = \frac{k_o\, e^{-E/RT}\, e^{-t/\tau}}{LHSV} \qquad (4\text{-}4)$$

where C_{A_o} is the initial concentration of reactant (i.e., heteroatom or CCR). The catalyst deactivation time constant, τ, and the activation energy, E, can be calculated from the material balances of the constant temperature aging run and the catalyst activity measurements at different temperatures, respectively.

Process Modeling

Fixed Bed Reactor. In the commercial operation of a fixed bed reactor, the reactor temperature is gradually increased to compensate for catalyst deactivation so that a constant sulfur concentration in the product is obtained. To design or simulate the fixed bed reactor Equation 4-4 can be used directly.

Ebullated or Fluidized Bed Reactor. For an ebullated or fluidized bed reactor, catalyst activity is maintained by continuous addition of fresh catalyst along with a removal of similar amounts of aged catalyst. To simplify the kinetic model, the catalyst is assumed to be well-stirred. The two limiting cases of plug flow and mixed flow are considered for the liquid phase (i.e., SRC).

Average Catalyst Activity. The average catalyst activity is defined as:

$$\bar{k} = \int_0^\infty k\, g(t)\, dt \qquad (4\text{-}5)$$

where k is the present catalyst rate constant, and g(t) is the catalyst age distribution.

For mixed flow of the catalyst, the age distribution is:

$$g(t) = \frac{1}{\bar{t}_c} e^{-t/\bar{t}_c} \tag{4-6}$$

$$\bar{t}_c = W_r/W_c \tag{4-7}$$

where $\bar{t}_c$ = catalyst space time
W_r = weight of catalyst in the reactor
W_c = catalyst make-up rate, weight per unit time

For any catalyst particle the present catalyst activity is given in Equation 4-2:

$$k = k_f\ e^{-t/\tau} \tag{4-2}$$

By introducing Equations 4-2, 4-6, and 4-7 into Equation 4-5 and then integrating, the average catalyst activity in the reactor is:

$$\bar{k} = \frac{k_f}{1 + (\bar{t}_c/\tau)} \tag{4-8}$$

Plug Flow Model of the Liquid Phase. Incorporation of the average catalyst activity into the rate equation yields:

$$-\frac{dC_A}{d(1/\mathrm{LHSV})} = \frac{k_o\ e^{-E/RT}\ C_A^{\ 2}}{1 + (\bar{t}_c/\tau)} \tag{4-9}$$

For the plug flow model of the liquid phase, the overall second order kinetic equation becomes:

$$\mathrm{LHSV}\ \left(\frac{1}{C_A} - \frac{1}{C_{A_o}}\right) = \frac{k_o\ e^{-E/RT}}{1 + (\bar{t}_c/\tau)} \tag{4-10}$$

The fresh catalyst make-up rate expressed in terms of pounds per ton of SRC becomes:

$$\text{make-up} = \frac{\rho_c}{\rho_{bl}\ X_{SRC}} \cdot \frac{2000}{\bar{t}_c \cdot \mathrm{LHSV}} \tag{4-11}$$

$$\text{make-up} = \frac{2000}{\bar{t}_c \cdot \text{WHSV}} \qquad (4\text{-}12)$$

where ρ_c = catalyst packed density, lbs/ft^3

ρ_{bl} = density of SRC blend, lbs/ft^3

X_{SRC}= weight fraction of SRC in blend

LHSV= liquid phase hourly space velocity based on volumetric flow rate of SRC blends, hr^{-1}

WHSV= liquid phase hourly space velocity based on gravimetric flow rate of SRC only, hr^{-1}

Mixed Flow Model of the Liquid Phase. For the mixed flow model the overall kinetic expression for a second order reaction is:

$$\text{LHSV}\left[\frac{C_{A_o} - C_A}{C_A^2}\right] = \frac{k_o\, e^{-E/RT}}{1 + (\bar{t}_c/\tau)} \qquad (4\text{-}13)$$

To calculate fresh catalyst make-up rate, Equation 4-12 can be used.

4.2 AGING RUN OF 70% INDIANA V SRC OVER MOBIL HCL-2

The material balances in which a 70% (high sulfur) Indiana V regular SRC blend was hydroprocessed over Mobil HCL-2 are given in Tables 4-1 and 4-2. The run was operated smoothly for 17 days and was then terminated due to incipient plugging in the reactor. The operating conditions were as follows: 2000 psi, 775°F, and 0.5 LHSV. The total liquid product sulfur content ranged from 0.15 wt % at the start of run to 0.36 wt % at the end of run. From these material balances the kinetic constants (k_o, E, τ) in Equation 4-4 were fit for heteroatom removal (and CCR reduction). The estimated constants, shown with the range of product heteroatom concentrations, are:

	k_o	E	τ	C_A Range	mean deviation
	$\left[\frac{1}{\text{wt \%-hr}}\right]$	$\left[\frac{\text{Btu}}{\text{lb mole}}\right]$	[Days]	[wt %]	[wt %]
Sulfur	1.052×10^{10}	53,700	10.6	0.13-0.36	0.02
Nitrogen	1.050×10^{5}	34,200	17.0	1.3 -1.8	0.08
Oxygen	1.368×10^{13}	77,800	10.4	1.0 -2.5	0.15
CCR	2.097×10^{1}	18,400	10.4	22-37	1.90

TABLE 4-1

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-2
PROGRAM: 2893 UNIT: 146

	CHARGE	MB-988	MB-989	MB-990	MB-991	MB-992	MB-993	MB-994	MB-995
OPERATING CONDITIONS									
PRESSURE, PSIG	-	1975	1975	1975	1975	1975	1975	1975	1990
AVG REACTOR TEMP., °F	-	750	796	755	772	776	776	774	774
LHSV VFF/HR/VCAT	-	0.46	0.46	0.46	0.47	0.48	0.47	0.47	0.47
LIQUID PRODUCT PROPERTIES									
GRAVITY, API	-13.1	-5.6	-1.9	-3.9	-5.3	-5.8	-6.3	-7.2	-7.4
HYDROGEN, WT PCT	6.35	7.50	7.54	7.22	7.01	6.33	6.87	7.03	6.64
SULFUR, WT PCT	0.750	0.244	0.147	0.153	0.189	0.208	0.217	0.235	0.257
NITROGEN, WT PCT	1.80	1.36	1.24	1.30	1.53	1.47	1.60	1.45	1.50
OXYGEN, WT PCT	3.20	1.80	0.98	1.80	1.50	1.70	1.70	2.04	2.07
CCR, WT PCT	42.00	25.15	22.99	22.08	25.37	29.28	29.64	30.72	30.67
K.V.(100 C)	-	414.60	21.06	89.58	201.10	226.90	293.60	245.54	309.00
HETEROATOM REMOVAL									
DESULFURIZATION, PCT WT	-	68.0	81.3	80.3	75.6	73.3	72.0	69.5	66.8
DENITROGENATION, PCT WT	-	23.9	32.6	28.6	15.9	19.7	11.9	19.7	17.5
DEOXYGENATION, PCT WT	-	42.9	69.8	43.9	53.2	47.3	46.9	35.9	35.3
CCR REMOVAL, PCT WT	-	41.8	48.3	49.8	42.3	33.8	32.4	29.6	30.1
HYDROGEN CONSUMPTION, SCF/B									
TOTAL H2 CONSUMPTION	-	1092	1387	1036	810	273	685	801	471
H2 CONSUMED BY C1-C5	-	60	377	296	236	250	241	218	254
H2 CONSUMED BY C6+	-	805	671	493	337	-207	239	390	33
H2 CONSUMED BY S	-	25	30	30	28	27	26	26	25
H2 CONSUMED BY N	-	71	97	85	47	58	35	58	52
H2 CONSUMED BY O	-	131	213	134	162	144	143	109	108

TABLE 4-1 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-2
PROGRAM: 2893 UNIT: 146

	CHARGE	MB- 996	MB- 997	MB- 998	MB- 999	MB-1000	MB-1001	MB-1002	MB-1003
OPERATING CONDITIONS									
PRESSURE, PSIG	-	1990	197	1975	1980	1977	1985	1990	1975
AVG REACTOR TEMP., °F	-	774	774	774	774	774	773	771	772
LHSV VFF/HR/VCAT	-	0.47	0.47	0.46	0.40	0.42	0.40	0.39	0.37
LIQUID PRODUCT PROPERTIES									
GRAVITY, API	¯13.1	¯7.9	¯7.9	¯8.6	¯8.5	¯7.9	¯9.6	¯8.6	¯9.9
HYDROGEN, WT PCT	6.35	6.34	6.58	6.76	6.93	6.59	6.42	6.85	6.69
SULFUR, WT PCT	0.750	0.252	0.292	0.320	0.271	0.278	0.260	0.310	0.360
NITROGEN, WT PCT	1.80	1.57	1.48	1.51	1.41	1.47	1.55	1.46	1.73
OXYGEN, WT PCT	3.20	2.33	2.04	2.00	2.13	2.02	2.15	2.29	2.51
CCR, WT PCT	42.00	32.23	32.73	31.32	32.35	29.51	34.86	31.13	37.04
K.V.(100 C)	-	440.90	302.60	806.00	860.50	566.80	803.40	1500.00	9113.00
HETEROATOM REMOVAL									
DESULFURIZATION, PCT WT	-	67.4	62.4	58.9	65.3	64.5	66.7	60.0	53.5
DENITROGENATION, PCT WT	-	13.3	18.7	24.3	29.4	26.6	22.5	26.4	12.8
DEOXYGENATION, PCT WT	-	27.0	36.4	45.1	41.7	44.9	41.2	36.9	30.8
CCR REMOVAL, PCT WT	-	26.3	25.6	27.3	25.1	31.9	19.3	27.4	13.6
HYDROGEN CONSUMPTION, SCF/B									
TOTAL H2 CONSUMPTION	-	459	482	823	985	715	532	870	744
H2 CONSUMED BY C1-C5	-	264	286	316	349	354	314	289	354
H2 CONSUMED BY C6+	-	49	7	245	368	91	¯27	341	220
H2 CONSUMED BY S	-	25	23	22	25	24	25	23	20
H2 CONSUMED BY N	-	39	55	80	97	88	74	87	42
H2 CONSUMED BY O	-	82	111	159	147	158	145	130	108

TABLE 4-2

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
YIELDS AND DISTILLATION
CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-2
PROGRAM: 2893 UNIT: 146

	MB-988	MB-989	MB-990	MB-991	MB-992	MB-993	MB-994	MB-995
MATERIAL BALANCE, PCT WT	99.2	105.2	98.7	99.3	100.4	99.2	98.2	99.0
OPERATING CONDITIONS								
PRESSURE, PSIG	1975	1975	1975	1975	1975	1975	1975	1990
AVG REACTOR TEMP., °F	750	796	755	772	776	776	774	774
LHSV VFF/HR/VCAT	0.46	0.46	0.46	0.47	0.48	0.47	0.47	0.47
H2 CHG, SCFB FF NLB	7034	6934	5912	5635	5098	5310	6102	6711
CATALYST AGE, DAYS	3.1	3.9	4.7	5.7	6.7	7.6	8.7	9.7
YIELDS (BASED ON CHARGE)								
TOT C1-C3, WT PCT	0.28	1.93	1.52	1.19	1.26	1.21	1.09	1.26
TOT C4, WT PCT	0.13	0.43	0.32	0.29	0.29	0.29	0.26	0.33
TOT C5, WT PCT	0.01	0.02	0.02	0.02	0.02	0.01	0.02	0.02
C6+ PRODUCT, WT PCT	98.43	95.62	96.70	96.74	96.12	96.89	97.43	96.86
H2S, WT PCT	0.54	0.65	0.64	0.60	0.58	0.57	0.55	0.53
NH3, WT PCT	0.51	0.70	0.61	0.34	0.42	0.25	0.42	0.37
H20, WT PCT	1.50	2.44	1.53	1.86	1.65	1.64	1.25	1.23
DISTILLATION (D2887),°F								
IBP	258	340	220	324	317	335	342	335
5 PCT VOL	469	453	445	431	460	451	455	441
10 '' ''	509	500	498	475	501	496	492	483
30 '' ''	668	626	648	595	624	641	624	624
50 '' ''	-	-	-	1006	-	-	-	-
70 '' ''	-	-	-	-	-	-	-	-
90 '' ''	-	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-

TABLE 4-2 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
YIELDS AND DISTILLATION
CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-2
PROGRAM: 2893 UNIT: 146

	MB- 996	*MB-* 997	*MB-* 998	*MB-* 999	*MB-*1000	*MB-*1001	*MB-*1002	*MB-*1003
MATERIAL BALANCE, PCT WT	99.0	100.0	98.3	101.2	100.3	104.8	100.9	100.7
OPERATING CONDITIONS								
PRESSURE, PSIG	1990	197	1975	1980	1977	1985	1990	1975
AVG REACTOR TEMP., °F	774	774	774	774	774	773	771	772
LHSV VFF/HR/VCAT	0.47	0.47	0.46	0.40	0.42	0.40	0.39	0.37
H2 CHG, SCFB FF NLB	7052	7655	7539	8197	8268	7493	9067	9145
CATALYST AGE, DAYS	10.7	11.6	12.6	13.4	13.9	14.4	15.1	16.1
YIELDS (BASED ON CHARGE)								
TOT C1-C3, WT PCT	1.34	1.44	1.55	1.64	1.75	1.58	1.44	1.71
TOT C4, WT PCT	0.32	0.34	0.38	0.58	0.41	0.33	0.33	0.46
TOT C5, WT PCT	0.01	0.01	0.02	0.02	0.02	0.01	0.01	0.03
C6+ PRODUCT, WT PCT	97.16	96.66	96.26	96.15	95.81	96.06	96.77	96.80
H2S, WT PCT	0.54	0.50	0.47	0.52	0.51	0.53	0.48	0.43
NH3, WT PCT	0.29	0.40	0.57	0.69	0.62	0.52	0.62	0.30
H2O, WT PCT	0.94	1.27	1.78	1.65	1.77	1.63	1.46	1.22
DISTILLATION (D2887),°F								
IBP	346	362	296	328	372	-	-	-
5 *PCT VOL*	459	464	479	470	483	-	-	-
10 '' ''	500	495	511	515	526	-	-	-
30 '' ''	656	624	636	694	698	-	-	-
50 '' ''	-	-	-	-	-	-	-	-
70 '' ''	-	-	-	-	-	-	-	-
90 '' ''	-	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-

The temperature rise necessary to maintain a constant product sulfur concentration is approximately 5°F/day. The measured and model-predicted values for sulfur and nitrogen contents are shown in Figure 4-1; comparable values for oxygen and CCR concentrations are shown in Figure 4-2. As shown in these two figures, close agreement between measured and model-predicted data has been achieved. These kinetic constants were used to simulate process conditions for producing a low sulfur residual fuel; i.e., 0.4 wt % of sulfur remaining in the 900°F$^+$ fraction, using a 70% Indiana V SRC as feedstock. Both fixed bed reactor and ebullated bed reactors were examined.

For the operation of a fixed bed reactor, the reactor temperature is gradually increased to compensate for catalyst deactivation so that a constant sulfur concentration in the product is obtained. To design the fixed bed reactor Equation 4-4 can be used directly. It is estimated that the total liquid products of 0.3 wt % sulfur should give a 900°F$^+$ sulfur content of 0.4 wt % (cf. Appendix D). Figure 4-3 shows the reactor temperature required to produce a 0.3 wt % sulfur TLP as a function of days on-stream for LHSV of 0.1, 0.3, and 0.5. The cycle length, a function of both LHSV and the limit of reactor temperature, can be determined from Figure 4-3. For example, at 0.3 LHSV and a maximum reactor temperature of 850°F the cycle length is 30 days. However, better performance (longer cycle time) may be expected, since no temperature correction of the catalyst deactivation has been made in Equation 4-4. The current catalyst deactivation constants are obtained at a reactor temperature of 775°F, which approximates the midpoint condition in a fixed bed run.

For the operation of an ebullated bed reactor, the reactor temperature is kept essentially constant; catalyst activity is maintained by the continuous addition of fresh catalyst along with removal of similar amounts of aged catalyst. As discussed previously, for an ebullated bed reactor the liquid phase can be operated in plug flow or well-mixed flow regime. The relation between SRC space velocity and the fresh catalyst make-up rate for producing a total liquid product sulfur level of 0.3 wt % is shown in Figure 4-4 for 775°F and 800°F.

As shown in Figure 4-4 the ebullated bed reactor should, in principle, be operated in the plug flow regime. In practice, catalyst ebullation enables a more stable reactor operation; e.g., minimal temperature gradients. In the H-Coal® reactor for coal hydroliquefaction, internal recycling of the coal liquid is used to maintain catalyst ebullation. Therefore, the kinetic performance of the H-Coal® reactor operated with a high recycle ratio (10:1), may be very close to the mixed

Figure 4-1

AGING RUN WITH INDIANA V SRC

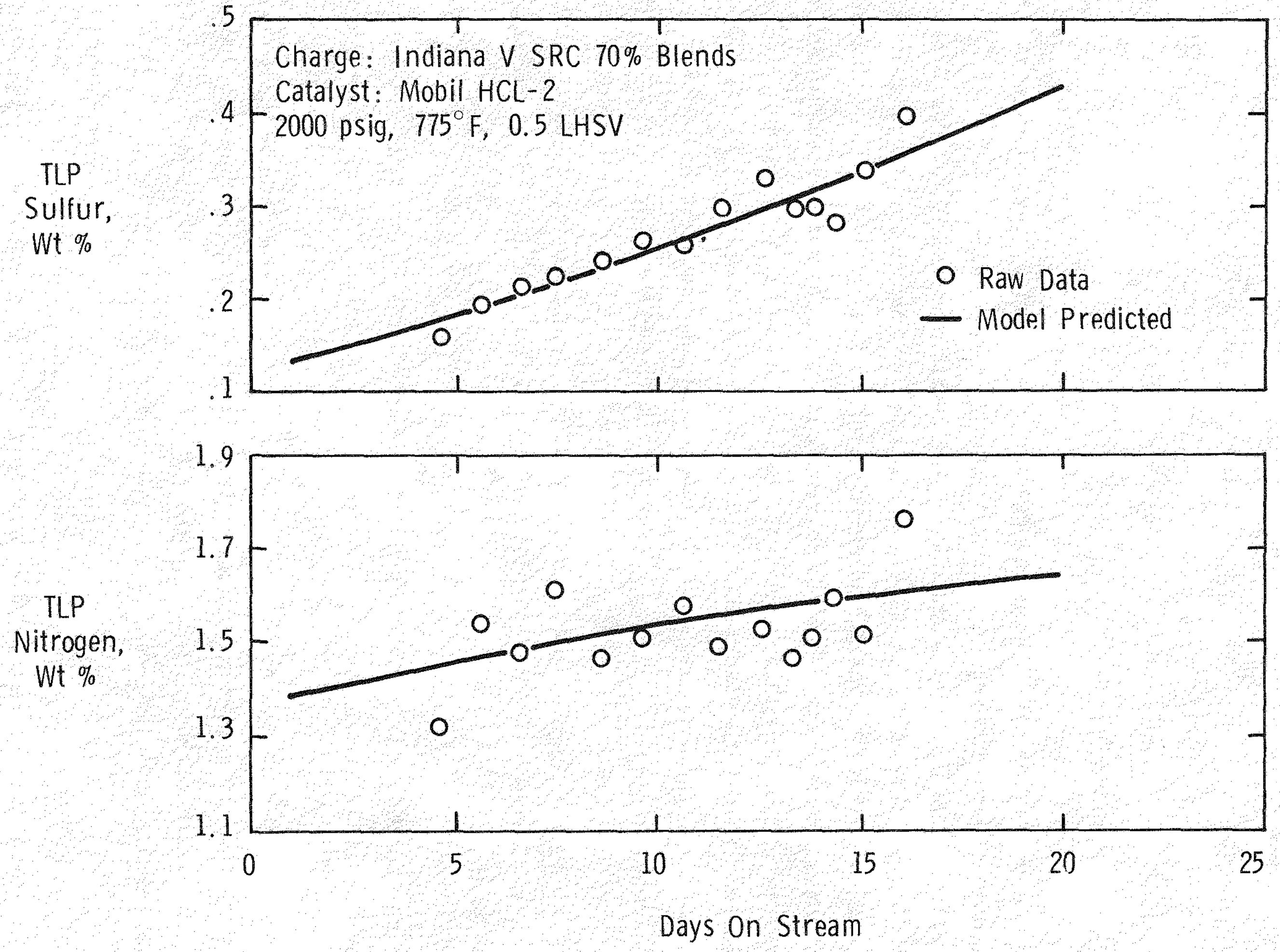


Figure 4-2

AGING RUN WITH INDIANA V SRC

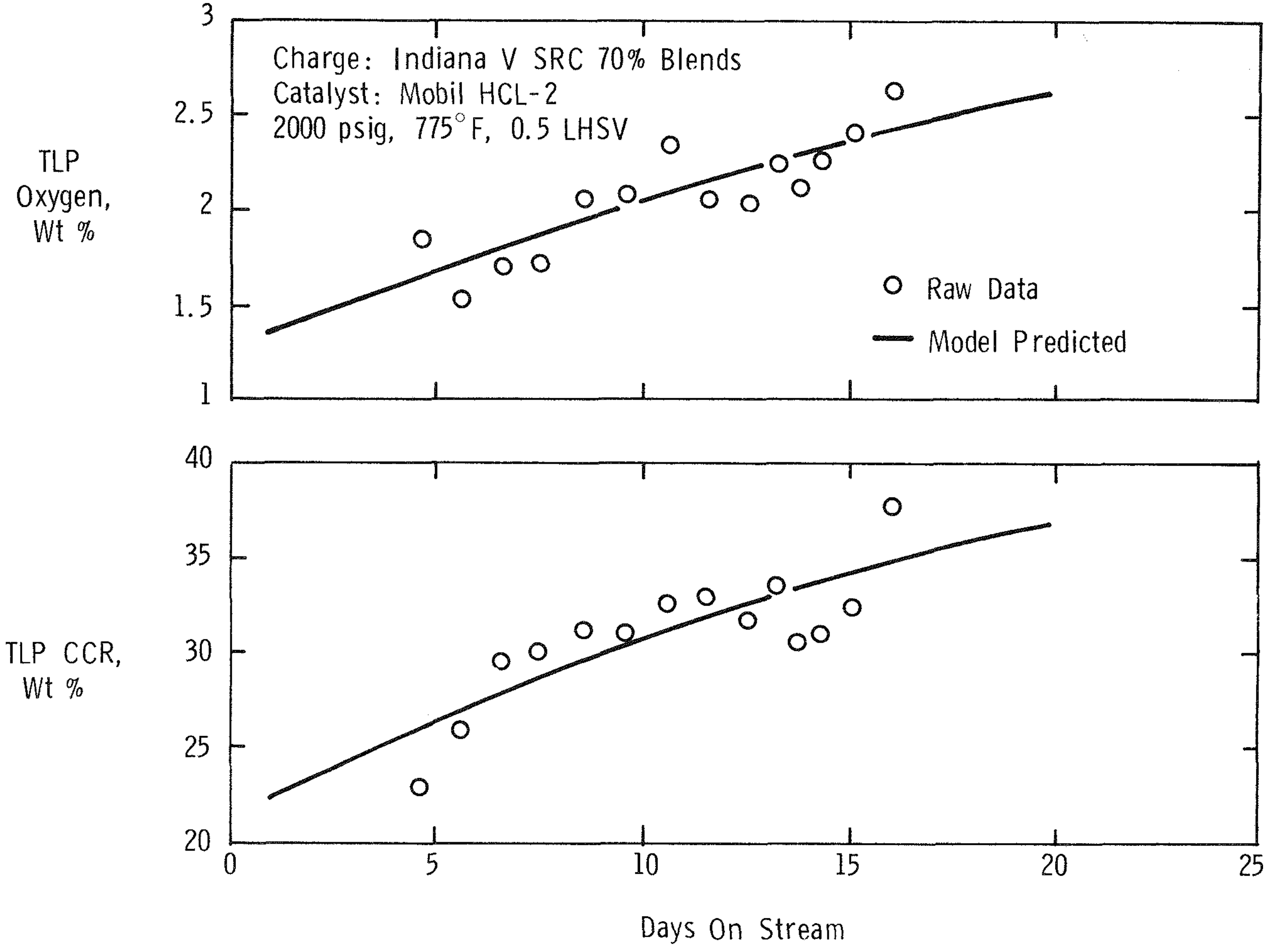


Figure 4-3

DESIGN VARIABLES FOR FIXED BED REACTOR

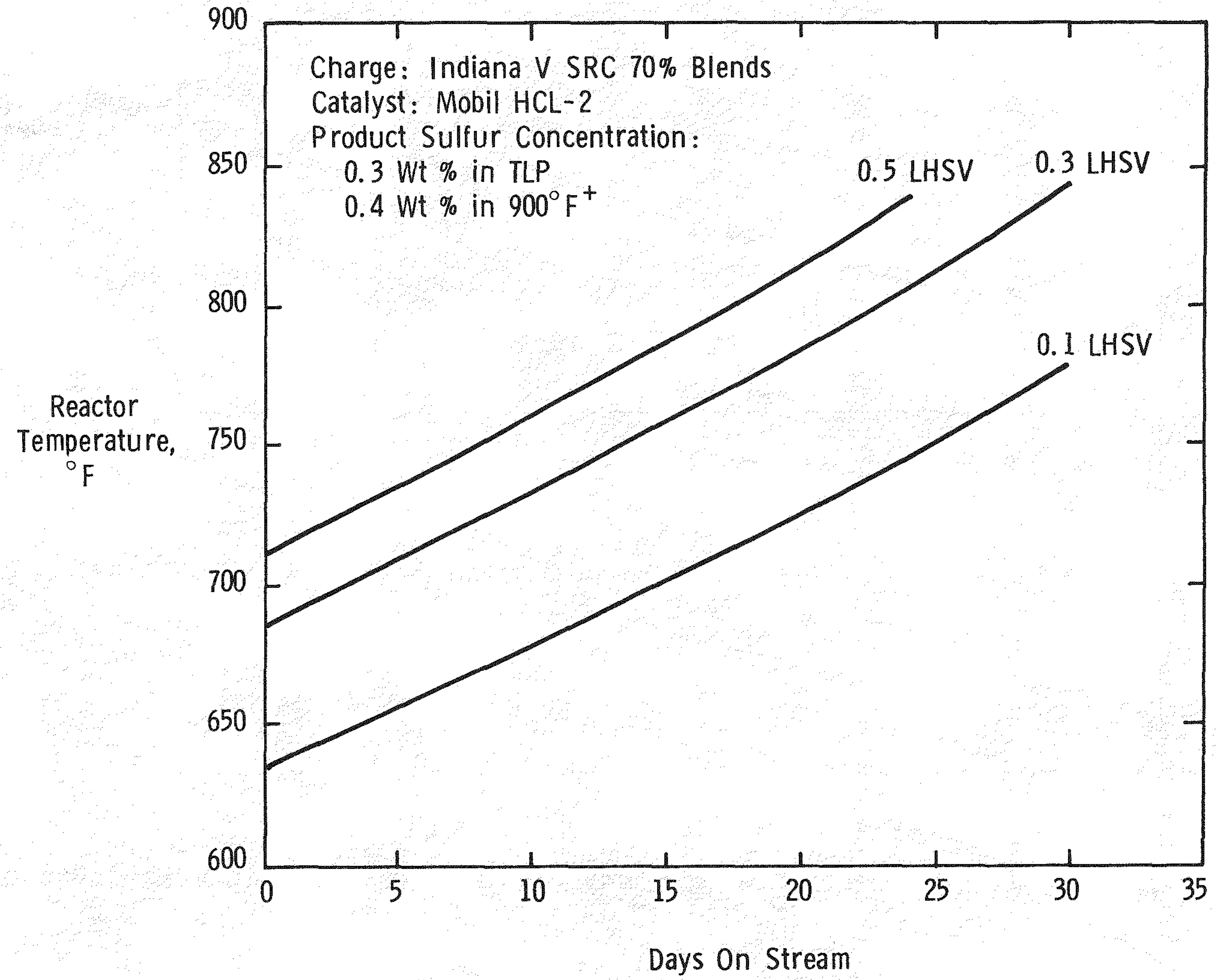
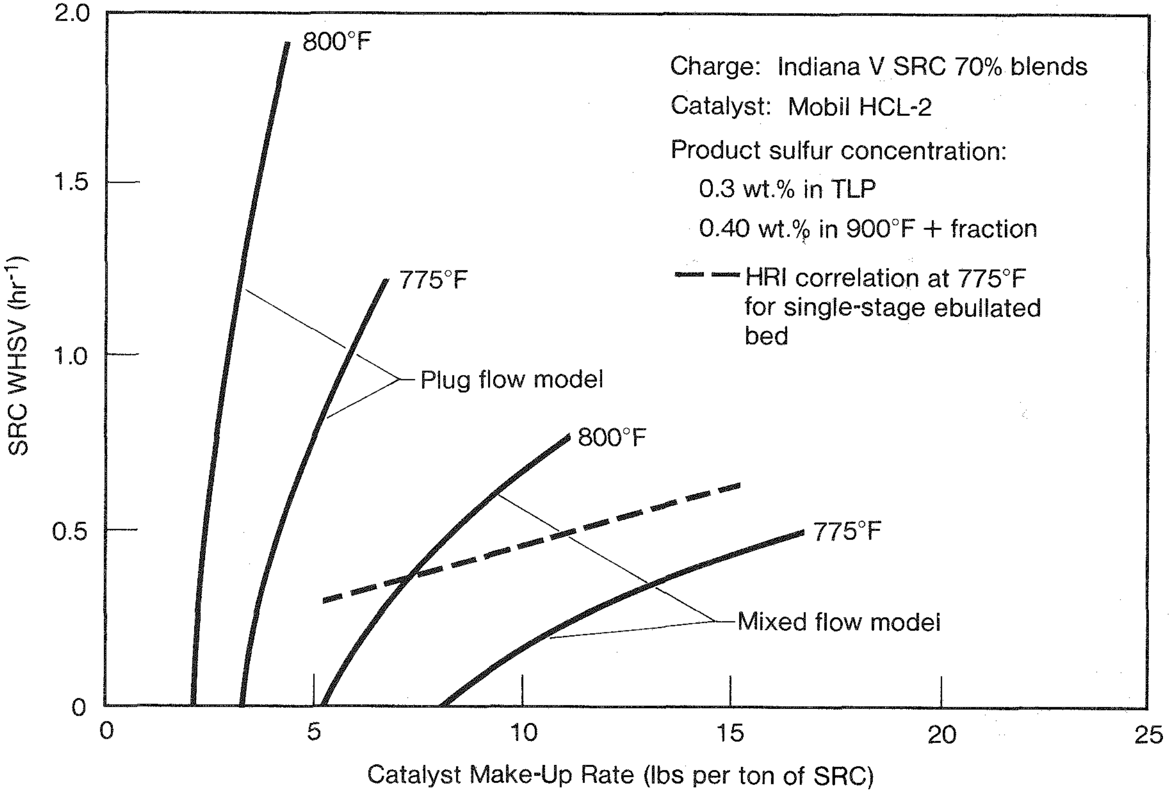


Figure 4-4

Design Variables for Ebullated Bed Reactor



flow model. This operation will require a high catalyst make-up rate. Alternately, the catalyst make-up rate can be reduced (close to the plug flow regime) if two or more reactors are operated in series. As the number of reactor increases, the overall kinetic performance approaches the plug flow limit. An economically optimal design will probably be two or three reactors in series. Also shown in Figure 4-4 is an HRI estimation of the WHSV vs. make-up rate relationship, based on proprietary H-Oil correlations (1).

4.3 AGING RUN OF 70% INDIANA V SRC OVER CYANAMID HDS-1443

The material balances in which a 70% Indiana V regular SRC blend over HDS-1443 are given in Table 4-3 and Table 4-4. The run was terminated after six days on-stream due to incipient plugging in the reactor. The operating conditions were as follows: 2000 psig, 775°F, and 0.5 LHSV. The sulfur content of the liquid product was 0.13 wt %, equivalent to a 0.40 wt % sulfur level or lower in the residual fuel (900°F$^+$). Due to the short cycle length, no kinetic analysis was performed.

4.4 AGING RUN OF 50% W. KENTUCKY SHORT CONTACT TIME SRC OVER AMOCAT 1A

The material balances in which a 50% W. Kentucky SRT SRC blend was hydroprocessed over Amocat 1A at constant conditions (2000 psig, 775°F, and 0.5 LHSV) are given in Table 4-5 and Table 4-6. The run was operated smoothly for 15 days and was then terminated due to incipient plugging in the reactor. The total liquid product sulfur content ranged from 0.07 wt % at the start of run to 0.12 wt % at the end of run.

Kinetic analyses were performed using the models and equations described in section 4.1. The estimated catalyst deactivation constant, pre-exponential factor, and activation energy in Equation 4-4 for heteroatom removal and CCR reduction are as follows:

	k_o $\left[\frac{1}{\text{wt \%-hr}}\right]$	E $\left[\frac{\text{Btu}}{\text{lb mole}}\right]$	τ [Days]	C_A Range [wt %]	mean deviation [wt %]
Sulfur	6.669×10^{10}	56,600	22.2	0.03-0.3	0.025
Nitrogen	3.369×10^{5}	34,600	36.1	0.7 -1.3	0.087
Oxygen	6.895×10^{9}	57,800	8.9	1-3	0.34
CCR	2.553×10^{7}	50,400	21.9	5-20	1.3

TABLE 4-3

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: CYANAMID HDS-1443
PROGRAM: 2880 UNIT: 146

	CHARGE	MB-973	MB-974	MB-975	MB-976	MB-977	MB-978
OPERATING CONDITIONS							
PRESSURE, PSIG	-	1980	1980	1980	1980	1980	1978
AVG REACTOR TEMP., °F	-	725	750	774	776	772	773
LHSV VFF/HR/VCAT	-	0.45	0.39	0.40	0.36	0.31	0.36
LIQUID PRODUCT PROPERTIES							
GRAVITY, API	-13.0	-4.4	-3.8	-4.0	-3.8	-3.3	-4.0
HYDROGEN, WT PCT	6.40	7.54	5.84	8.07	7.36	7.69	8.26
SULFUR, WT PCT	0.750	0.150	0.120	0.130	0.118	0.159	0.310
NITROGEN, WT PCT	1.76	1.39	1.42	1.19	1.24	1.27	1.22
OXYGEN, WT PCT	3.40	1.52	1.07	1.20	1.00	1.01	2.23
CCR, WT PCT	41.53	31.34	26.37	20.94	21.53	25.16	18.84
K.V.(100 C)	-	318.90	-	24.29	29.18	107.60	-
HETEROATOM REMOVAL							
DESULFURIZATION, PCT WT	-	80.5	84.6	83.2	84.9	79.6	59.0
DENITROGENATION, PCT WT	-	22.9	22.2	34.5	32.5	30.7	23.3
DEOXYGENATION, PCT WT	-	55.8	69.3	65.4	71.5	71.1	34.1
CCR REMOVAL, PCT WT	-	26.3	38.8	51.1	50.3	41.8	55.0
HYDROGEN CONSUMPTION, SCF/B							
TOTAL H2 CONSUMPTION	-	1196	1269	1692	1119	1410	1728
H2 CONSUMED BY C1-C5	-	152	215	222	236	273	107
H2 CONSUMED BY C6+	-	759	725	1118	516	779	1416
H2 CONSUMED BY S	-	30	31	31	32	30	22
H2 CONSUMED BY N	-	68	66	103	97	92	70
H2 CONSUMED BY O	-	186	231	218	238	237	114

TABLE 4-4

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
YIELDS AND DISTILLATION

CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: CYANAMID HDS-1443
PROGRAM: 2880 UNIT: 146

	MB-973	MB-974	MB-975	MB-976	MB-977	MB-978
MATERIAL BALANCE, PCT WT	99.5	99.9	96.9	102.3	97.9	96.8
OPERATING CONDITIONS						
PRESSURE, PSIG	1980	1980	1980	1980	1980	1978
AVG REACTOR TEMP., °F	725	750	774	776	772	773
LHSV VFF/HR/VCAT	0.45	0.39	0.40	0.36	0.31	0.36
H2 CHG, SCFB FF NLB	5895	8090	6569	7754	7617	7136
CATALYST AGE, DAYS	2.3	3.0	4.0	4.7	5.4	6.7
YIELDS (BASED ON CHARGE)						
TOT C1-C3, WT PCT	0.82	1.10	1.08	1.15	1.34	0.53
TOT C4, WT PCT	0.15	0.30	0.30	0.32	0.37	0.16
TOT C5, WT PCT	0.02	0.01	0.02	0.01	0.02	0.01
C6+ PRODUCT, WT PCT	97.29	96.43	96.88	95.87	96.09	99.24
H2S, WT PCT	0.64	0.67	0.66	0.68	0.63	0.47
NH3, WT PCT	0.49	0.47	0.74	0.69	0.66	0.50
H2O, WT PCT	2.11	2.62	2.47	2.70	2.69	1.29
DISTILLATION (D2887), °F						
IBP	-	-	271	149	311	-
5 PCT VOL	-	-	394	319	438	-
10 " "	-	-	448	380	455	-
30 " "	-	-	569	502	610	-
50 " "	-	-	878	629	-	-
70 " "	-	-	-	-	-	-
90 " "	-	-	-	-	-	-
95 " "	-	-	-	-	-	-
EP	-	-	-	-	-	-

TABLE 4-5

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOVENT
CATALYST: AMOCAT 1A
PROGRAM: 2916 UNIT: 146

	CHARGE	*MB-1004*	*MB-1005*	*MB-1006*	*MB-1007*	*MB-1008*	*MB-1009*	*MB-1010*	*MB-1011*
OPERATING CONDITIONS									
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	721	751	769	774	775	772	771	769
LHSV VFF/HR/VCAT	-	0.48	0.61	0.42	0.48	0.51	0.27	0.41	0.43
LIQUID PRODUCT PROPERTIES									
GRAVITY, API	-7.1	0.3	2.8	8.1	6.7	6.7	8.1	6.7	6.7
HYDROGEN, WT PCT	6.80	7.38	7.97	8.61	8.55	8.40	8.82	8.63	8.47
SULFUR, WT PCT	0.800	0.198	0.096	0.047	0.071	0.081	0.056	0.064	0.071
NITROGEN, WT PCT	1.41	0.99	0.91	0.78	0.76	0.63	0.64	0.88	0.79
OXYGEN, WT PCT	4.50	2.97	1.97	1.17	1.50	1.43	1.30	1.53	1.44
CCR, WT PCT	22.90	16.08	13.26	9.34	9.71	10.39	7.72	8.77	9.91
K.V.(100 C)	-	25.41	7.33	3.45	4.10	3.35	3.02	3.23	3.77
HETEROATOM REMOVAL									
DESULFURIZATION, PCT WT	-	72.5	86.8	93.6	90.3	88.2	93.5	92.4	91.6
DENITROGENATION, PCT WT	-	29.2	35.5	45.4	46.6	50.9	55.5	38.0	44.6
DEOXYGENATION, PCT WT	-	35.8	57.8	75.2	68.1	69.7	72.7	67.4	69.5
CCR REMOVAL, PCT WT	-	30.0	42.9	60.2	58.5	55.7	67.4	62.4	57.7
HYDROGEN CONSUMPTION, SCF/B									
TOTAL H2 CONSUMPTION	-	454	997	1610	1556	1507	1952	1679	1572
H2 CONSUMED BY C1-C5	-	69	128	248	243	222	398	243	252
H2 CONSUMED BY C6+	-	144	516	911	891	844	1087	1031	892
H2 CONSUMED BY S	-	24	29	31	30	34	36	35	35
H2 CONSUMED BY N	-	64	78	100	102	112	122	83	98
H2 CONSUMED BY O	-	152	246	320	290	296	308	286	295

TABLE 4-5 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOVENT
CATALYST: AMOCAT 1A
PROGRAM: 2916 UNIT: 146

	CHARGE	MB-1012	MB-1013	MB-1014	MB-1015	MB-1016	MB-1017	MB-1018
OPERATING CONDITIONS								
PRESSURE, PSIG	-	2000	1990	1983	1990	1986	1975	2065
AVG REACTOR TEMP., °F	-	770	776	775	774	776	774	773
LHSV VFF/HR/VCAT	-	0.44	0.47	0.46	0.40	0.47	0.43	0.39
LIQUID PRODUCT PROPERTIES								
GRAVITY, API	-7.1	6.7	0.3	4.1	3.3	3.3	3.0	3.8
HYDROGEN, WT PCT	6.80	8.51	8.53	8.18	8.70	8.49	8.62	8.66
SULFUR, WT PCT	0.800	0.072	0.079	0.099	0.093	0.119	0.105	0.084
NITROGEN, WT PCT	1.41	0.84	0.89	0.96	0.97	0.90	0.90	0.83
OXYGEN, WT PCT	4.50	1.35	1.73	2.05	1.67	2.43	3.68	2.98
CCR, WT PCT	22.90	10.11	10.75	11.61	12.82	14.12	11.47	10.78
K.V.(100 C)	-	3.65	4.45	5.72	6.04	10.39	6.79	5.17
HETEROATOM REMOVAL								
DESULFURIZATION, PCT WT	-	91.5	89.2	88.1	88.8	85.6	87.1	89.8
DENITROGENATION, PCT WT	-	41.2	39.5	34.5	34.0	38.4	37.3	42.8
DEOXYGENATION, PCT WT	-	71.4	63.8	56.1	64.4	47.9	19.7	35.6
CCR REMOVAL, PCT WT	-	57.0	55.4	51.7	46.8	41.1	51.3	54.7
HYDROGEN CONSUMPTION, SCF/B								
TOTAL H2 CONSUMPTION	-	1626	1720	1389	1864	1692	1747	1816
H2 CONSUMED BY C1-C5	-	289	267	213	291	300	274	295
H2 CONSUMED BY C6+	-	908	1051	820	1181	1062	1267	1233
H2 CONSUMED BY S	-	35	34	34	34	33	33	34
H2 CONSUMED BY N	-	90	92	80	79	89	87	99
H2 CONSUMED BY O	-	303	276	243	278	207	85	154

TABLE 4-6

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
YIELDS AND DISTILLATION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOVENT
CATALYST: AMOCAT 1A
PROGRAM: 2916 UNIT: 146

	MB-1004	MB-1005	MB-1006	MB-1007	MB-1008	MB-1009	MB-1010	MB-1011
MATERIAL BALANCE, PCT WT	90.6	99.2	99.7	97.7	101.0	141.3	99.9	100.6
OPERATING CONDITIONS								
PRESSURE, PSIG	2000	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	721	751	769	774	775	772	771	769
LHSV VFF/HR/VCAT	0.48	0.61	0.42	0.48	0.51	0.27	0.41	0.43
H2 CHG, SCFB FF NLB	6296	5868	8503	7786	7098	13055	9170	9078
CATALYST AGE, DAYS	1.2	1.9	2.8	3.5	4.5	5.8	6.8	7.8
YIELDS (BASED ON CHARGE)								
TOT C1-C3, WT PCT	0.40	0.70	1.35	1.37	1.23	2.11	1.30	1.33
TOT C4, WT PCT	0.04	0.13	0.25	0.17	0.19	0.49	0.28	0.31
TOT C5, WT PCT	-	0.01	0.01	0.01	0.01	0.02	0.01	0.01
C6+ PRODUCT, WT PCT	97.33	96.33	95.28	95.63	95.46	94.59	95.81	95.41
H2S, WT PCT	0.54	0.65	0.70	0.67	0.76	0.80	0.80	0.79
NH3, WT PCT	0.48	0.59	0.75	0.77	0.84	0.92	0.63	0.74
H20, WT PCT	1.81	2.93	3.81	3.45	3.53	3.68	3.42	3.52
DISTILLATION (D2887), °F								
IBP	-	210	323	317	263	280	315	312
5 PCT VOL	-	393	420	410	362	410	434	416
10 '' ''	-	441	459	445	417	452	475	455
30 '' ''	-	547	543	511	514	529	561	524
50 '' ''	-	642	651	576	589	587	660	592
70 '' ''	-	-	-	794	733	673	-	723
90 '' ''	-	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-

TABLE 4-6 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- AGING STUDY
YIELDS AND DISTILLATION
CHARGE: 50/50 *W. KENTUCKY SCT SRC / RECYCLE SOVENT*
CATALYST: AMOCAT 1A
PROGRAM: 2916 *UNIT:* 146

	MB-1012	*MB*-1013	*MB*-1014	*MB*-1015	*MB*-1016	*MB*-1017	*MB*-1018
MATERIAL BALANCE, PCT WT	100.3	93.5	99.0	94.0	95.3	94.6	91.4
OPERATING CONDITIONS							
PRESSURE, PSIG	2000	1990	1983	1990	1986	1975	2065
AVG REACTOR TEMP., °F	770	776	775	774	776	774	773
LHSV VFF/HR/VCAT	0.44	0.47	0.46	0.40	0.47	0.43	0.39
H_2 *CHG, SCFB FF NLB*	8174	7884	8195	9694	8005	8074	8429
CATALYST AGE, DAYS	8.8	9.8	10.8	11.8	12.8	13.8	14.8
YIELDS (BASED ON CHARGE)							
TOT C_1-C_3, *WT PCT*	1.56	1.41	1.13	1.56	1.61	1.46	1.58
TOT C_4, *WT PCT*	0.30	0.28	0.22	0.29	0.29	0.28	0.29
TOT C_5, *WT PCT*	0.01	0.01	0.01	0.01	0.01	0.01	0.01
C_6+ *PRODUCT, WT PCT*	95.21	95.88	96.28	95.98	96.50	98.16	97.20
H_2S, *WT PCT*	0.79	0.76	0.75	0.76	0.73	0.74	0.76
NH_3, *WT PCT*	0.68	0.68	0.59	0.58	0.66	0.64	0.73
H_2O, *WT PCT*	3.62	3.23	2.84	3.26	2.43	1.00	1.81
DISTILLATION (D2887), °*F*							
IBP	213	243	179	265	271	179	250
5 *PCT VOL*	344	364	428	393	409	389	383
10 " "	403	431	470	450	468	446	433
30 " "	510	534	572	550	566	562	522
50 " "	584	610	645	619	637	639	596
70 " "	702	-	968	797	-	-	738
90 " "	-	-	-	-	-	-	-
95 " "	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-

Figure 4-5

AGING RUN WITH W. KENTUCKY SCT SRC

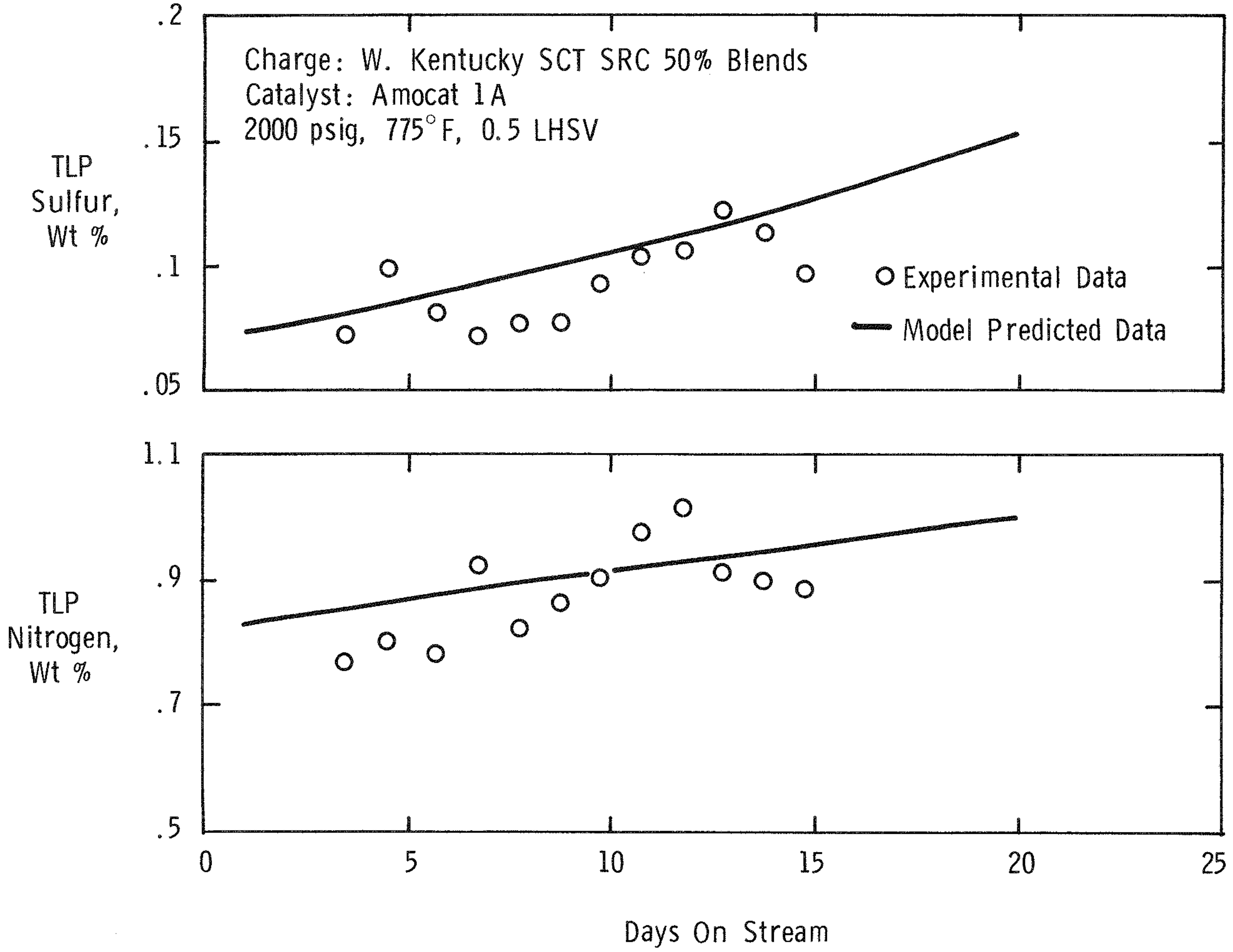
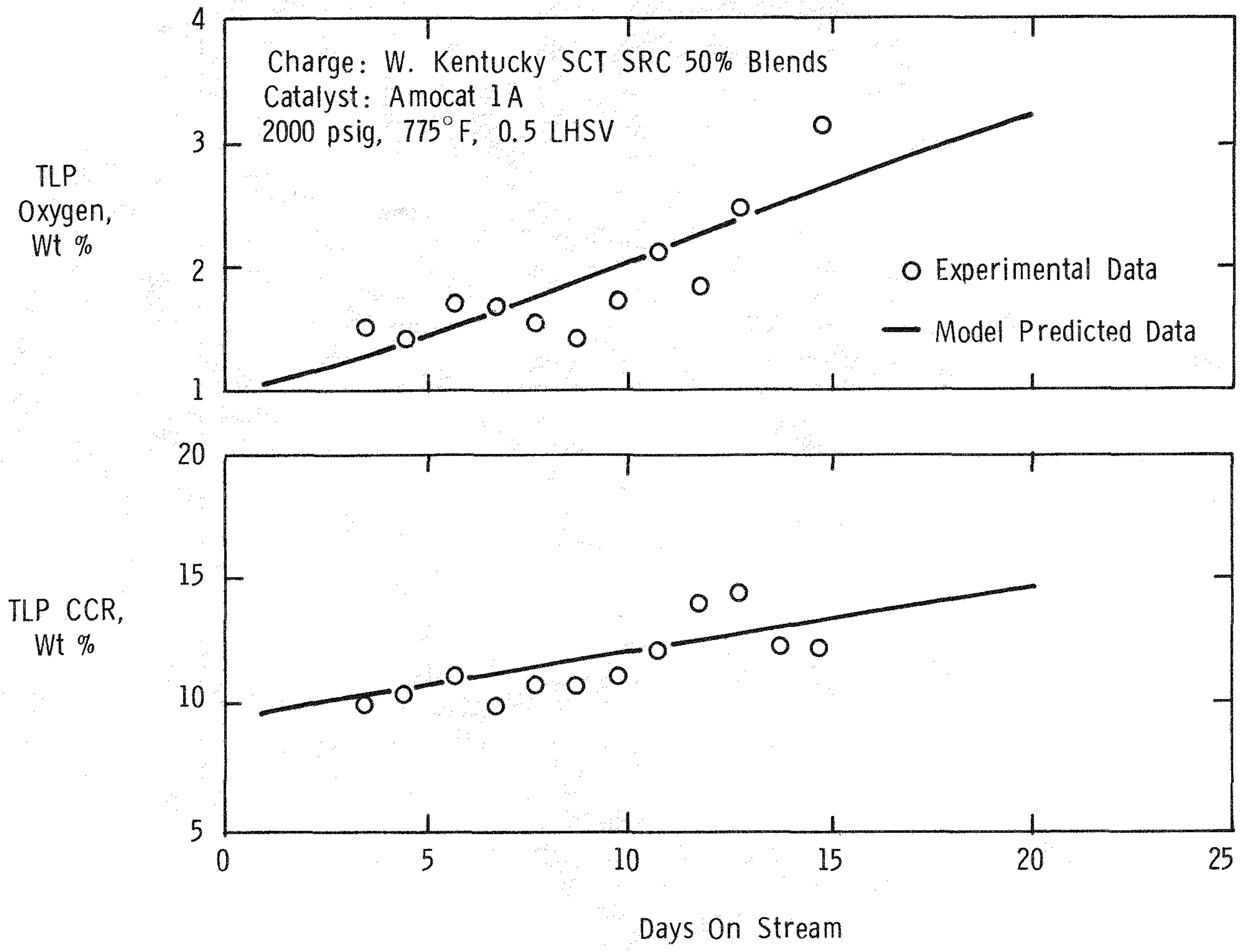


Figure 4-6

AGING RUN WITH W. KENTUCKY SCT SRC



The temperature rise necessary to maintain a constant product sulfur concentration is approximately 3°F/day. The measured and model-predicted values for sulfur and nitrogen are shown in Figure 4-5; comparable values for oxygen and CCR concentrations are shown in Figure 4-6.

The simulated operating conditions for the fixed bed reactor to produce a 900°F$^+$ fuel with a sulfur content of 0.4 wt % are shown in Figure 4-7, using a charge of 50% W. Kentucky SCT SRC. It is estimated that, for a 900°F$^+$ product with 0.4 wt % sulfur, the total liquid product sulfur content should be 0.15 wt % (cf. Appendix C). Figure 4-7 shows the reactor temperature required to achieve these sulfur levels as a function of days on-stream for LHSV of 0.1, 0.3, and 0.5. The temperature rise is 2.5-3.0°F/day. The cycle length, a function of both LHSV and the limit of reactor temperature, can be determined from Figure 4-7. For example, at 0.3 LHSV and a maximum reactor temperature of 850°F, the cycle length is 50 days.

In Figure 4-8, the SRC weight hourly space velocity (WHSV) required to meet a total liquid product sulfur level of 0.15 wt % (0.4 wt % in the 900°F$^+$ fraction) is plotted as a function of catalyst make-up rate. The required WHSV has been estimated for 775°F and 800°F. At a given WHSV, the make-up rate is approximately two times greater than in hydroprocessing 70% Indiana V regular SRC (cf. Figure 4-4). Although catalyst activity and charge stock reactivity may differ, the change in catalyst make-up rate is primarily attributable to SRC (and CCR) concentration differences, coupled with the second order kinetics. This high catalyst consumption in an ebullated bed, along with the 50-day cycle time projected in a fixed bed reactor, indicates that a 50% W. Kentucky SCT SRC is more suitably hydroprocessed in a fixed bed reactor. However, as discussed in section 4.2, the use of multiple reactors could significantly reduce the catalyst make-up rates. Also shown in Figure 4-8 is an estimated WHSV vs. make-up rate relationship at 775°F, provided by HRI with their proprietary H-Oil correlations (1). The relatively high catalyst make-up rates projected by both MRDC and HRI may be somewhat pessimistic. Ebullated bed operation has several advantages, not accounted for in this simple model, over fixed bed operation:

- improved gas/liquid/solid contacting via better interparticle mass transfer
- improved catalyst stability by continuous abrasion of metal contaminants
- improved catalyst stability by reducing propagation of interparticle coke formation
- decreased problems associated with "hot spots".

All of these advantages point to a decrease in catalyst make-up rates. Furthermore, more reactive SRC's such as Kerr-McGee Critical Solvent Deashed SRC may further lessen catalyst requirements. It is our recommendation that ebullated bed confirmation runs be made to test these projections.

Figure 4-7

DESIGN VARIABLES FOR FIXED BED REACTOR

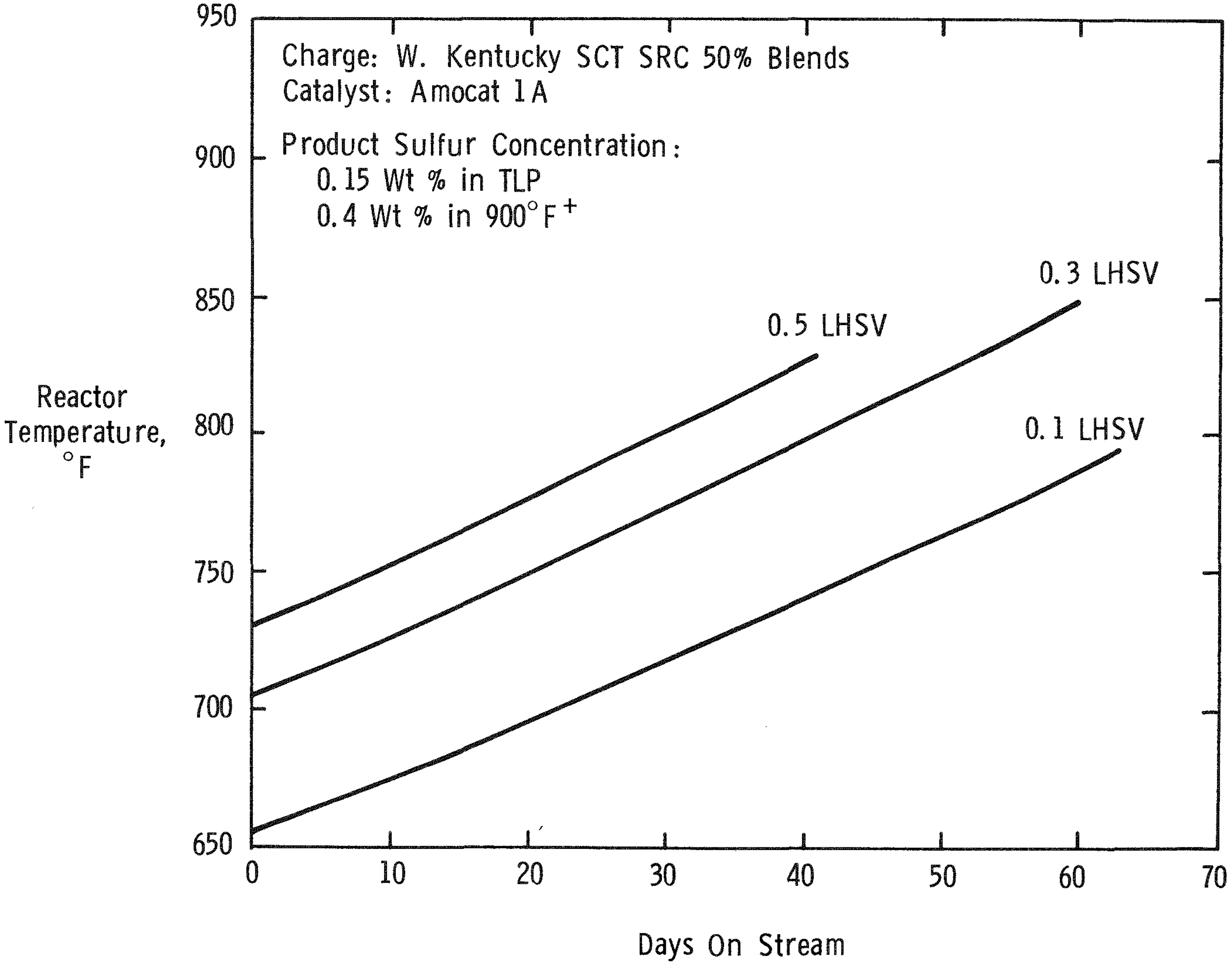
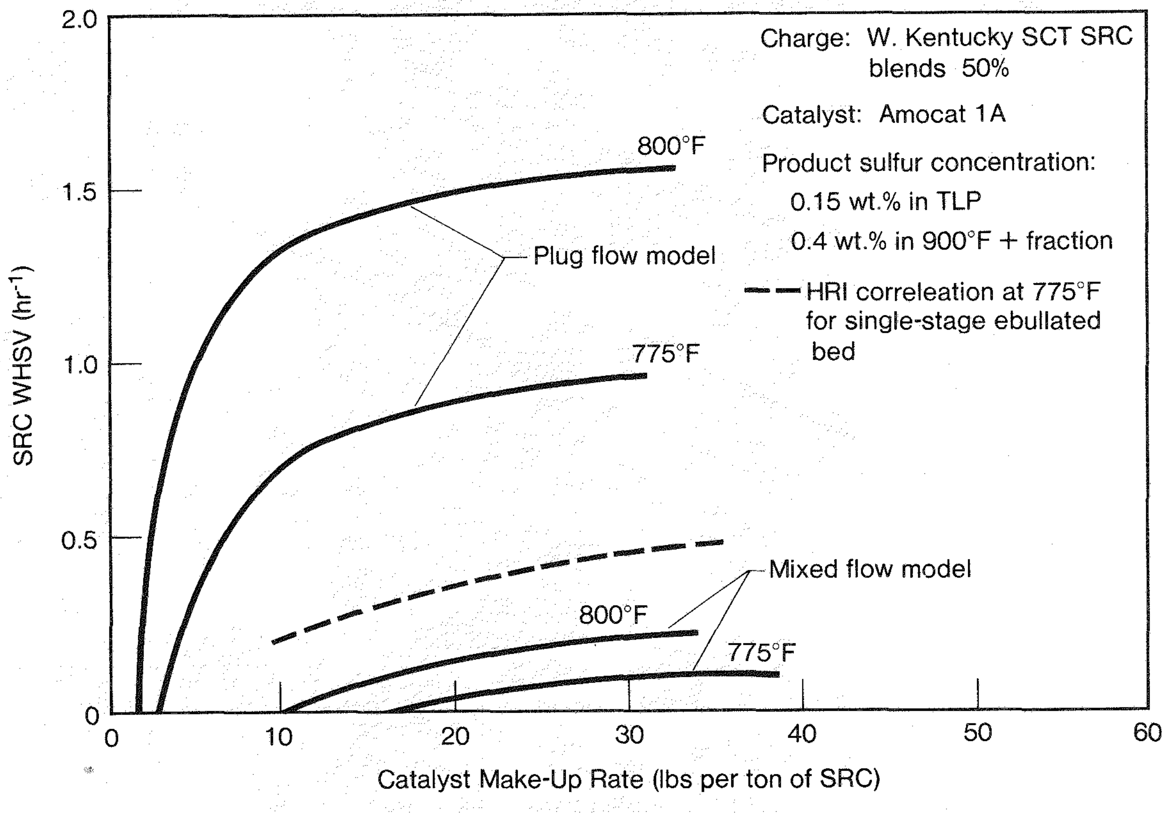


Figure 4-8

Design Variables for Ebullated Bed Reactor



Section 5

COAL LIQUID CHARACTERIZATION

One of the major areas of this study has been the characterization of raw and hydrotreated coal liquids. The types of characterization employed vary from physical and chemical measurement of collective properties, such as elemental composition and boiling point ranges, to the more detailed analyses of chemical classes, molecular size distributions, and molecular entities. Analytical methods used include gradient elution chromatography (GEC), gel permeation chromatography (GPC), and high resolution mass spectrometry. A more fundamental understanding of the chemical nature of coal-derived liquids, as well as of the alterations effected by catalytic hydroprocessing, will ultimately enable the development of optimal processing methods.

The chemistry of trace alkali metals in coal-derived distillates was also studied, and various separation methods were investigated.

5.1 GEC ANALYSES

Hydrotreated Monterey SRC

Material balances in which a 90% Monterey SRC blend was hydroprocessed over Harshaw 618X were reported in the 1978 Annual Report. The charge and several liquid products from this study have since been distilled to a 650°F cut point, and the overhead and residual fractions were analyzed for elemental composition. The 650°F$^+$ fractions have also been characterized by gradient elution chromatography, and the elemental compositions determined for each of the GEC fractions. These data appear in Tables 5-1 and 5-2. As has been seen with blends containing less SRC (i.e., 33% and 70%), increasing hydroprocessing severity increases the hydrogen content of the total liquid product and the 650°F$^-$ and 650°F$^+$ product fractions while decreasing the heteroatom contents of each. An increase in the amount of distillate material also results. The 650°F$^-$ yields are necessarily lower with this 90% SRC blend since the blend contains a lower initial concentration of the 650°F$^-$ solvent. Analyses of the 650°F$^+$ fractions tend to show only the effect of the SRC upgrading since most of the solvent has been removed with

TABLE 5-1

HYDROPROCESSING OF 90°/° MONTEREY SRC / 10°/° RECYCLE SOLVENT
GEC AND ELEMENTAL ANALYSES
CATALYST: HARSHAW 618X
UNIT: 146 PROGRAM: 2628

	MONTEREY SRC	CHARGE	MB-871	MB-872	MB-877	MB-878
OPERATING CONDITIONS						
TEMPERATURE, °F	-	-	720	778	775	798
LHSV, VFF/HR/VCAT	-	-	0.24	0.28	0.20	0.15
PRESSURE, PSIG	-	-	2000	2000	2000	2000
H2 CONSUMPTIONS, SCF/B	-	-	1665	2332	2881	3125
TLP PROPERTIES						
HYDROGEN, WT PCT	6.22	6.11	7.78	8.60	8.72	8.86
OXYGEN, WT PCT	4.0	4.3	1.9	0.5	0.7	0.3
NITROGEN, WT PCT	1.75	1.86	0.98	0.58	0.65	0.41
SULFUR, WT PCT	0.70	1.00	0.11	<0.05	<0.05	0.02
TLP YIELDS, WT PCT						
650°F-	-	-	16.76	30.21	25.83	32.58
SATURATES (CUT 1)	0.13	0.15	1.15	1.34	2.31	1.23
AROM. OILS (CUT 2-4)	11.88	12.47	32.10	39.77	42.71	42.22
RESINS/ASPH. (CUT 5-7)	35.92	33.51	33.78	21.79	22.00	18.42
POLAR ASPH. (CUT 8-13)	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
ELEMENTAL ANALYSES						
IBP-650°F						
HYDROGEN, WT PCT	-	-	9.84	10.28	10.44	10.43
OXYGEN, WT PCT	-	-	0.93	0.27	0.45	0.50
NITROGEN, WT PCT	-	-	0.32	0.12	0.20	0.16
SULFUR, WT PCT	-	-	0.010	<0.002	<0.002	<0.002
650°F+ BTMS						
HYDROGEN, WT PCT	-	-	7.54	7.96	7.30	7.78
OXYGEN, WT PCT	-	-	2.1	1.1	0.6	0.8
NITROGEN, WT PCT	-	-	1.36	0.86	0.86	0.77
SULFUR, WT PCT	-	-	0.13	-	<0.03	<0.03

Table 5-2

ELEMENTAL ANALYSIS OF GEC FRACTIONS FOR HYDROPROCESSING OF 90% MONTEREY SRC BLEND (PROGRAM 2628)

CATALYST - HARSHAW 618X

	J8991 Charge	MB-871	MB-862	MB-877	MB-878
Temperature, °F	-	720	778	775	798
LHSV	-	.24	.28	.20	.15
Pressure, psig	-	2000	2000	2000	2000
H_2 Consumption, scf/B	-	1665	3337	2881	3125
Hydrogen, wt %					
650°F Residue		7.54	7.96	7.30	7.78
Cut 1	(a)	12.26	12.76	12.05	12.81
Cut 2-4	7.92	7.99	8.01	7.95	8.30
Cut 5-7	6.40	7.28	6.79	6.77	6.35
Cut 8-12	5.82	6.18	5.54	5.32	5.16
Oxygen, wt %					
650°F Residue		2.1	1.1	.6	.8
Cut 1	0	0	0	0	0
Cut 2-4	1.0	1.0	.3	<.1	.2
Cut 5-7	3.4	2.2	1.5	1.6	1.4
Cut 8-12	6.3	4.5	3.4	2.9	3.1
Nitrogen, wt %					
650°F Residue		1.36	.86	.86	.77
Cut 1	0	0	0	0	0
Cut 2-4	.58	.57	.25	.27	.17
Cut 5-7	2.59	2.12	1.95	1.92	1.93
Cut 8-12	2.01	1.60	1.41	1.35	1.32
Sulfur, wt %					
650°F Residue		.13	(a)	<.03	<.03
Cut 1	0	0	0	0	0
Cut 2-4	1.11	.20	<.03	<.03	<.03
Cut 5-7	.64	.14	.40	0.0	.20
Cut 8-12	.90	.25	(a)	(a)	(a)

(a) Analysis not determined.

the 650°F$^-$ fraction. For a given SRC and catalyst used, elemental analyses of the GEC and bottoms fractions are very similar regardless of the initial recycle solvent concentration.

At the high severity level used during this study, increasing severity decreases the amounts of both the resin and asphaltene fractions, while increasing the yields of the lighter fractions. The polar asphaltene fraction was reduced by as much as 90% at the most severe condition listed. The elemental analyses of the GEC fractions show hydrogen to be added predominantly to the non-polar asphaltene fractions, with the polar asphaltenes actually tending to become more condensed (i.e., hydrogen deficient). The resins show an initial increase in hydrogen content after which increasing processing severity decreases the amount of hydrogen. Oxygen, nitrogen, and sulfur contents of each of the GEC fractions tend to decrease with severity. Oxygen and sulfur contents are seen to increase in the higher molecular weight cuts, with the nitrogen content peaking in the resin fraction as has been noted previously.

Hydrotreated Monterey and Wyodak SRC -- Effects of Catalyst Pore Size

We have, over the last year (1), reported the results for hydroprocessing blends of Wyodak and Monterey SRC over HDS-1441A (CoMo/Al_2O_3) and Harshaw 618X (NiMo/Al_2O_3). We have now analyzed these results collectively to determine how the hydroprocessing selectivity is affected by changes in the catalyst and/or coal source.

The 650°F$^+$ fraction of these liquids is essentially the SRC fraction of the blend. Figures 5-1 and 5-2 show that the oxygen and nitrogen content of this fraction can be related directly to the hydrogen content independent of the SRC source or catalyst used. A similar plot for sulfur shows the consistently lower sulfur content of the Wyodak (Figure 5-3). These plots also show that sulfur decreases most rapidly with increasing hydrogen content, followed by oxygen and then nitrogen.

Figure 5-4 shows that the small pore catalyst (HDS-1441A) puts more hydrogen into the 650°F$^-$ fraction at a given 650°F$^+$ hydrogen content than does the large pore catalyst (Harshaw 618X). There is considerable scatter in this plot which suggests that temperature may be a secondary variable in this effect. Figure 5-5 shows that the small pore catalyst is deoxygenating the 650°F$^-$ (solvent) fraction at a faster rate than the large pore catalyst. The hydrogenation and deoxygenation of

Figure 5-1

OXYGEN VS. HYDROGEN CONTENT IN 650°F FRACTION OF RAW AND HYDROTREATED SRC

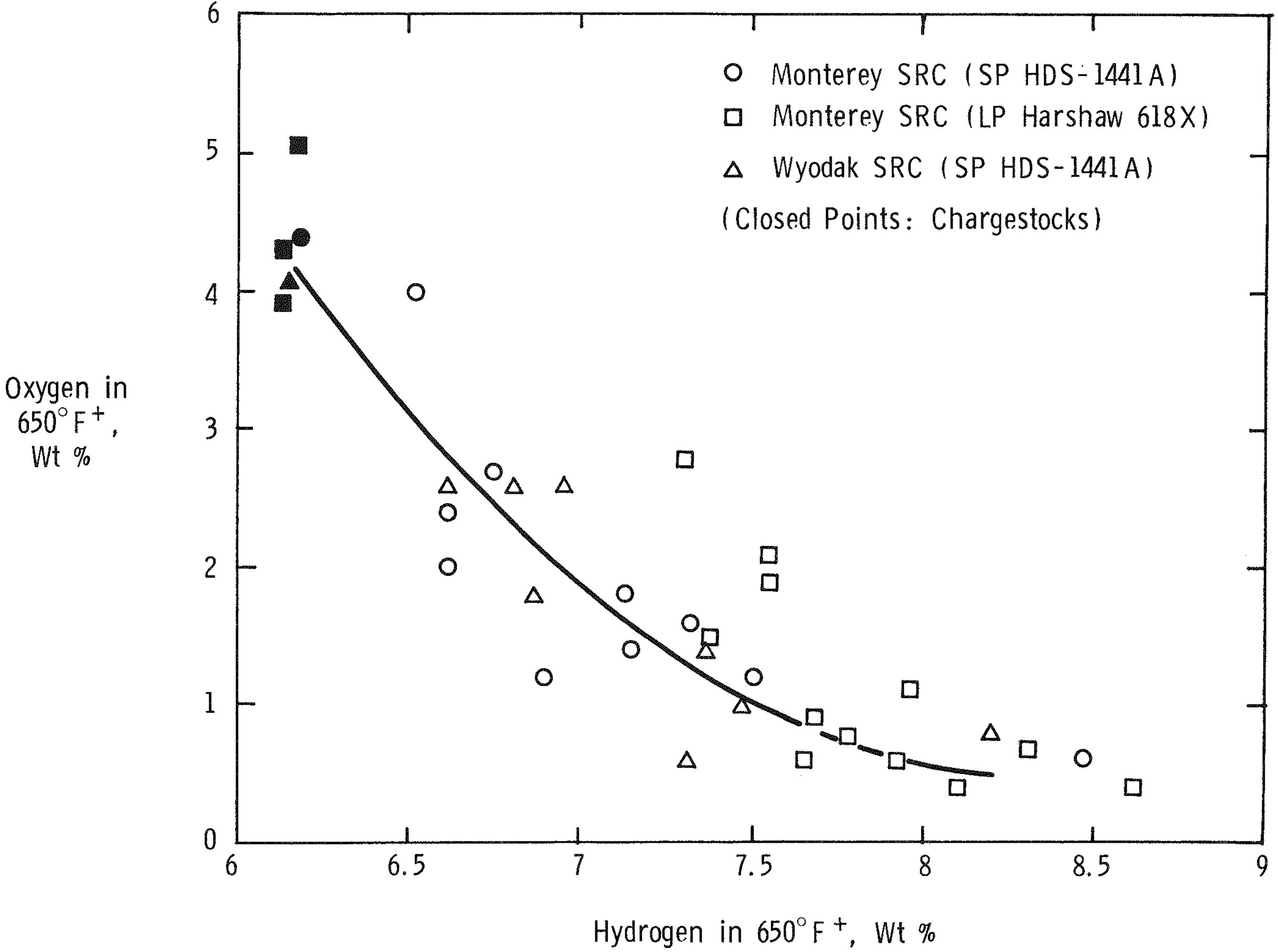


Figure 5-2

NITROGEN VS. HYDROGEN CONTENT IN 650°F+ FRACTION OF RAW AND HYDROTREATED SRC

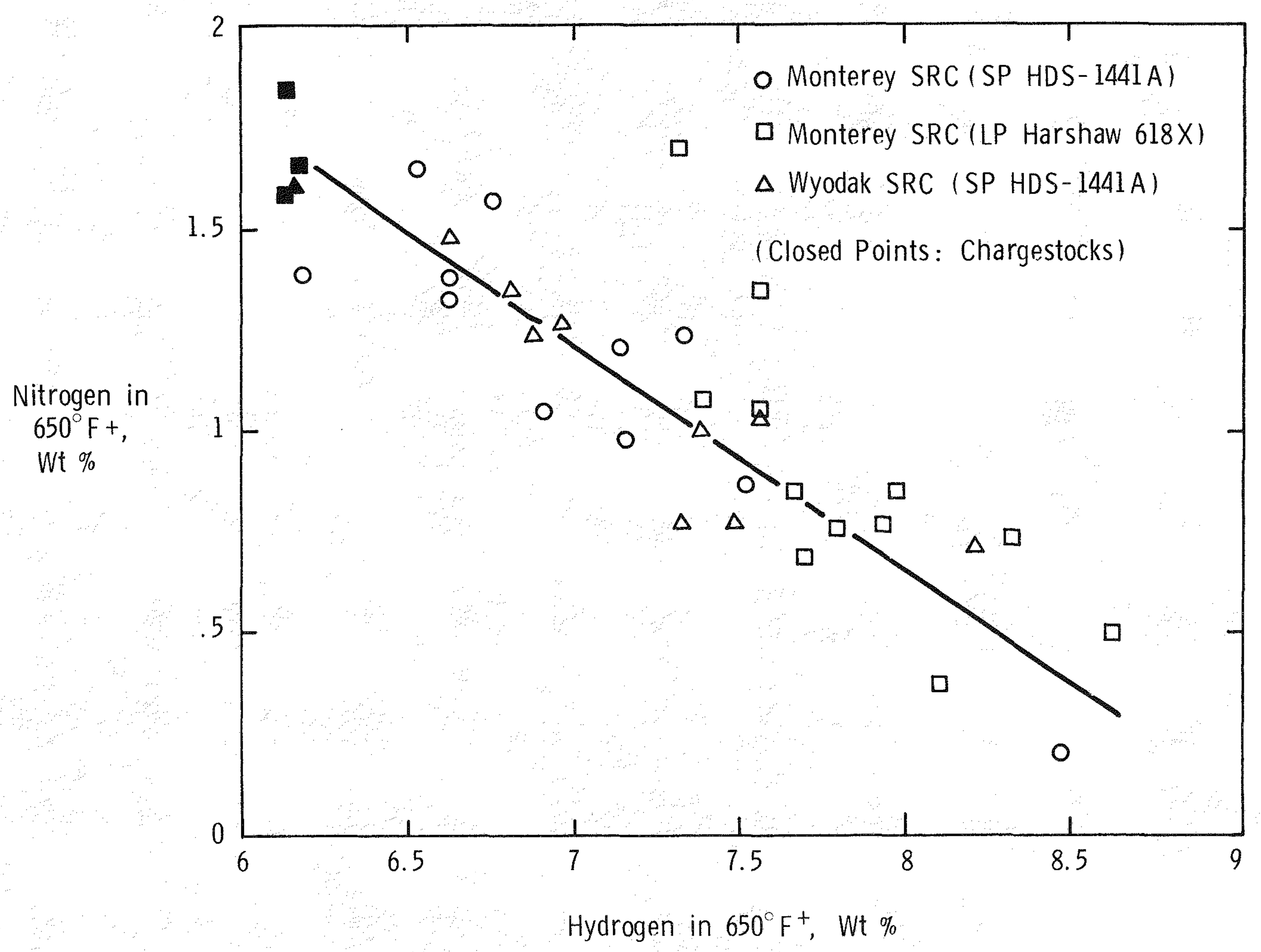


Figure 5-3

SULFUR VS. HYDROGEN CONTENT IN 650°F+ FRACTION OF RAW AND HYDROTREATED SRC

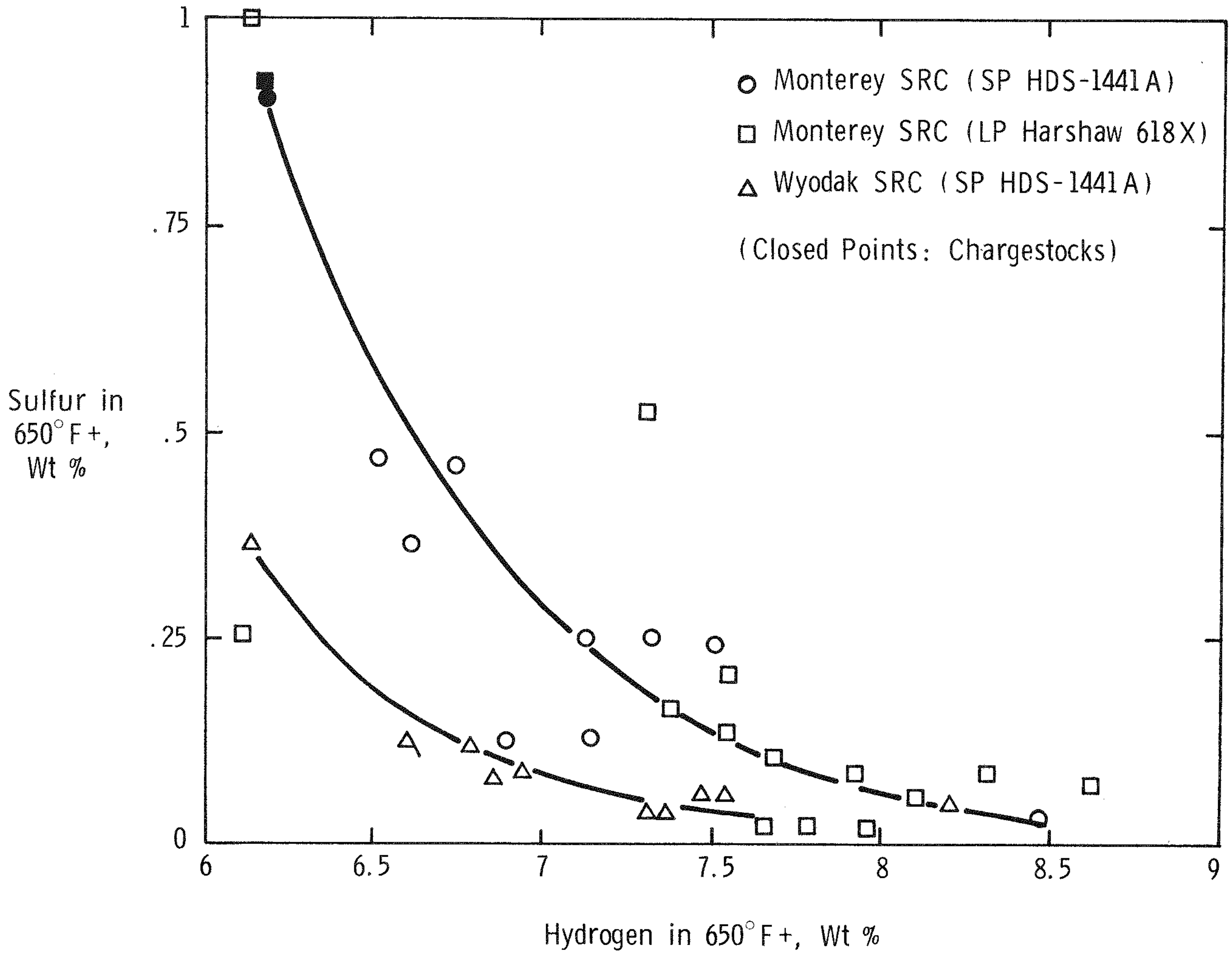


Figure 5-4

650°F⁻ VS. 650°F+ SELECTIVITY FOR HYDROGEN

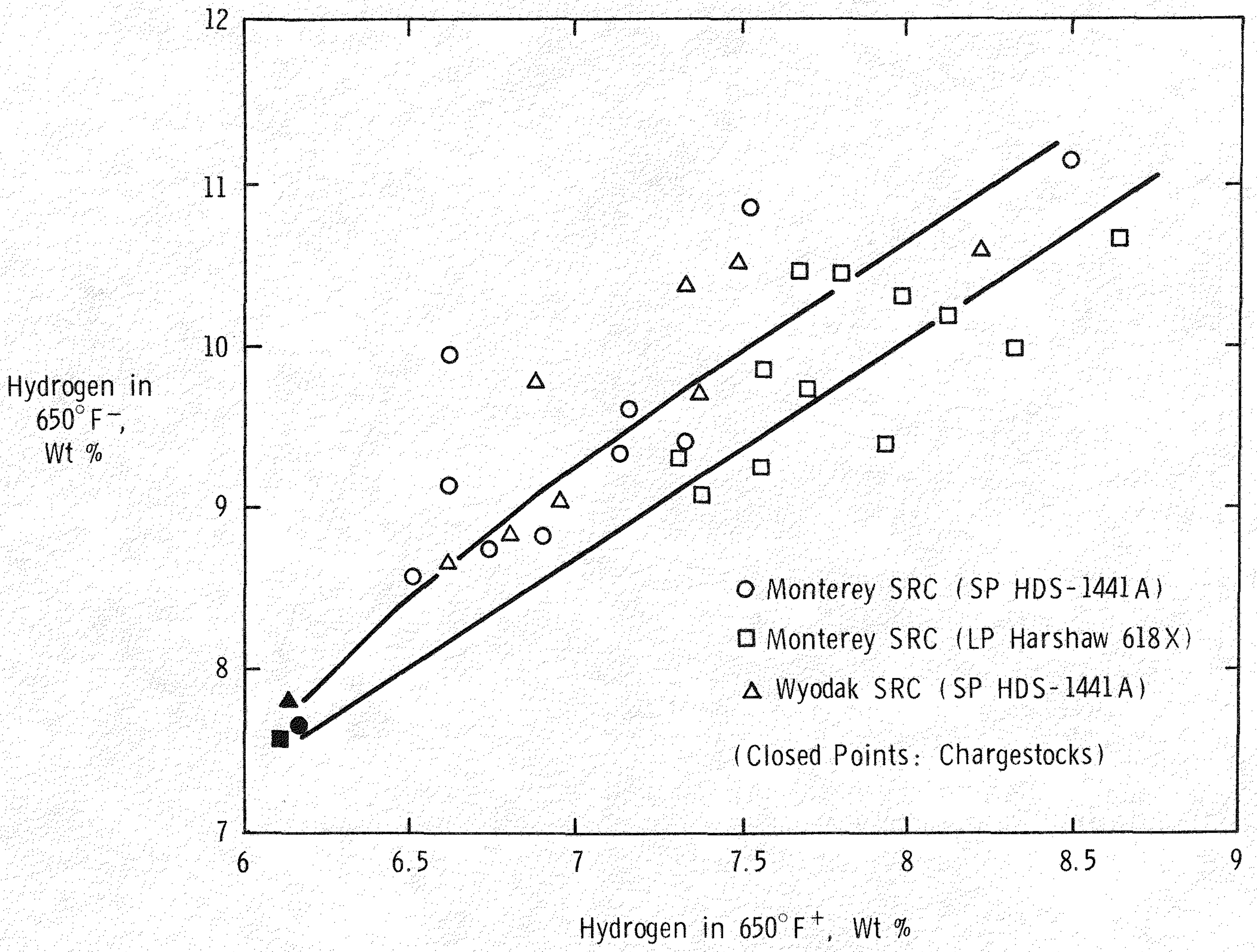
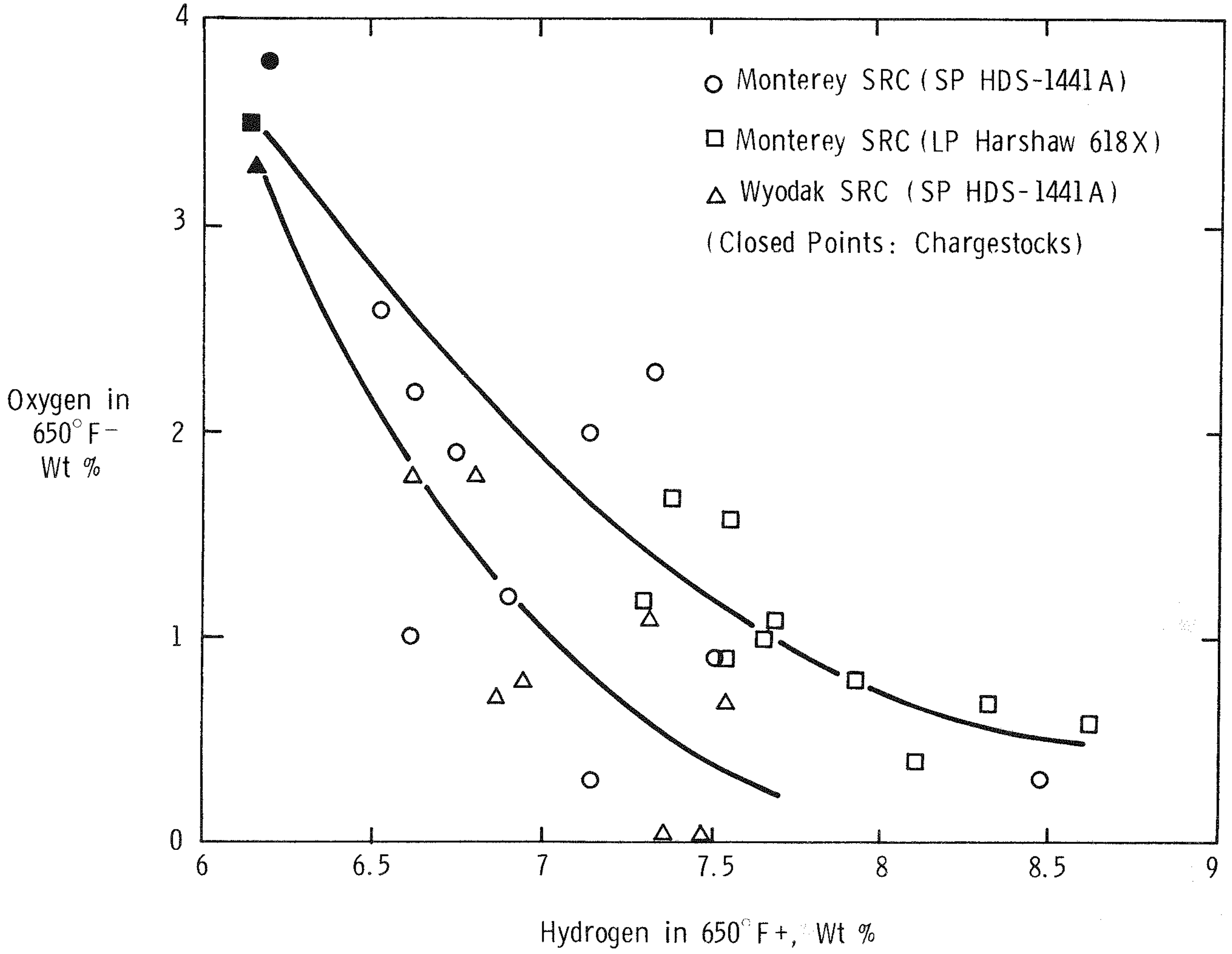


Figure 5-5

650°F⁻ OXYGEN VS. 650°F+ HYDROGEN SELECTIVITY



the $650°F^-$ fraction greatly decrease its solvent power and are probably responsible for the two phase products sometimes observed with this catalyst.

Elemental analyses of the GEC fractions show that the oxygen contents of the resins and asphaltenes are reduced simultaneously (Figure 5-6). However, the oxygen does not go below 1.5 and 3.0 wt % for the resins and asphaltenes, respectively. Apparently, further oxygen removal drops the molecules to a lower fraction.

Hydrotreated Indiana V SRC

The GEC analyses are given in Table 5-3 for raw and hydrotreated $650°F^+$ products obtained from 70% Indiana V SRC blends when hydroprocessed over Harshaw 618X. The polar asphaltenes (GEC cuts 8-13) are converted to resins and oils, which are subsequently converted to saturates and $650°F^-$ materials with increasing process severity. Elemental analyses and yields of both $650°F^+$ and $650°F^-$ fractions are also included in Table 5-3. The hydrogen consumptions for hydrotreating 70% Indiana V SRC are relatively low, requiring 700-800 scf/B to reach a sulfur level of 0.4 wt % in the $650°F^+$ fraction.

Hydrotreated W. Kentucky SCT SRC

During the past year 50% W. Kentucky SCT SRC blends were used as feedstocks for the catalyst evaluation study, reported in section 3. Portions of the liquid products from the catalyst evaluation study were also distilled to a 650°F cut point. Both the overhead product and the bottom residue were analyzed for elemental compositions. GEC analyses were also made on the $650°F^+$ fractions (Tables 5-4 and 5-5). At increasing severity of treatment, more hydrogen is added to the product and fewer heteroatoms remain. Another result is an increase in the amount of distillate material.

Although a variety of different catalysts has been investigated, the reaction mechanism of molecular structure conversions (based on GEC results) seems to be similar for all the catalysts. Functionally, undiluted W. Kentucky SCT SRC can be considered as polar asphaltenes (80 wt % are GEC cuts 8-13). As shown in Figure 5-7, the polar asphaltenes are plotted as a function of total liquid product (TLP) hydrogen content; the polar asphaltenes decrease quickly with increasing processing severity (i.e., TLP hydrogen content). Amocat 1B and Cyanamid HDS-1443 are found to be significantly more selective for polar asphaltene conversion than

Figure 5-6

DISTRIBUTION OF OXYGEN IN RESINS AND ASPHALTENES FOR MONTEREY SRC

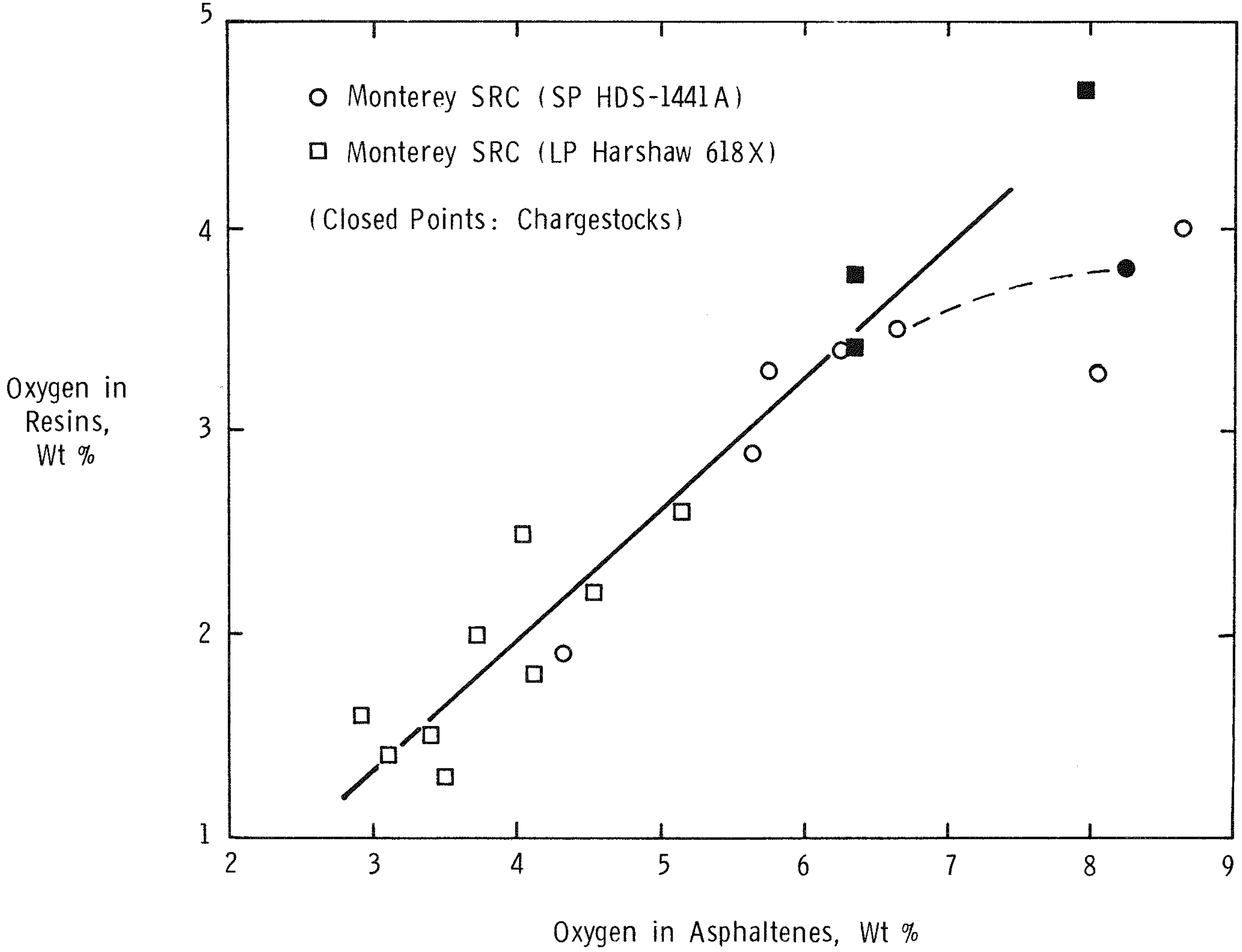


TABLE 5-3

GEC AND ELEMENTAL ANALYSES
CHARGE: 70/30 *INDIANA V SRC / RECYCLE SOLVENT BLEND*
CATALYST: HARSHAW 618X
PROGRAM: 2782 *UNIT:* 146

	CHARGE	*MB*-922	*MB*-925	*MB*-927	*MB*-928
OPERATING CONDITIONS					
TEMPERATURE, °F	-	723	790	777	728
LHSV, VFF/HR/VCAT	-	0.81	1.81	0.58	0.48
PRESSURE, PSIG	-	2000	2000	2000	2000
*H*2 *CONSUMPTIONS, SCF/B*	-	709	588	1349	1331
TLP PROPERTIES					
HYDROGEN, WT PCT	6.43	7.20	6.93	7.64	7.18
OXYGEN, WT PCT	3.80	2.97	3.08	1.75	1.37
NITROGEN, WT PCT	1.72	1.68	1.28	1.38	1.16
SULFUR, WT PCT	0.62	0.30	0.24	0.19	0.09
TLP YIELDS, WT PCT					
650°*F*-	23.0	22.5	29.9	29.5	28.6
SATURATES (CUT 1)	0.6	0.7	0.8	0.9	1.6
AROM. OILS (CUT 2-4)	5.2	10.2	12.3	15.6	22.9
RESINS/ASPH. (CUT 5-7)	18.5	31.0	28.3	29.9	28.3
POLAR ASPH. (CUT 8-13)	52.7	35.6	28.7	24.1	18.6
TOTAL	100.0	100.0	100.0	100.0	100.0
ELEMENTAL ANALYSES					
IBP-650°*F*					
HYDROGEN, WT PCT	7.93	8.41	8.56	9.17	9.27
OXYGEN, WT PCT	2.88	1.97	1.98	1.30	1.13
NITROGEN, WT PCT	0.59	0.52	0.58	0.53	0.48
SULFUR, WT PCT	0.40	0.07	0.06	0.03	0.01
650°*F+ BTMS*					
HYDROGEN, WT PCT	5.23	5.91	5.98	6.20	6.34
OXYGEN, WT PCT	2.95	2.49	2.02	1.47	1.33
NITROGEN, WT PCT	2.34	1.25	1.22	1.37	1.84
SULFUR, WT PCT	0.90	0.40	0.36	0.20	0.13

TABLE 5-4

GEC AND ELEMENTAL ANALYSES
CHARGE: 50/50 *W. KENTUCKY SCT SRC / RECYCLE SOLVENT BLEND*
CATALYST: HARSHAW 618*X*
PROGRAM: 2723 *UNIT:* 146

	CHARGE	*MB*-903	*MB*-905	*MB*-907	*MB*-908	*MB*-909	*MB*-910
OPERATING CONDITIONS							
TEMPERATURE, °F	-	725	768	802	803	799	732
LHSV, VFF/HR/VCAT	-	0.68	1.86	0.65	1.43	0.43	0.58
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
H2 CONSUMPTIONS, SCF/B	-	1504	1098	2500	1740	2538	1938
TLP PROPERTIES							
HYDROGEN, WT PCT	6.41	8.04	7.45	8.47	8.13	8.78	8.46
OXYGEN, WT PCT	4.30	2.40	2.40	1.00	1.70	0.70	1.90
NITROGEN, WT PCT	1.42	1.04	1.14	0.74	0.92	0.64	0.75
SULFUR, WT PCT	0.66	0.19	0.25	0.08	0.13	0.06	0.13
TLP YIELDS, WT PCT							
650°*F*-	36.5	45.7	40.6	48.9	49.8	60.2	45.5
SATURATES (CUT 1)	0.9	1.3	1.4	-	1.7	1.8	-
AROM. OILS (CUT 2-4)	8.3	10.9	11.5	-	15.6	16.6	-
RESINS/ASPH. (CUT 5-7)	10.3	16.3	16.1	-	14.8	11.5	-
POLAR ASPH. (CUT 8-13)	44.0	25.8	30.1	-	18.1	9.9	-
TOTAL	100.0	100.0	100.0	-	100.0	100.0	-
ELEMENTAL ANALYSES							
IBP-650°F							
HYDROGEN, WT PCT	7.95	9.15	8.85	9.35	9.00	9.63	8.60
OXYGEN, WT PCT	2.75	1.59	1.66	0.72	1.34	0.86	1.36
NITROGEN, WT PCT	0.51	0.43	0.56	0.32	0.52	0.31	0.40
SULFUR, WT PCT	0.40	0.01	0.03	0.01	0.01	0.01	0.01
650°*F+ BTMS*							
HYDROGEN, WT PCT	5.95	7.05	6.86	7.37	7.10	7.20	7.36
OXYGEN, WT PCT	5.41	4.66	3.57	1.89	2.49	1.70	2.98
NITROGEN, WT PCT	1.93	1.36	1.51	1.10	1.32	1.22	1.26
SULFUR, WT PCT	1.00	0.40	0.46	0.12	0.35	0.22	0.37

TABLE 5-5

GEC AND ELEMENTAL ANALYSES
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT

CATALYST		AMOCAT 1B				MOBIL HCL-2			
	CHARGE	MB-930	MB-932	MB-935	MB-936	MB-939	MB-942	MB-943	MB-946
OPERATING CONDITIONS									
TEMPERATURE, °F	-	715	775	801	778	725	765	775	797
LHSV, VFF/HR/VCAT	-	0.83	1.58	0.34	0.45	0.46	0.46	1.82	0.44
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	1975
H2 CONSUMPTIONS, SCF/B	-	415	941	3010	1984	1857	3872	1181	2182
TLP PROPERTIES									
HYDROGEN, WT PCT	6.71	7.02	7.53	8.83	8.34	7.98	9.30	7.94	8.94
OXYGEN, WT PCT	4.42	3.50	2.68	0.59	1.81	1.69	1.17	2.37	0.71
NITROGEN, WT PCT	1.30	1.11	1.08	0.70	0.90	0.92	0.69	1.00	0.59
SULFUR, WT PCT	0.870	0.470	0.380	0.170	0.210	0.112	0.034	0.189	0.011
TLP YIELDS, WT PCT									
650°F-	29.1	45.9	51.7	62.3	56.4	54.3	55.4	49.0	65.1
SATURATES (CUT 1)	1.1	1.3	-	-	1.0	-	1.9	1.2	-
AROM. OILS (CUT 2-4)	13.6	9.4	-	-	14.4	-	18.9	10.5	-
RESINS/ASPH. (CUT 5-7)	12.9	15.6	-	-	17.9	-	16.8	16.3	-
POLAR ASPH. (CUT 8-13)	43.3	27.8	-	-	10.3	-	7.5	23.0	-
TOTAL	100.0	100.0	-	-	100.0	-	100.0	100.0	-
ELEMENTAL ANALYSES									
IBP-650°F									
HYDROGEN, WT PCT	8.14	8.50	8.54	9.42	9.00	9.98	9.90	8.86	9.92
OXYGEN, WT PCT	2.92	2.09	2.08	0.77	1.14	1.04	0.80	1.61	0.54
NITROGEN, WT PCT	0.523	0.597	0.609	0.398	0.470	0.406	0.357	0.607	0.305
SULFUR, WT PCT	0.370	0.260	0.215	0.059	0.113	0.002	0.002	0.037	0.002
650°F+ BTMS									
HYDROGEN, WT PCT	5.78	6.66	6.65	7.24	6.80	7.45	7.76	6.79	7.47
OXYGEN, WT PCT	5.16	3.69	2.98	0.76	1.55	2.29	0.94	2.88	0.68
NITROGEN, WT PCT	1.81	1.78	1.92	0.96	1.60	1.62	1.38	1.95	1.23
SULFUR, WT PCT	0.98	0.68	0.61	0.06	0.25	0.15	0.10	0.35	0.06

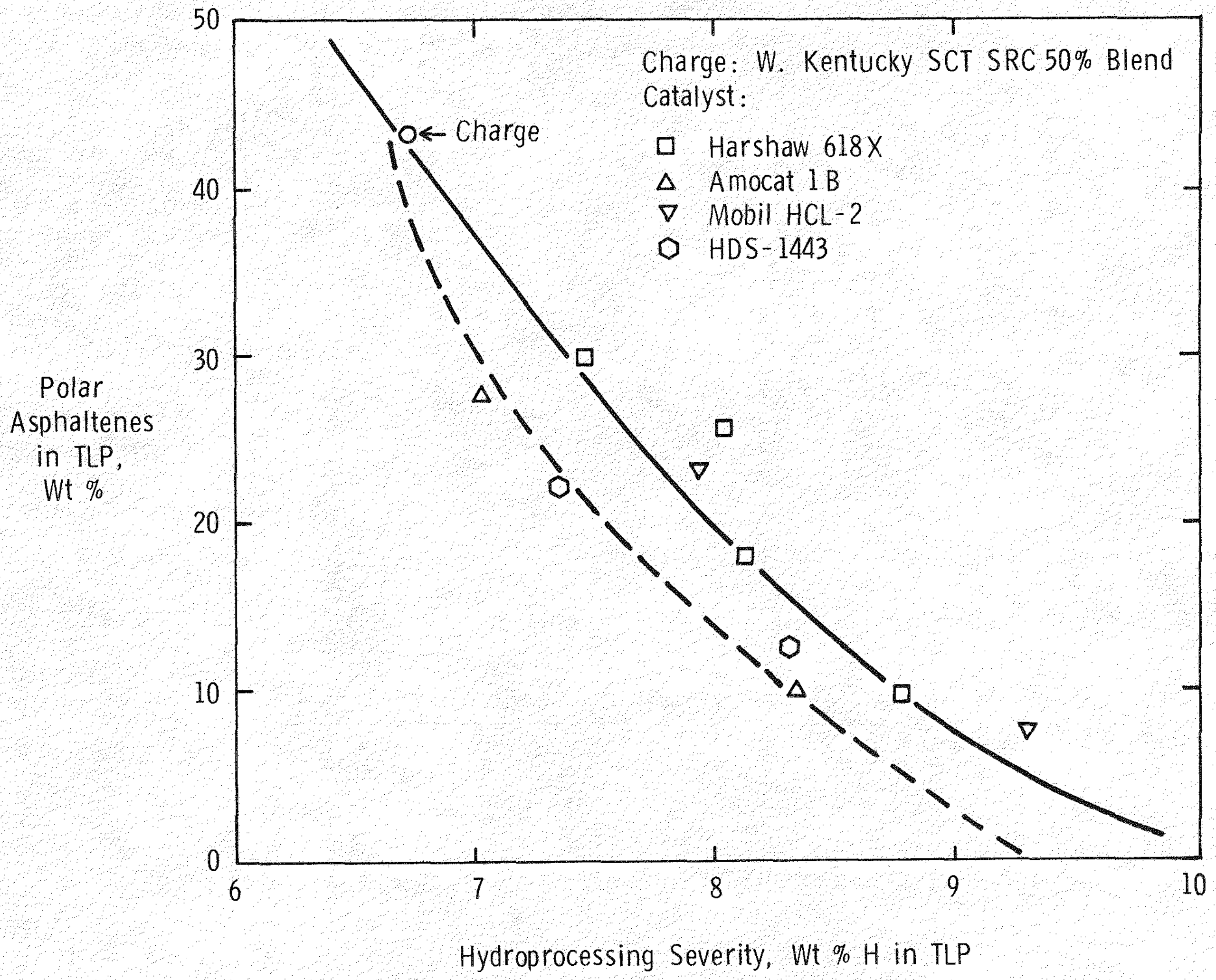
TABLE 5-5 (CONTINUED)

GEC AND ELEMENTAL ANALYSES
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT

CATALYST	HDS-1443				MOBIL HCL-3			AMOCAT 1A		
	MB-950	MB-951	MB-953	MB-956	MB-966	MB-968	MB-969	MB-980	MB-982	MB-983
OPERATING CONDITIONS										
TEMPERATURE, °F	725	777	776	791	802	792	729	807	777	727
LHSV, VFF/HR/VCAT	0.85	90.00	1.63	0.48	0.38	0.96	0.95	0.56	0.91	0.92
PRESSURE, PSIG	1975	1975	1975	1975	1795	1975	1975	2000	2000	2000
H2 CONSUMPTIONS, SCF/B	659	1573	1296	2150	3364	2338	1627	2045	1164	512
TLP PROPERTIES										
HYDROGEN, WT PCT	7.35	8.32	8.02	8.94	9.79	8.87	8.18	8.58	7.73	7.11
OXYGEN, WT PCT	2.38	1.25	2.08	0.78	0.17	1.37	2.66	1.15	1.54	2.89
NITROGEN, WT PCT	1.15	0.93	1.10	0.64	0.26	0.84	1.16	0.77	1.07	1.26
SULFUR, WT PCT	0.161	0.083	0.133	0.015	0.004	0.052	0.216	0.029	0.145	0.310
TLP YIELDS, WT PCT										
650°F-	52.1	55.5	50.2	59.2	67.0	55.8	47.0	58.0	48.6	40.4
SATURATES (CUT 1)	0.8	1.2	-	-	-	-	-	-	-	-
AROM. OILS (CUT 2-4)	8.7	13.0	-	-	-	-	-	-	-	-
RESINS/ASPH. (CUT 5-7)	16.2	17.7	-	-	-	-	-	-	-	-
POLAR ASPH. (CUT 8-13)	22.2	12.6	-	-	-	-	-	-	-	-
TOTAL	100.0	100.0	-	-	-	-	-	-	-	-
ELEMENTAL ANALYSES										
IBP-650°F										
HYDROGEN, WT PCT	8.90	9.86	9.09	10.20	9.38	9.61	8.73	8.95	7.98	8.46
OXYGEN, WT PCT	1.59	0.90	1.58	0.62	0.42	0.93	2.05	0.74	1.38	2.01
NITROGEN, WT PCT	0.511	0.456	0.539	0.264	0.141	0.356	0.519	0.364	0.502	0.606
SULFUR, WT PCT	0.020	0.005	0.022	0.002	0.003	0.007	0.207	0.006	0.040	0.180
650°F+ BTMS										
HYDROGEN, WT PCT	7.00	7.41	7.04	7.22	7.93	7.35	5.92	7.20	6.53	6.05
OXYGEN, WT PCT	2.98	1.54	2.38	0.53	0.95	1.54	3.48	1.21	3.11	4.09
NITROGEN, WT PCT	1.71	1.62	1.73	1.31	0.78	1.21	1.28	1.34	1.42	1.53
SULFUR, WT PCT	0.33	0.16	0.25	0.06	0.06	0.06	0.36	0.07	0.21	0.45

Figure 5-7

POLAR ASPHALTENES CONVERSION AS A FUNCTION OF HYDROPROCESSING SEVERITY



Harshaw 618X, which is lacking in 200 Å$^+$ diameter pores (cf. Table 3-1). Mobil HCL-2 converts polar asphaltenes in a manner similar to Harshaw 618X. This low conversion selectivity is a desirable catalytic property when a high yield in boiler fuel is an economic necessity.

Resins (GEC cuts 5-7) are reaction intermediates for the conversion of SRC to oils, saturates and 650°F$^-$ materials, and have a maximum concentration in reaction path as shown in Figure 5-8. The location of the maximum concentration is dependent on the ratio of the formation and conversion rates of the resins. The kinetics of the oils, saturates, and 650°F$^-$ materials are much more complex. They are determined by catalyst activity as well as the dynamic concentration of resins in the system, which in term is controlled by the hydrogenation activity. Mobil HCL-2, a low hydrogenation/desulfurization selectivity catalyst (cf. section 3), is found to be less active for formation of oils, saturates, and 650°F$^-$ materials than Harshaw 618X, as shown in Figure 5-9. Consequently, the crossover of oils, saturates, and 650°F$^-$ materials for Harshaw 618X and Amocat 1B appears to be due to the high dynamic concentration of the resin coupled with the low hydrogenation activity of Amocat 1B.

The low desulfurization and high conversion properties of Amocat 1B can also be shown by the sulfur distribution of the products. In Figure 5-10 the ratio of the sulfur concentration in the 650°F$^-$ fraction to that in the 650°F$^+$ fraction is plotted as a function of hydroprocessing severity. For all catalysts except Amocat 1B, the ratio decreases monotonically with increasing process severity. These results, along the high conversion of polar asphaltenes (Figure 5-7), are consistent with the high activity of Amocat 1B in coal hydroliquefaction processes.

Hydrotreated H-Coal Fuel Oil

The charge and six hydrotreated products from our H-Coal fuel oil run, reported in the 1978 Annual Report (1), have now been distilled to obtain IBP-650°F and 650°F$^+$ fractions. The distillation yields and elemental analyses are given in Table 5-6.

The objective in hydroprocessing this low sulfur stock was to reduce its nitrogen content and increase its hydrogen content to produce an acceptable gas turbine fuel. Figure 5-11 gives a plot of nitrogen versus hydrogen for the total liquid product and the IBP-650°F and 650°F$^+$ fractions. These products cover a range of hydrogen contents from 7.1 to 10.9 wt % and a range of nitrogen contents from

Figure 5-8

ASPHALTENES AND RESINS CONVERSION AS A FUNCTION OF HYDROPROCESSING SEVERITY, W. KENTUCKY SCT SRC

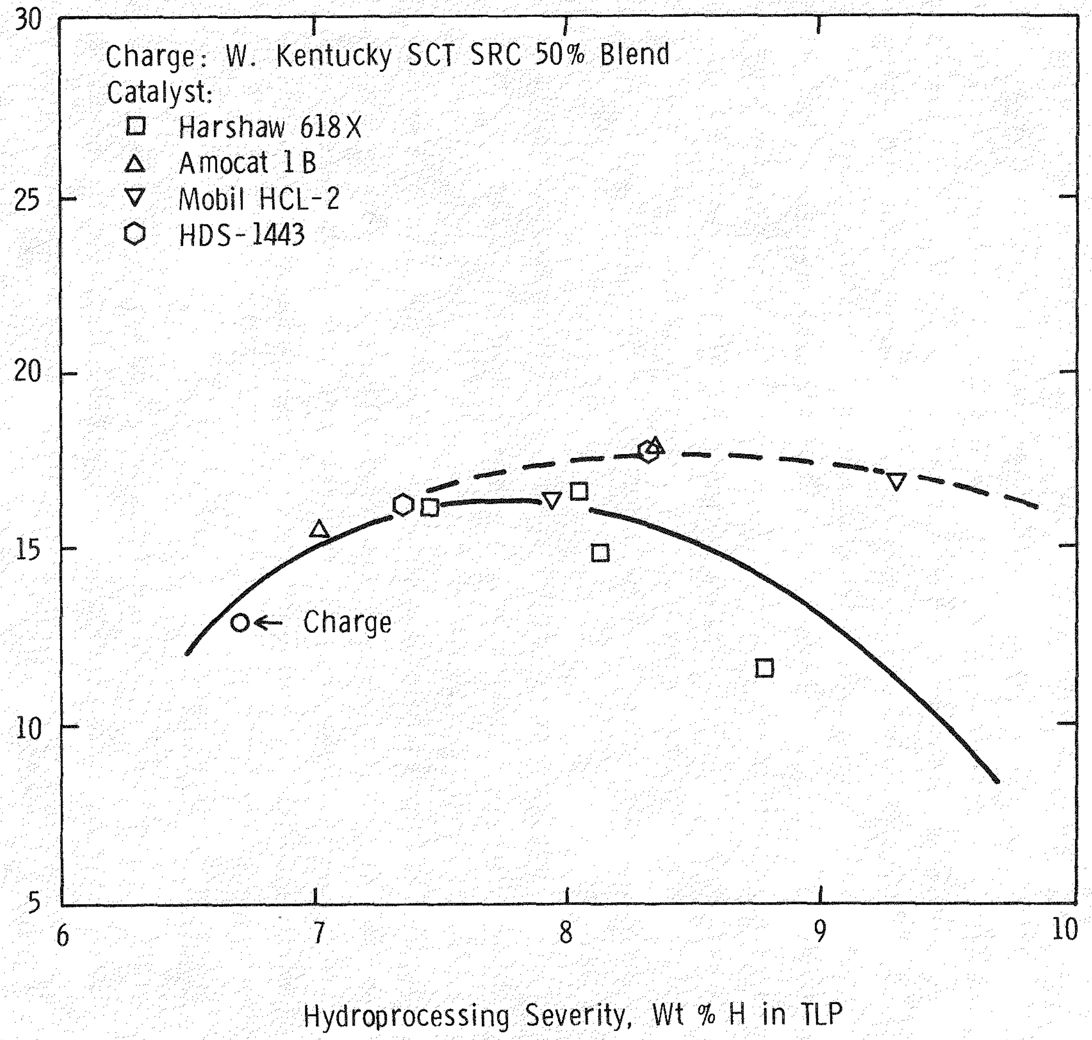


Figure 5-9

OILS, SATURATES, AND 650°F⁻ CONTENT OF SCT SRC AS A FUNCTION OF HYDROPROCESSING SEVERITY

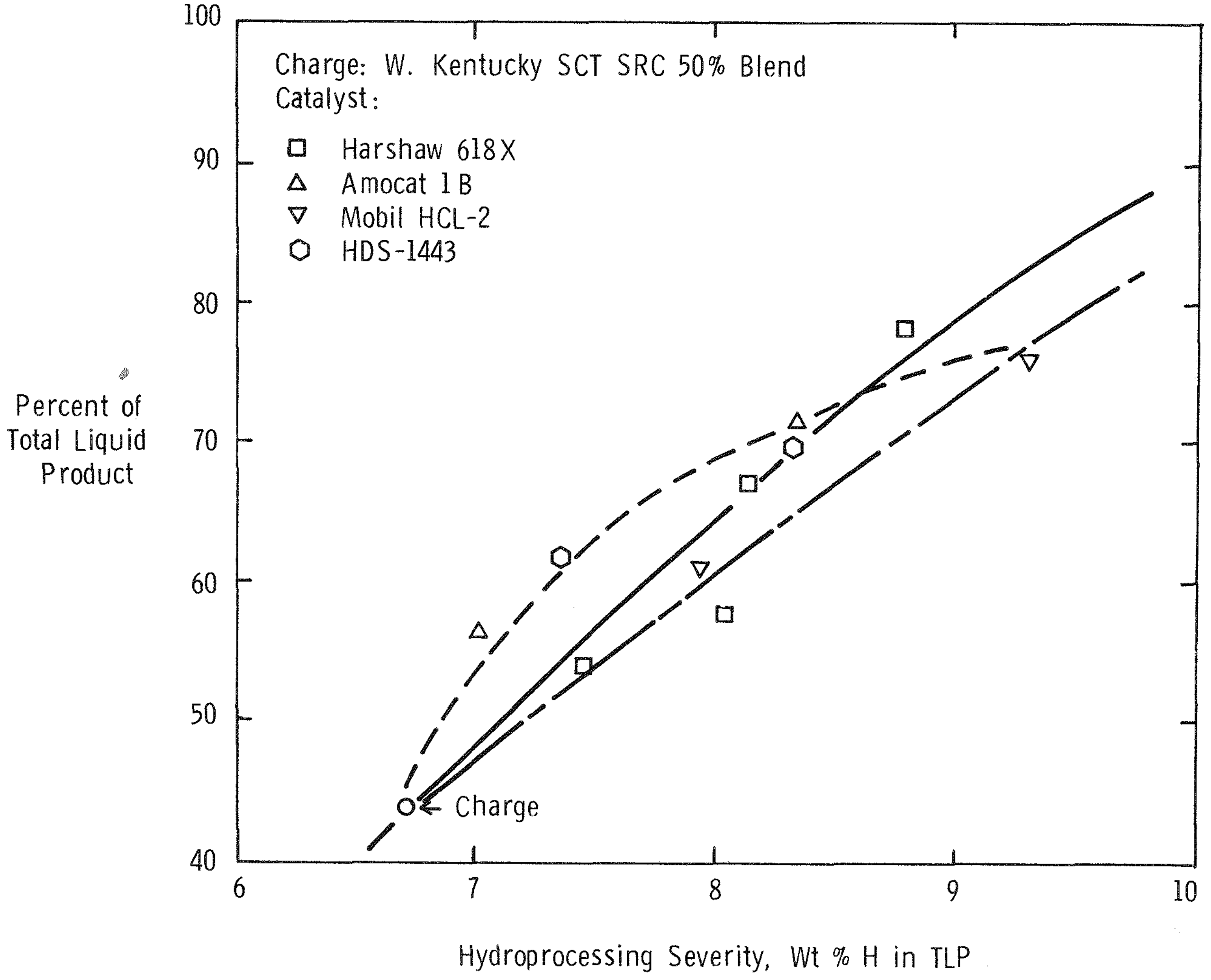


Figure 5-10

DISTRIBUTION OF SULFUR IN SRC AS A FUNCTION OF HYDROPROCESSING SEVERITY

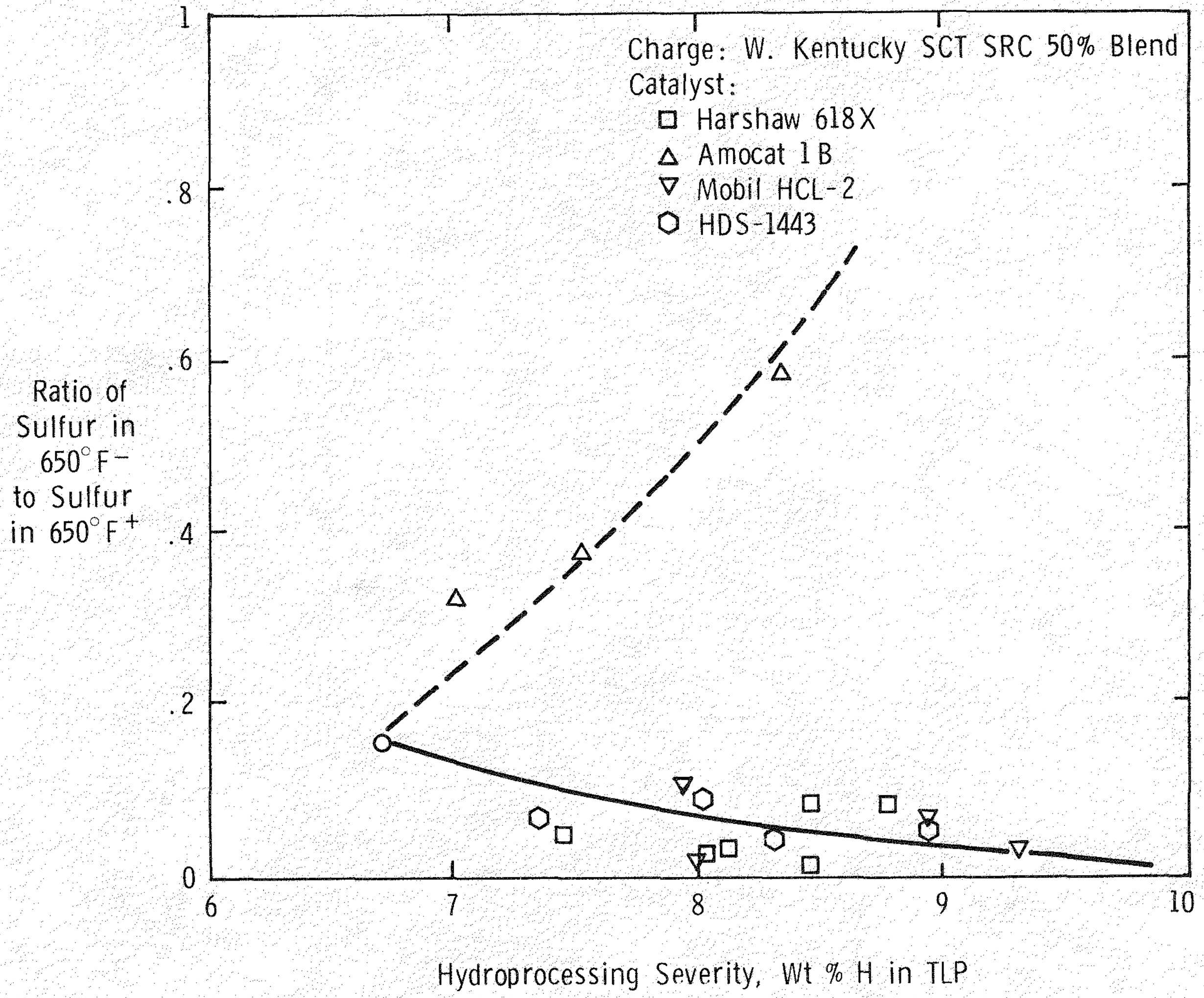


Table 5-6

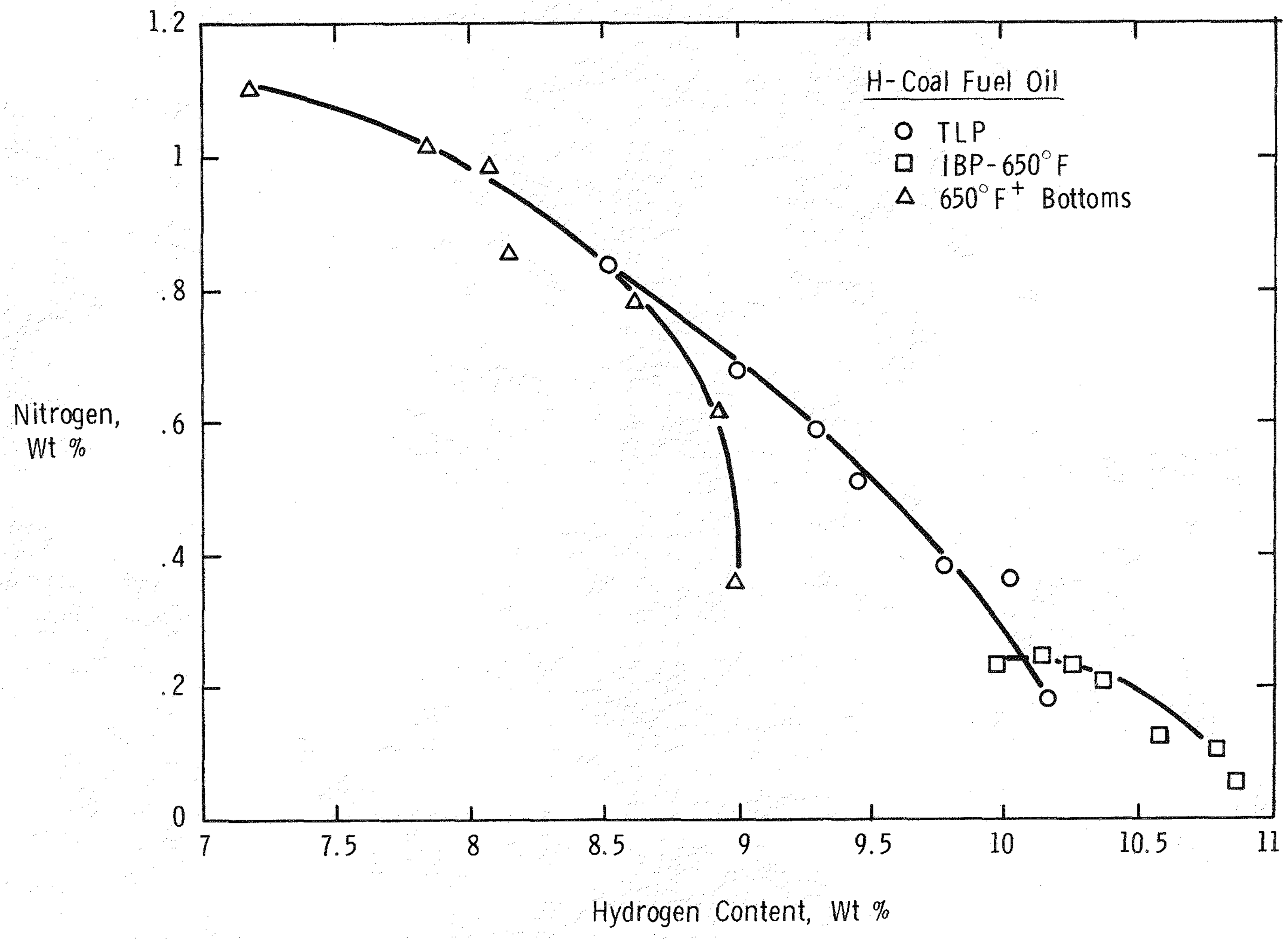
DISTILLATION CUT YIELDS AND PROPERTIES FOR RAW AND HYDROTREATED H-COAL FUEL OIL

UNIT: 146 PROGRAM: 2469

	Charge J-8723	MB-812	MB-813	MB-814	MB-815	MB-816	MB-817
Temperature, °F	-	674	729	779	675	724	775
LHSV	-	1.00	1.01	1.00	0.36	0.35	0.36
Pressure, psig	-	2000	2000	2000	2000	2000	2000
H_2 Consumed, SCF/Bbl	-	409	803	1058	635	1309	1946
Total Liquid Product							
H, wt %	8.49	8.97	9.43	9.76	9.27	10.01	10.16
O, " "	1.70	0.70	0.50	0.10	0.70	0.60	<0.10
N, " "	0.84	0.68	0.51	0.38	0.59	0.36	0.18
S, " "	0.23	0.11	0.06	0.03	0.09	0.05	0.02
650°F$^-$, wt %	44.9	46.8	53.1	52.5	50.3	51.2	62.1
IBP-650°F Cut							
H, wt %	9.96	10.14	10.36	10.58	10.24	10.80	10.87
O, " "	1.08	0.74	0.57	<1.10	0.70	0.30	0.11
N, " "	0.227	0.256	0.213	0.125	0.235	0.102	0.051
S, " "	0.045	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
650°F$^+$ Btms.							
H, wt %	7.13	7.80	8.12	8.58	8.04	8.90	8.97
O, " "	2.89	1.76	0.83	0.71	1.37	0.86	0.52
N, " "	1.11	1.02	0.86	0.79	0.99	0.62	0.36
S, " "	0.32	0.14	0.11	0.03	0.11	<0.03	<0.03

Figure 5-11

NITROGEN VS. HYDROGEN FOR H-COAL FUEL OIL FRACTIONS



0.05 to 1.1 wt %. Therefore, upgraded H-Coal fuel oil could provide turbine fuels with a wide range of product qualities. The degree of upgrading and boiling range of the fuel would be dictated by the requirements of the particular turbine combustor.

5.2 MOLECULAR SIZE DISTRIBUTIONS OF RAW AND HYDROTREATED SRC

Previously we reported significant differences between regular SRC and short contact time SRC with respect to viscosity and reactivity (2). Besides differences in hydrogen and heteroatom contents, we speculated that molecular size differences might exist between the two SRC types. The molecular size distribution of Indiana V regular SRC and W. Kentucky SCT SRC, which both were hydroprocessed in the current program, were measured using gel permeation chromatography (GPC). As shown in Figure 5-12, W. Kentucky SCT SRC has a mean molecular size approximately 25% larger than Indiana V regular SRC. More than 20% of the short contact time SRC is found in molecules with effective diameters greater than 50 Å, compared to only 6% of the regular SRC. These results indicate that phase incompatibility as a result of hydroprocessing might be more pronounced in short contact time SRC, consistent with product inhomogeneity found in runs with HDN-1197 (cf. section 3). The GPC data represent the molecular size distributions under infinitely dilute conditions, and effective sizes may vary under hydroprocessing conditions because of molecular association at higher concentrations. In Figure 5-13 the molecular size distributions of raw and two hydrotreated W. Kentucky SCT SRC's are shown. The size of five SRC's (one raw and four hydroprocessed) are plotted as a function of product hydrogen content in Figure 5-14. The average size of the SRC is strongly dependent on processing severity, as represented by hydrogen content.

5.3 ANALYSES OF SHORT CONTACT TIME PROCESS SOLVENT

Two drums of short contact time process solvent arrived from Gulf in their original Wilsonville drums and were distilled to remove the material boiling below 475°F. Solid sludge material found in the bottom of one of these drums was not added to the still pot before distillation. These drums were only partially full, leaving only one drum of 475°F$^+$ solvent after taking 27.1 wt % overhead. The third drum, which Gulf had transferred to a new drum and also found to be contaminated, was saved. The analyses of the as-received solvent show it to be slightly higher in hydrogen and sulfur and lower in nitrogen than a previous solvent from a regular SRC operation (Table 5-7).

Figure 5-12

SIZE DISTRIBUTION OF SRC

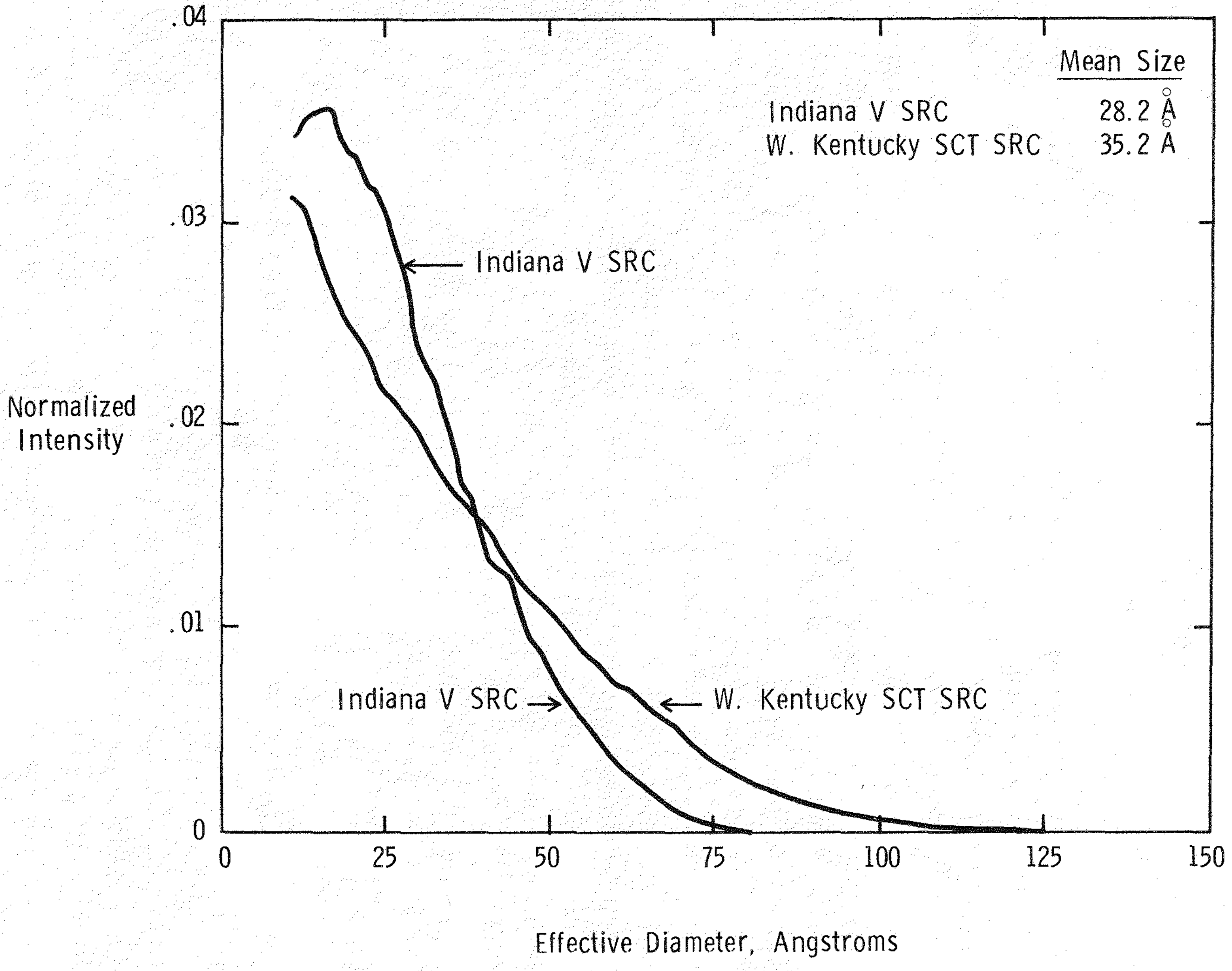


Figure 5-13

SIZE DISTRIBUTION OF RAW AND HYDROTREATED SRC

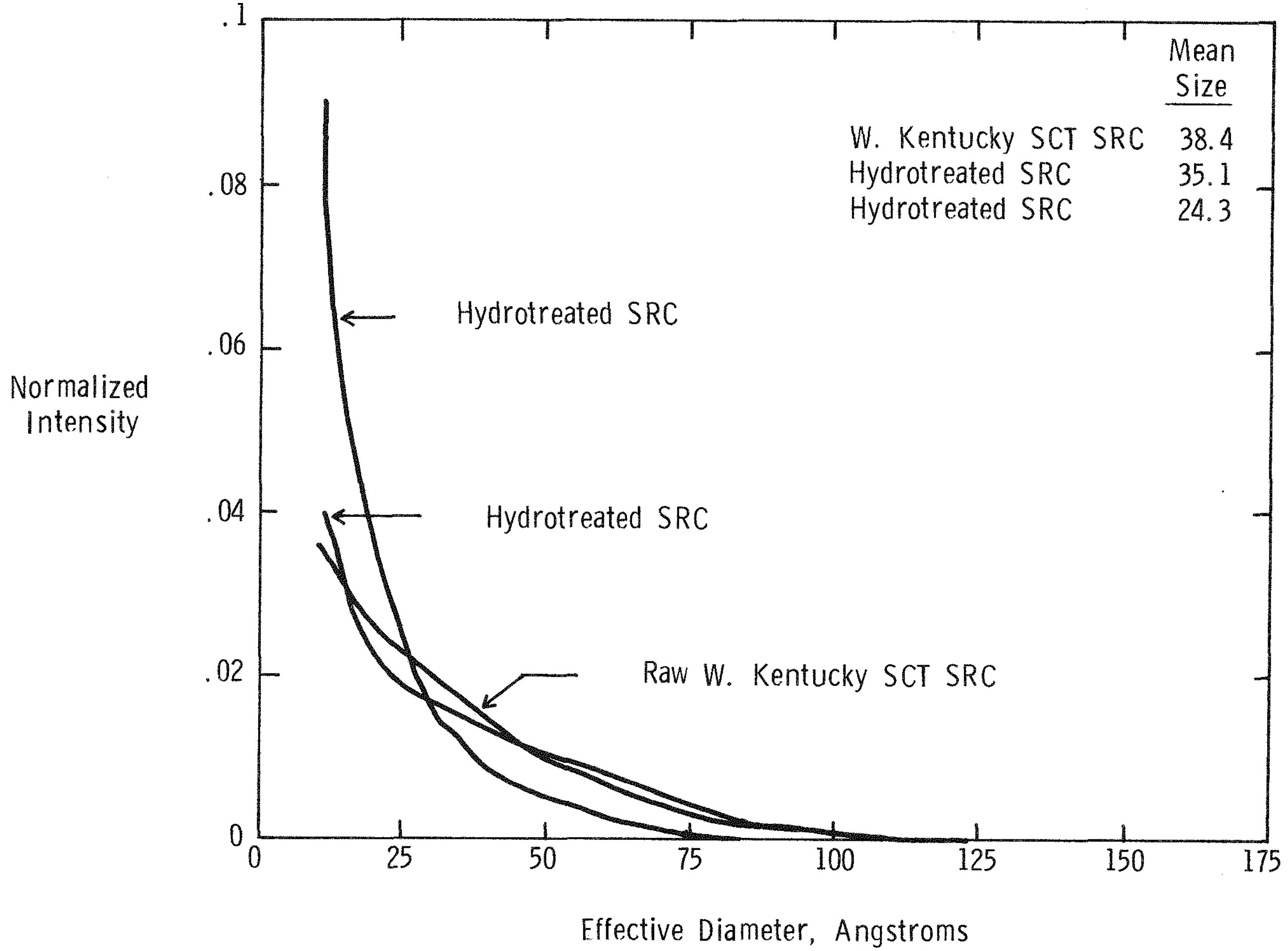


Figure 5-14

MEAN SIZE OF RAW AND HYDROTREATED SRC AS A FUNCTION OF HYDROGEN CONTENT

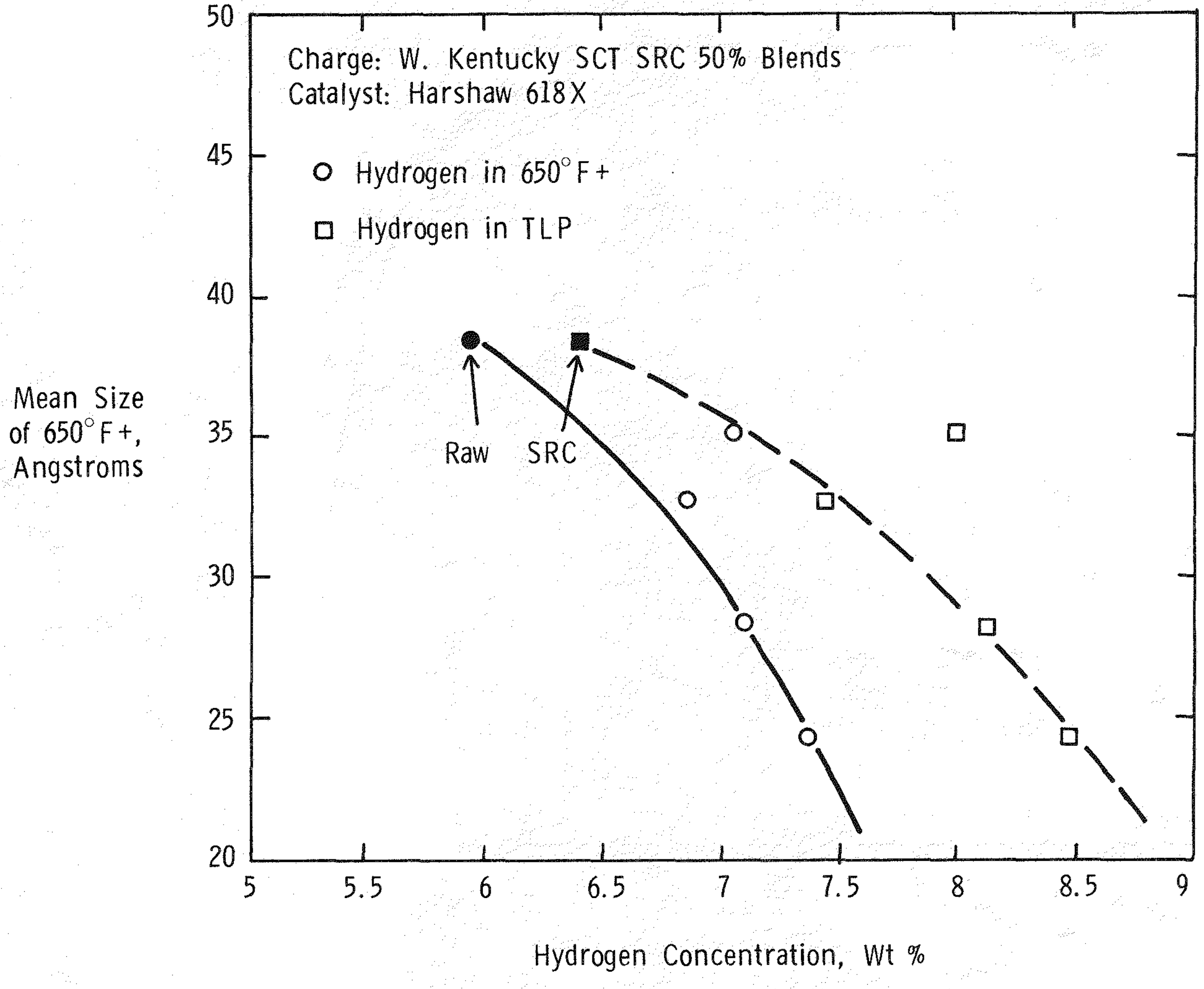


Table 5-7

PROPERTIES OF RECYCLE SOLVENTS

	Preheater Effluent Solvent	Regular Process Recycle Solvent	
		New	Old*
Mobil No.	78D1288	78D1591	75D3019
Sender No.	5786	34572	-
°API	7.3	7.7	5.5
Hydrogen, wt %	8.25	8.80,8.77	7.65
Oxygen, wt %	2.20	3.2	3.77
Sulfur, wt %	0.34	0.274	0.41
Nitrogen, wt %	0.52	0.88,0.93	0.45
Distillation, °F (D-2887)			
IBP	339	403	301
5%	400	433	336
10%	410	443	360
30%	453	481	400
50%	480	527	441
70%	518	603	509
90%	597	727	614
95%	625	784	653
EP	677	931	830

* Solvent was received in 1975.

An additional drum of short contact time SRC solvent was later received from Conoco along with two drums of recycle solvent from Wilsonville. The SCT solvent is similar to the previous samples obtained from Gulf. Properties are given in Table 5-7; the process recycle solvent is significantly different from previously analyzed recycle solvents. Although it is higher boiling than previous solvents, it has a higher API gravity and hydrogen content. It is apparently more highly saturated and has lower oxygen and sulfur contents. The higher average boiling point, however, leads to a higher nitrogen content (0.9 vs. 0.45 wt %). Except for the nitrogen, this solvent looks like a mildly hydroprocessed product from the old solvent.

5.4 STRUCTURAL COMPARISON OF SCT SRC RECYCLE SOLVENT AND PROCESS-GENERATED RECYCLE SOLVENT

At short contact times the SRC process is solvent deficient. Additional solvent range material can be obtained when the short contact time SRC is hydrotreated. The objective of this work was to compare the properties of SCT SRC recycle solvent and process-generated solvent. Solvent properties which may be important for primary coal dissolution were examined.

Three solvent range (460-720°F) samples were obtained from the liquid products in hydroprocessing runs with 50% W. Kentucky SCT SRC over Harshaw 618X. Yields and elemental analyses for these process-generated solvents are shown in Tables 5-8, 5-9, and 5-10; they are representative of solvents obtained by mild, moderate, and severe hydroprocessing conditions, respectively. The properties of the naphthas ($460°F^-$) and residual fuels ($720°F^+$) have been included for reference purposes. About 8 to 12% of the original solvent has been converted to naphtha, which has a hydrogen content above 10 wt %. The $720°F^+$ product is a clean residual fuel, having a sulfur content of 0.35 wt % or lower. Compared to the original solvent 78D-839 (Table 3-1), the process-generated solvents are higher in hydrogen content and lower in heteroatom content. These solvents have been sent to our Central Research Division Laboratory in Princeton where their use in the production of short contact time SRC will be evaluated. Two of the process-generated recycle solvents and the short contact time SRC recycle solvent were analyzed by GC-MS. The two extremes in processing severity (MB 905 and MB 911) were selected for analysis.

Sample GC-MS reconstructed total ion chromatograms are shown in Figures 5-15 and 5-16 for the SCT SRC recycle solvent and the (high severity) hydroprocessed recycle solvent (MB 911), respectively. Major peaks have been numbered and are listed in Tables 5-11 and 5-12, respectively. Approximate analyses of the three samples are shown in Table 5-13. The complexity of the samples can lead to co-elution of

TABLE 5-8

ANALYSES OF PROCESS-GENERATED DISTILLATES AND RESIDUAL FUEL
CHARGE: *W. KENTUCKY SCT SRC* 50°/° *BLENDS*
CATALYST: *HARSHAW* 618X
OPERATING CONDITIONS: 2000 *PSIG*, 768°*F*, 1.9 *LHSV*
UNIT: 146 *PROGRAM:* 2723 *MB:* 905

	TLP	IBP-460°F	460-720°F	720°F+
ELEMENTAL ANALYSES				
GRAVITY, API	-2.4	22.2	7.0	-17.3
HYDROGEN, WT PCT	7.45	-	8.96	6.69
SULFUR, WT PCT	0.25	0.01	0.05	0.35
NITROGEN, WT PCT	1.14	0.55	0.59	1.89
OXYGEN, WT PCT	2.40	1.75	1.67	3.46
TRACE METALS				
CALCIUM, PPM	-	-	100	100
SODIUM, PPM	-	-	1.6	7.1
YIELDS, WT PCT	100.0	4.3	48.2	47.5
DISTILLATION (D2887), °F				
10 VOL PCT	-	-	471	-
50 VOL PCT	-	-	539	-
90 VOL PCT	-	-	627	-

TABLE 5-9

ANALYSES OF PROCESS-GENERATED DISTILLATES AND RESIDUAL FUEL

CHARGE: *W. KENTUCKY SCT SRC* 50•/• *BLENDS*
CATALYST: *HARSHAW* 618X
OPERATING CONDITIONS: 2000 *PSIG*, 803•*F*, 1.4 *LHSV*
UNIT: 146 *PROGRAM:* 2723 *MB:* 908

	TLP	*IBP*-460•*F*	460-720•*F*	720•*F*+
ELEMENTAL ANALYSES				
GRAVITY, API	3.2	23.9	10.4	-18.8
HYDROGEN, WT PCT	8.13	11.18	9.01	7.20
SULFUR, WT PCT	0.13	-	0.01	0.14
NITROGEN, WT PCT	0.92	0.40	0.47	1.63
OXYGEN, WT PCT	1.70	1.26	1.14	2.37
TRACE METALS				
CALCIUM, PPM	-	-	100	200
SODIUM, PPM	-	-	2.5	2.8
YIELDS, WT PCT	100.0	6.4	36.3*	57.3
DISTILLATION (D2887), •F				
10 *VOL PCT*	480	-	483	-
50 *VOL PCT*	564	-	557	-
90 *VOL PCT*	-	-	609	-

**THE END BOILING POINT IS* 650•*F*.

TABLE 5-10

ANALYSES OF PROCESS-GENERATED DISTILLATES AND RESIDUAL FUEL
CHARGE: *W. KENTUCKY SCT SRC* 50•/• *BLENDS*
CATALYST: HARSHAW 618X
OPERATING CONDITIONS: 2000 *PSIG*, 791•*F*, 0.5 *LHSV*
UNIT: 146 *PROGRAM:* 2723 *MB:* 911

	TLP	*IBP*-460•*F*	460-720•*F*	720•*F*+
ELEMENTAL ANALYSES				
GRAVITY, API	9.6	27.1	9.5	-22.4
HYDROGEN, WT PCT	9.14	10.89	9.45	6.81
SULFUR, WT PCT	0.07	-	0.02	0.19
NITROGEN, WT PCT	0.66	0.29	0.42	1.53
OXYGEN, WT PCT	1.10	0.77	1.02	2.33
TRACE METALS				
CALCIUM, PPM	-	-	100	100
SODIUM, PPM	-	-	2.1	3.8
YIELDS, WT PCT	100.0	6.2	46.4	47.4
DISTILLATION* (*D*2887), •*F				
10 *VOL PCT*	328	-	499	-
50 *VOL PCT*	591	-	589	-
90 *VOL PCT*	-	-	675	-

Figure 5-15

GC-MS RECONSTRUCTED TOTAL ION CHROMATIOGRAM OF SRC RECYCLE SOLVENT (78D839)

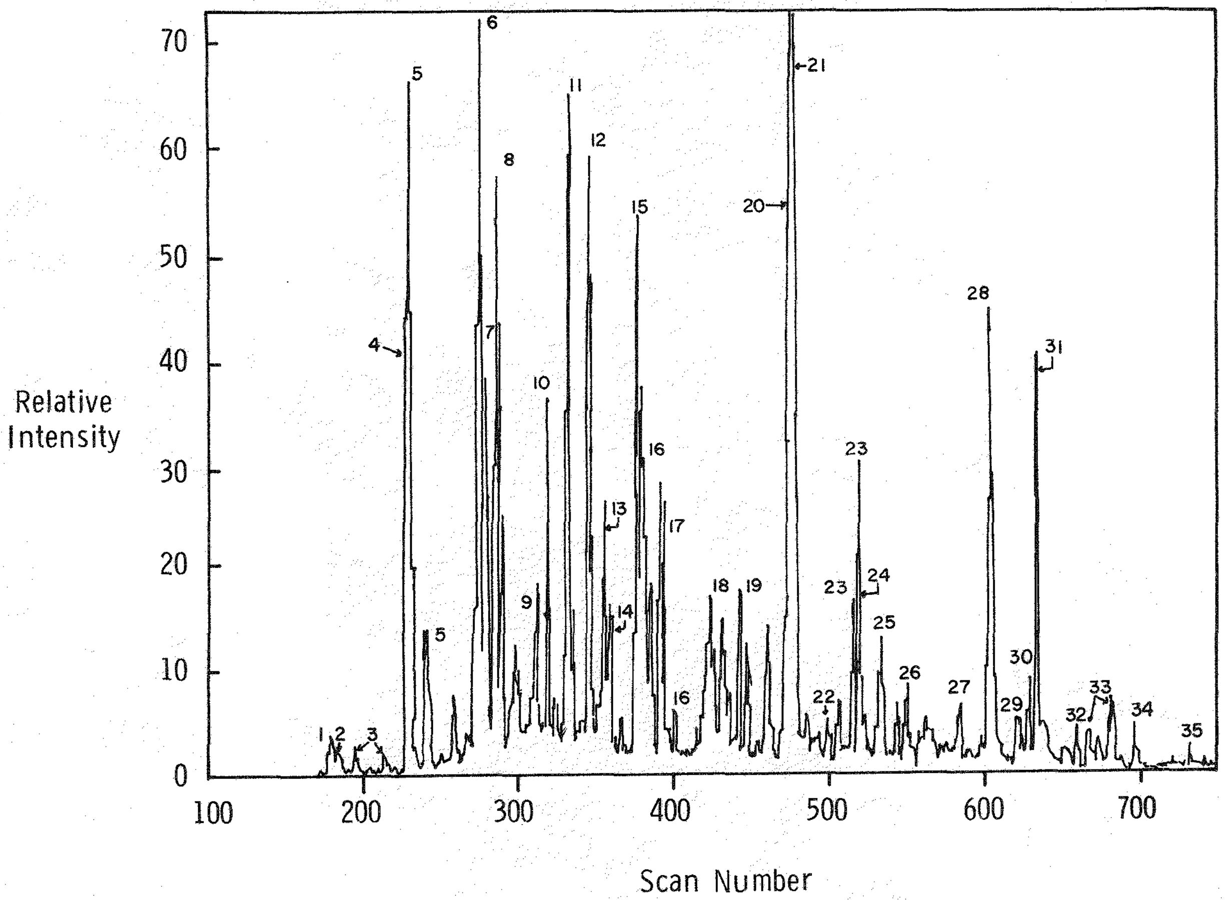


Table 5-11

GC-MS PEAK IDENTIFICATION FOR FIGURE 5-15; RECYCLE SOLVENT (78D839)

1	naphthalene
2	NC 14
3	methyl tetralins
4	NC 15
5	methyl naphthalenes
6	biphenyl
7	NC 16
8	diphenyl ether
9	pristane
10	NC 17
11	acenaphthene
12	dibenzofuran
13	phenyl-benzyl ether + pristanes
14	NC 18
15	fluorene
16	methyl dibenzofurans
17	NC 19
18	NC 20
19	dibenzothiophene
20	NC 21
21	anthracene
22	NC 22
23	methyl anthracenes
24	carbazole
25	methyl anthracenes
26	NC 23
27	NC 24
28	fluoranthene
29	NC 25
30	dihydro pyrene
31	pyrene
32	NC 26
33	methyl fluoranthenes + pyrenes
34	NC 27
35	NC 28

Figure 5-16

GC-MS RECONSTRUCTED TOTAL ION CHROMATOGRAM OF
460-720°F FRACTION OF HYDROTREATED
50% W. KENTUCKY SCT SRC; MB 911

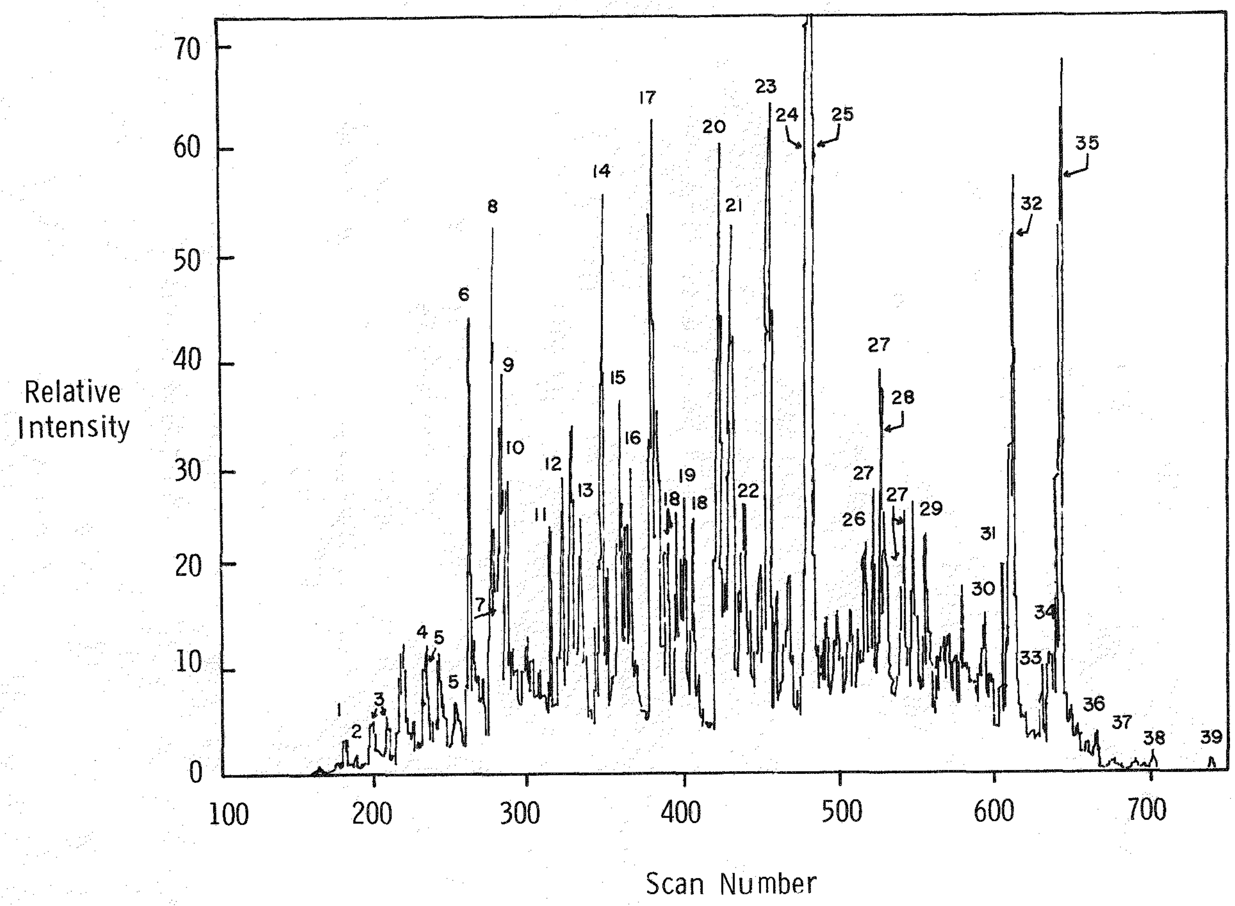


Table 5-12

GC-MS PEAK IDENTIFICATION FOR FIGURE 5-16;
460-720°F FRACTION OF HYDROTREATED
50% W. KENTUCKY SCT SRC FROM
CT-146-2723-911

1 naphthalene
2 NC 14
3 methyl tetralins
4 NC 15
5 methyl naphthalenes
6 cyclohexyl benzene
7 NC 16
8 biphenyl
9 methyl cyclohexyl benzenes
10 diphenyl ether
11 pristane
12 NC 17
13 acenaphthene
14 dibenzofuran
15 phytane + phenyl benzyl ether
16 NC 18
17 fluorene
18 methyl dibenzofurans
19 NC 19
20 octa hydro anthracene
21 di hydro anthracene
22 NC 20
23 tetra hydro anthracenes
24 NC 21
25 anthracene
26 NC 22
27 methyl anthracenes
28 carbazole
29 NC 23
30 NC 24
31 dihydrofluoranthene
32 fluoranthene
33 NC 25
34 dihydropyrene
35 pyrene
36 NC 26
37 methyl pyrenes + fluoranthenes
38 NC 27
39 NC 28

Table 5-13

SRC Recycle Solvent: GC Analysis of Major Components

	475+ Solvent 78D839 (wt %)	a) MB-905 (wt %)	b) MB-911 (wt %)
Naphthalene	0.46	-	-
1-Methylnaphthalene	3.66	0.63	0.44
2-Methylnaphthalene	1.30	0.25	0.20
Cyclohexyl-benzene	-	0.57	0.96
Biphenyl	3.90	3.61	2.72
Diphenyl Ether	1.69	0.97	0.71
Acenaphthene	6.42	4.30	2.29
Dibenzofuran	1.28	1.07	0.59
Phenylbenzyl Ether	3.37	2.79	2,03
Fluorene	2.98	3.45	3.35
Octahydroanthracene/Phenanthrene	-	1.94	2.71
Dihydroanthracene/Phenanthrene	-	1.57	1.52
Tetraanthracene/Phenanthrene	-	4.89	4.76
Anthracene/Phenanthrene (+ NC 21)	8.87	7.48	5.10
Dihydro-fluoranthrene	-	1.57	1.76
Flouranthrene	3.09	2.26	3.52
Pyrene + Dihydro Pyrene	2.46	2.99	4.17

a) Low Severity Processing

b) High Severity Processing

different components (e.g., anthracene/phenanthrene and NC 21). The process-generated recycle solvents are generally more hydroaromatic than the SRC solvent. The identifiable hydrogenated products arise mainly from bare ring polynuclear aromatics (PNA's) and bare ring heteroatomic species. Specific alkylated homologs are not easily identified due both to their low concentrations and the wide range of possible stereochemical variations. The major hydrogenated products observed are tetralins, hydrogenated anthracenes, fluoranthrene and pyrene, and cyclohexyl benzene. The amount of tetralins present was limited by the initial boiling point (IBP). A shift in the IBP will strongly affect the concentration of tetralin. While more hydrogen donors are produced for the recycle process, the total solvent hydrogen donability may be reduced due to the low level of phenols and tetralins. These results indicate that a lower IBP for the recycle solvent may be needed to insure an adequate concentration of hydrogen donors.

The recycle solvents were also analyzed for heteroatom compounds. The major oxygen compounds observed by GC-MS are dibenzofuran, diphenyl ether, and benzyl phenyl ether. Small amounts (2-4%) of phenolic material are observed by IR in the starting SRC recycle solvent. The IBP of the process-generated solvents excludes most phenols, which was confirmed both by IR and GC-MS.

As shown in Figure 5-17, the sulfur compounds of the SCT SRC recycle solvent and the process-generated recycle solvent from MB 911 were determined by gas chromatography using a sulfur-specific detector. Major components have been labeled. Sulfur compounds in the SCT SRC recycle solvent are C_1-C_3 benzothiophene, dibenzothiophenes, and C_1-C_3 homologs, and a high boiling unidentified compound. The hydrogenated solvent was significantly lower in sulfur content (0.49 vs. 0.02 wt %), with the remaining compounds being dibenzothiophene and C_1-C_2 homologs.

The nitrogen compounds were also determined by gas chromatography using a nitrogen-specific detector, as shown in Figure 5-18. The nitrogen compounds of the SCT SRC recycle solvent are distributed among indoles, dihydroindole, quinoline, isoquinoline, tetrahydroquinoline, isoquinoline, benzoquinoline, benzoisoquinoline, acridine, and carbazole. The nitrogen removal, upon hydroprocessing, occurs mainly in compounds boiling below anthracene and phenanthrene, as determined with a flame ionization detector. Little or no reduction in carbazole concentration occurs during hydroprocessing.

Figure 5-17

CHROMATOGRAMS OF COAL-DERIVED SOLVENTS USING SULFUR-SPECIFIC DETECTION

(A) SCT SRC Recycle Solvent (78 D 839)
(B) 460-720° F Fraction From CT-146-2723-911

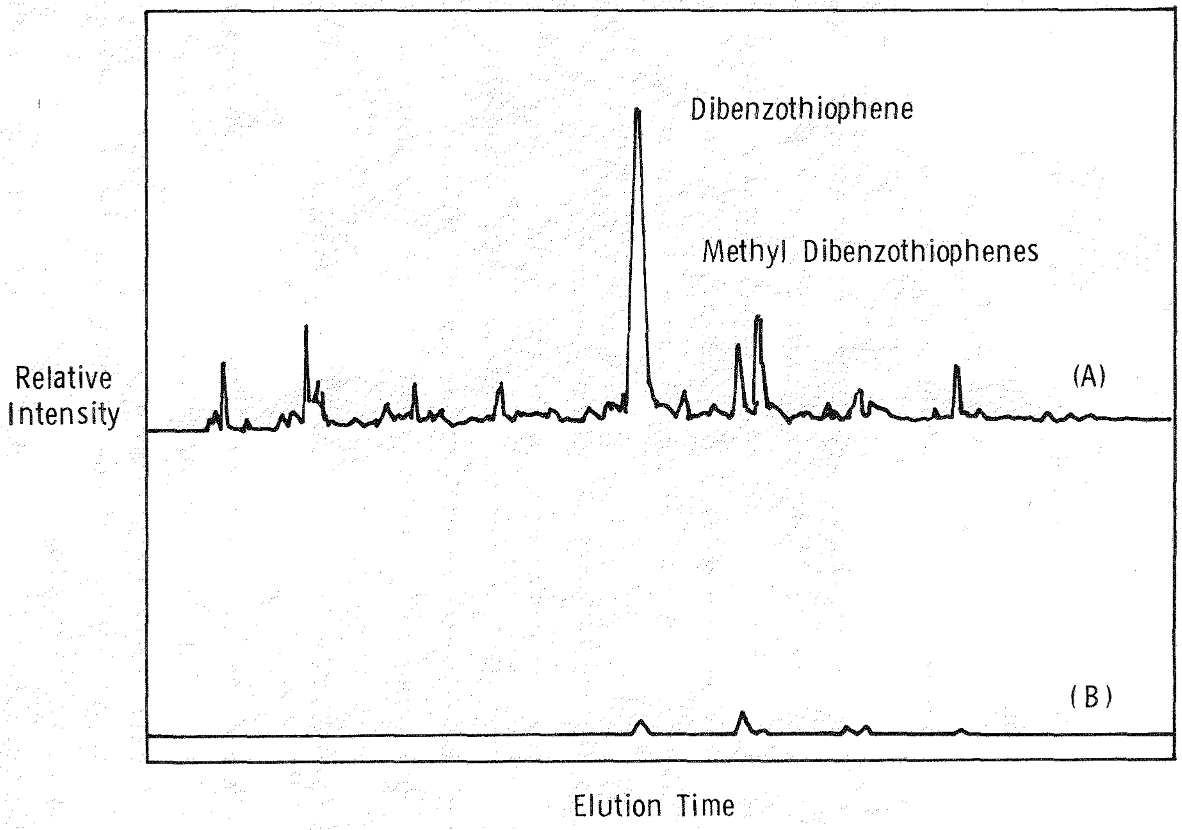
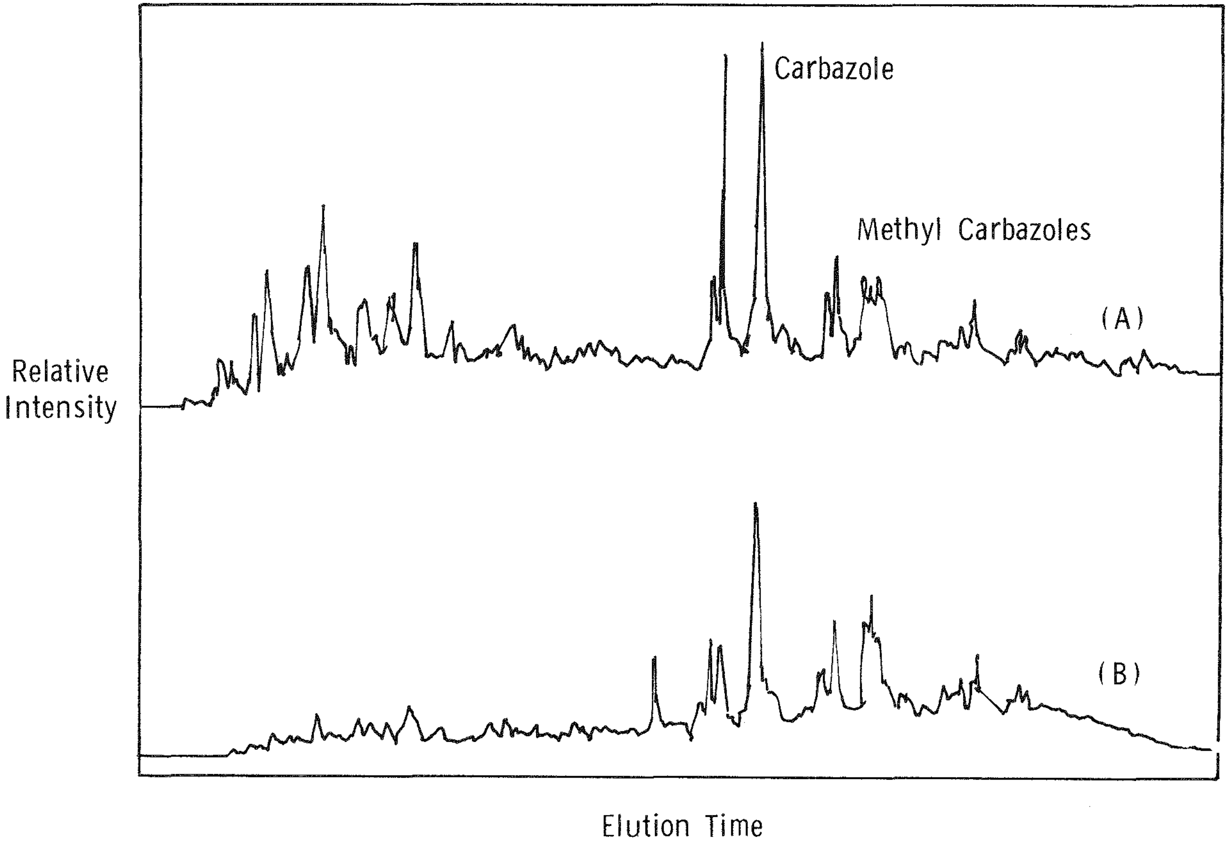


Figure 5-18

CHROMATOGRAMS OF COAL-DERIVED SOLVENTS USING NITROGEN-SPECIFIC DETECTION

(A) SCT SRC Recycle Solvent (78D839)
(B) 460-720° F Fraction From CT-146-2723-911



5.5 ALKALI METALS IN COAL-DERIVED DISTILLATE

In the 1978 Annual Report samples of H-Coal distillates were found to have 1.1 ppm of alkali metals (sodium, potassium, and calcium), significantly higher than the turbine fuels specification of 0.2 ppm alkali metals. During the past year efforts were made to elucidate the origin of the alkali metals, the chemical nature of the alkali metals in the coal-derived distillates, and the feasibility of several separation techniques.

In a short literature review it was found that the inorganic material in coal can occur as a result of incorporation during formation, from percolation of mineral-containing water at later periods or from the inorganic minerals inherent in the organic structure of the coal-forming vegetation. This third source is relatively small compared to the first two.

The major sodium-containing minerals are the silicates and aluminosilicates such as muscovite, illite and montmorillonite. Halite is also a common source of sodium in coal. The alkali metals present in the living vegetation are present to act as mobile charge carriers. While performing their function, these metals are in the form of complexes with cyclic oxygen donors for a brief time.

However, these complexes are not likely to survive any type of chemical or thermal processing. Sodium salts of organic acids are also unlikely contaminants as they are likely to decompose during processing or be retained in the bottoms during distillation. Therefore, it is believed that the alkali metals in distillate coal liquids are due primarily to contamination or to physical carry-over of inorganic ash in the SRC process. Several simple experiments were performed to substantiate this, as described below.

The short contact time recycle solvent was analyzed to determine the levels of sodium, potassium and water present. A sample was then placed in five different containers. The containers were thoroughly cleaned, rinsed with de-ionized water, filled with the coal liquid and allowed to stand for 45 days. After standing the samples were again analyzed for sodium and potassium. Table 5-14 shows the results of the analyses. The only sample to show a significant difference in the level of contaminants was that stored in a steel can. This sample had more than twice the potassium and 50% more sodium than the other samples.

To obtain information on the nature of the alkali metals, samples of SRC preheater effluent solvent were washed and distilled. In the washing experiments, 150 ml of

Table 5-14

SODIUM AND POTASSIUM IN PREHEATER EFFLUENT SOLVENT (78D-792)

			After 45 Days Storage in				
	Original	Duplicate	Steel	Soft Glass	Epoxy-Lined Steel	Polyethylene	Teflon
Na, ppm	0.79	0.66	0.99	0.64	0.69	0.70	0.69
K, ppm	0.46	0.28	0.86	0.42	0.40	0.32	0.32
H_2O, wt %	0.23	0.25	-	-	-	-	-

recycle solvent was extracted three times with 50 ml portions of de-ionized water in a separatory funnel. (All glass was acid-washed and rinsed with de-ionized water.) The layers were separated by centrifugation. A clean separation was not possible because the layers did not form a distinct boundary. A 20% volume loss of recycle solvent to water occurred. A stable emulsion forms if the extraction is performed too vigorously. An identical washing experiment with 0.1 M HCl was also performed.

A vacuum distillation of the recycle solvent was carried out in a vacuum jacketed Vigreux column with automatic take-off. Starting with 170 ml of recycle solvent and distilling at 1 mm mercury pressure, a total of 130 ml of distillate was collected in a cold trap.

The distillate consisted of a fraction collected from 386°F to 603°F (adjusted to 760 mm mercury). The 76.5 wt % overhead was pale yellow in color. The $603°F^+$ material remaining in the pot was very dark in appearance and semi-solid.

The results of the above experiments, shown in Table 5-15, indicate that the sodium and potassium contents were reduced by an average of about 55% and 77%, respectively, on washing.

The addition of acid to the water appears to have little effect on the results. The sulfur was also reduced on water washing. The nitrogen content was unaffected. The distillation results show that sodium and potassium were reduced by 60 and 90%, respectively, in the overhead (76.5 wt % of sample).

As shown in Table 5-15, the alkali metals can be reduced significantly by water washing or distillation. This indicates that these metals are not organically bound in recycle solvent.

Table 5-15

PROPERTIES OF SHORT CONTACT TIME SRC RECYCLE SOLVENT

	As Received 78D-792	Water Washed	Acid Washed	Distillate 385°F to 600°F	600°F+
Hydrogen, wt %	8.02	8.25	8.13	8.13	7.59
Oxygen, wt %	3.45	3.95	3.80	3.69	3.62
Sulfur, wt %	0.42	0.197	0.31	0.244	0.72
Nitrogen, wt %	0.65	0.66	0.59	0.53	1.07
Water, wt %	0.23	4.57	1.63	0.14	0.09
Sodium, ppm	0.79	0.39	0.32	0.31	1.7
Potassium, ppm	0.46	0.10	0.11	0.04	0.86
Distillation, °F (D-2887)					
IBP	367	366	367	363	578
5%	404	402	402	401	602
10	428	431	431	412	625
30	477	475	476	457	660
50	512	511	517	488	685
70	579	581	585	531	710
90	662	656	666	586	752
95	691	679	694	592	775
EP	768	746	796	625	1024

Section 6

MODELING

6.1 MODELING OF CONCENTRATED MONTEREY SRC BLENDS

Preliminary results showing the effect of solvent concentration on the hydroprocessing of Monterey regular SRC were previously reported in the 1978 annual report. Using CCR content as an index of conversion of SRC, it was shown that at constant SRC space velocity a maximum CCR reduction is achieved when charging the SRC in a blend containing about 30 to 50% solvent. This would suggest that either the SRC inhibits its own conversion through strong adsorption on the catalyst surface or the recycle solvent is acting as a hydrogen donor and aiding in the conversion process. Another factor which may contribute to the observed behavior is the significant change in fluid properties (viscosity, density, and surface tension) with the addition of recycle solvent to the SRC.

Modeling of the heteroatom and CCR removals as a function of SRC concentration was performed to better quantify the observed data. The kinetic model takes into account the temperature, space velocity, catalyst age, and solvent concentration. The aging rate was treated as a function of the SRC concentration because the SRC, with its higher heteroatom and CCR content, presumably has more of a deactivating affect than does the recycle solvent. The rate expression is then:

$$\frac{dC_i}{dt} = - k \, x_s^n \, C_i^m \exp\left\{- \frac{E}{RT}\right\} \exp\left\{- \frac{(1 + K_D \, x_{SRC}) \, D}{\tau}\right\} \qquad (6\text{-}1)$$

where

C_i = concentration of CCR or heteroatom, wt %

x_s = solvent weight fraction

x_{SRC} = SRC weight fraction

k = rate constant

n, m, K_D = constants

E = activation energy, Btu/lb mole

D = days on-stream, day

τ = catalyst deactivation constant, day

In solving Equation 6-1, the solvent weight fraction, x_s, was assumed to remain constant at the feed value throughout the reactor. This is approximately true at most processing conditions. Upon integration, Equation 6-1 yields:

$$C_i = \left[C_{i_o}^{1-m} - k\ (1-m)\ x_{s_o}\ \exp\left\{-\frac{E}{RT}\right\} \exp\left\{-\frac{(1 + K_D\ x_{SRC})}{\tau}\right\} t \right]^{\frac{1}{1-m}} \qquad (6\text{-}2)$$

where $t = LHSV^{-2/3}$

The 2/3-power dependence of LHSV is attributable to hydrodynamic phenomena in trickle bed reactors (1).

The values of the constants in Equation 6-2 for the CCR and heteroatom removals were determined using a modified Marquardt fitting routine on the 32 material balances from the Monterey SRC/solvent blends studies. As a qualitative measure of the fit, Figure 6-1 shows the predicted CCR removal vs. the observed CCR removal. The constants determined for CCR and heteroatom removal are:

	k	n	m	K_D	τ	E	mean deviation
	$[hr^{-1}]$	-	-	-	[days]	[Btu/lb mole]	[wt %]
CCR	5.7×10^3	0.49	1.28	12.8	609	23.2×10^3	1.77
Nitrogen	3.9×10^4	0.23	0.76	93.3	1796	26.3×10^3	0.06
Sulfur	3.5×10^4	0.25	1.10	32.5	1860	23.2×10^3	0.04
Oxygen	4.3×10^6	0.20	1.41	23.5	516	43.3×10^3	0.23

It should be pointed out that although Equation 6-1 fits the data well and predicts observed trends, it is of a semi-empirical nature and will break down if extrapolated to extremes. For example, the reaction rate will not drop to zero if the solvent is removed.

The reaction rates are all close to first (0.76 - 1.41) order in reactant concentration and are 0.2 - 0.49 order in solvent concentration. The CCR, which is present only in the SRC component of the blends, is most strongly affected by the presence of recycle solvent. The trends observed previously in CCR removal are predicted well by this model. Figure 6-2 shows the predicted effect of SRC concentration upon CCR removal at three different SRC space velocities. A maximum in conversion (i.e., CCR removal) occurs at the 60-70% SRC concentration level.

Figure 6-1

PREDICTED CCR VS OBSERVED CCR: MONTEREY SRC / SOLVENT BLENDS

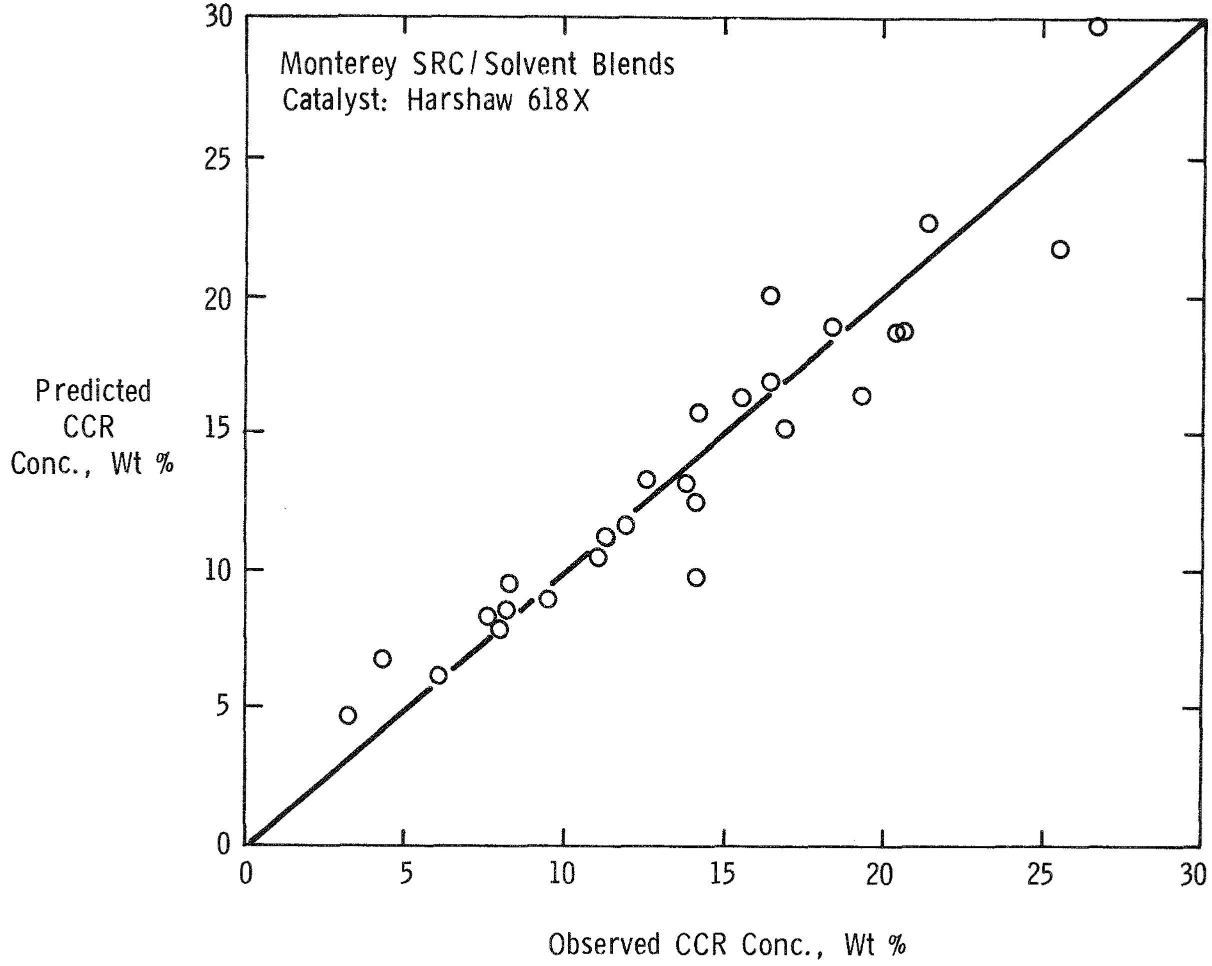
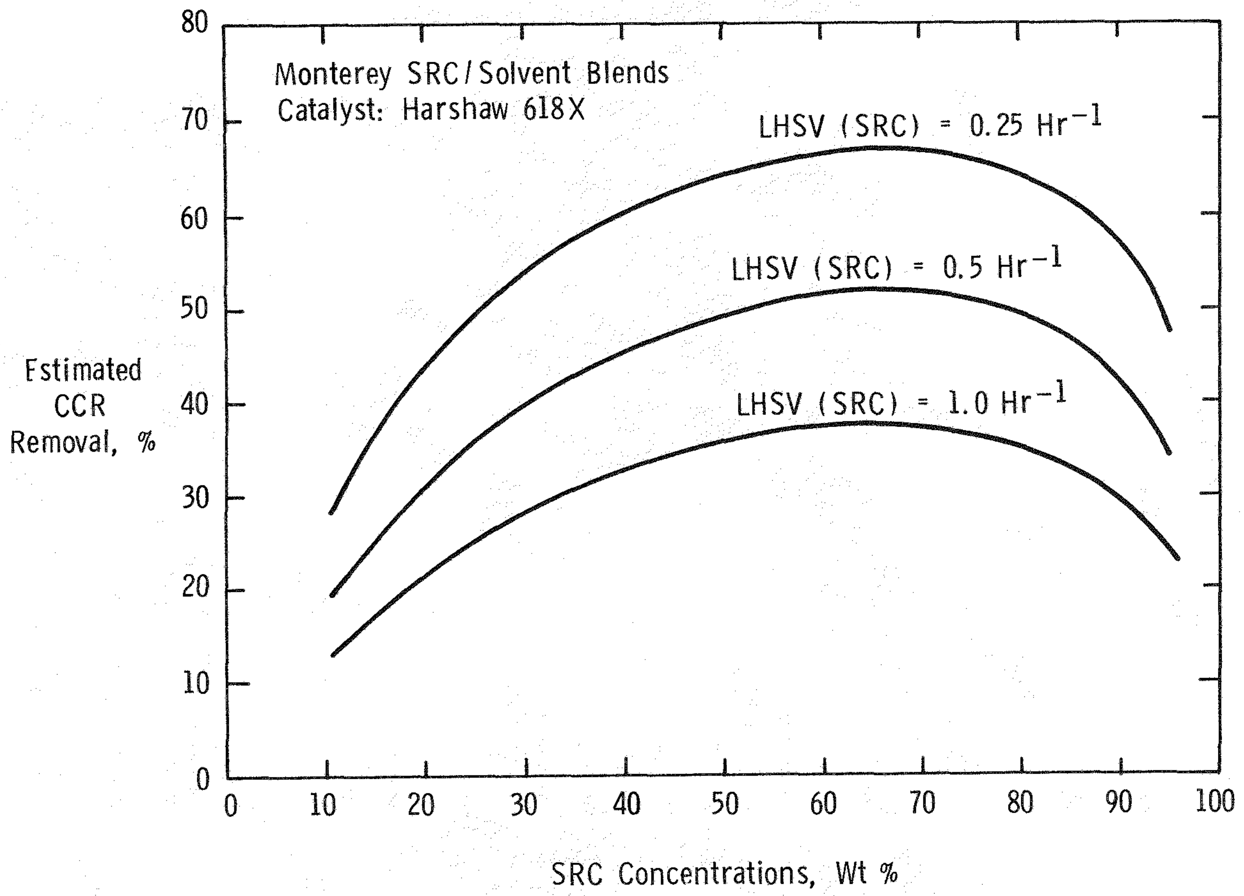


Figure 6-2

ESTIMATED CCR REMOVAL VS SRC CONCENTRATION:
MONTEREY SRC / SOLVENT BLENDS



Sulfur-, oxygen-, and nitrogen-containing molecules are present in both the SRC and the recycle solvent. However, since the quantities and types of heteroatom-containing molecules vary between the solvent and SRC, the addition of recycle solvent to the SRC changes the nature of the heteroatoms in the blend. This solvent addition may also facilitate the removal of the heteroatoms from the SRC. These two effects, however, are difficult to separate. Figure 6-3 shows the predicted effect of SRC concentration on nitrogen removal at three different space velocities.

Although the short-term run data used for this model (maximum days on-stream: 15 days) are not sufficient to accurately determine catalyst deactivation rates, some features of the catalyst deactivation can be derived from the model. The high values for K_D indicate that the catalyst deactivation is strongly dependent on SRC concentration. Figure 6-4 demonstrates the predicted aging for nitrogen removal. The model indicates that catalyst deactivation is not uniform for all heteroatom removals. This can be shown by taking the ratio of K_D to τ:

	K_D/τ
CCR	0.021
S	0.018
O	0.046
N	0.056

The high deactivation ratio (rapid deactivation) for the nitrogen removal may be due to the relative complexity of the denitrogenation reactions, known to involve both saturation and hydrogenolysis steps.

6.2 HEATING VALUE CORRELATION

The heating value of a petroleum- or coal-derived liquid provides a good indication of its overall quality as well as its ultimate value as a fuel. It is also a necessary parameter in an economic evaluation of any upgrading process. The time and expense required for direct, calorimetric measurement of heating values limit any experimental determination to small data sets. An alternate approach involves the development of an estimation procedure based on elemental properties. Correlations developed for coal and for petroleum liquids have been used in the past to estimate the heating value of coal-derived liquids. In order to relate the heating value and elemental composition of processed coal liquids, we have measured these properties in eighteen coal liquids from the SRC, H-Coal, and Synthoil processes

Figure 6-3

SIMULATED NITROGEN REMOVAL VS SRC CONCENTRATION: MONTEREY SRC / SOLVENT BLENDS; EFFECT OF SPACE VELOCITY

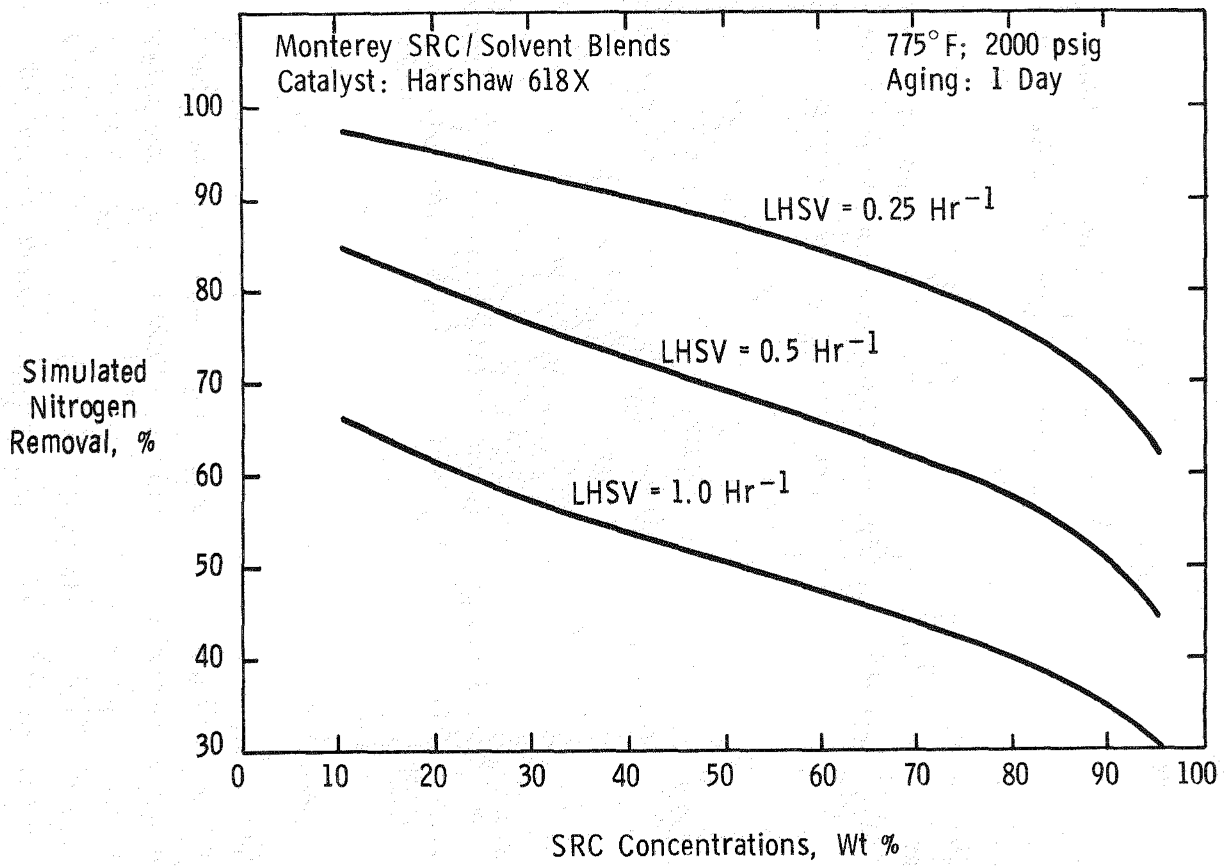


Figure 6-4

SIMULATED NITROGEN REMOVAL VS SRC CONCENTRATION: MONTEREY SRC / SOLVENT BLENDS; EFFECT OF CATALYST TIME ON-STREAM

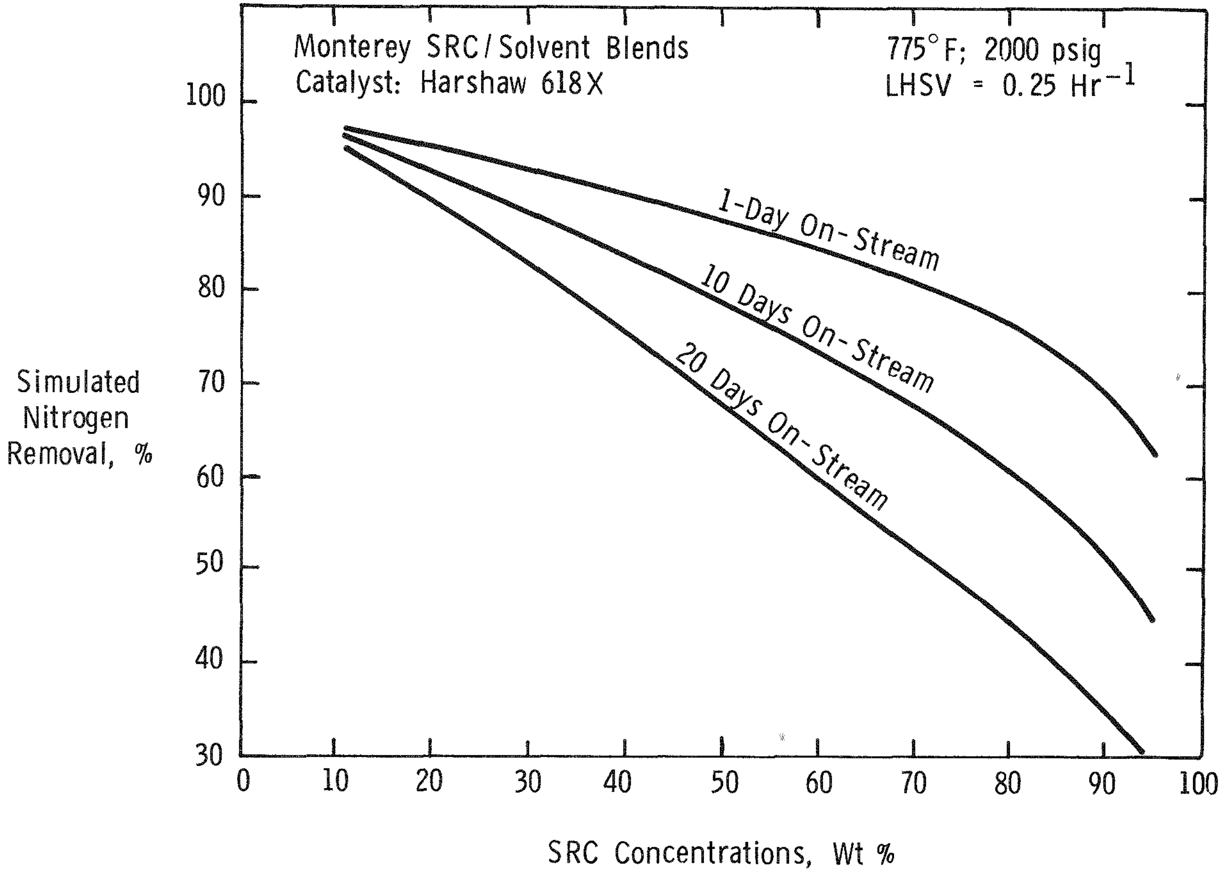


Table 6-1

ELEMENTAL COMPOSITION AND HEATING VALUE OF RAW AND HYDROTREATED COAL LIQUIDS

PROCESS	STREAM	NOMINAL BOILING RANGE °C	SPECIFIC GRAVITY 15.6°C/15.6°C	HEATING** VALUE kJ/kg	ELEMENTAL ANALYSIS, WT %				
					H	C	O	S	N
SRC	Light Organic Liquid	65-205	0.847	42,212	11.33	83.77	4.0	0.60	0.30
SRC	Light Organic Liquid	110-230	0.918	40,068	9.93	84.44	5.0	0.40	0.23
SRC	Light Organic Liquid	50-200	0.811	44,162	12.18	83.56	3.3	0.57	0.39
SRC	Wash Solvent	150-240	0.943	39,627	9.23	84.96	5.7	0.26	0.35
SRC	Recycle Solvent	180-370	1.039	38,790	7.56	87.43	4.1	0.32	0.59
SRC	Recycle Solvent	190-380	1.034	39,358	7.43	87.68	3.9	0.37	0.62
SRC	Recycle Solvent	170-340	1.032	39,137	7.65	87.25	4.1	0.41	0.59
SRC	SRC-Illinois #6 Coal	340-	1.184	37,090	5.72	88.50	3.5	0.57	1.71
SRC	SRC-Illinois #6 Coal	340-	-	36,872	5.81	86.87	4.3	0.96	2.06
SRC	SRC-Wyodak Coal	340-	-	37,086	5.60	88.80	3.8	0.10	1.70
H-Coal	Distillate-Syncrude Mode	190-350	0.968	42,056	9.14	88.87	1.5	0.10	0.39
H-Coal	Fuel Oil-Illinois #6 Coal	260-	-	40,498	7.94	88.77	2.1	0.42	0.77
SRC + HDT*	Recycle Solvent	150-360	0.979	41,233	8.88	88.22	2.4	0.06	0.44
SRC + HDT*	Recycle Solvent	105-340	0.937	43,198	10.32	88.96	0.6	0.01	0.11
SRC + HDT*	Recycle Solvent	100-330	0.914	43,968	10.99	88.78	0.2	0.01	0.02
H-Coal + HDT*	Distillate Syncrude Mode	180-330	0.942	43,326	10.46	89.12	0.3	<0.01	0.12
H-Coal + HDT*	Distillate Syncrude Mode	175-320	0.915	44,299	11.66	88.10	0.2	<0.01	0.04
Synthoil	Liquid Product	205-	1.100	39,288	7.97	88.53	2.1	0.43	0.97

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*Samples hydrotreated under EPRI Program RP 361-2.

**HHV

as well as hydroprocessed distillates from the SRC and H-Coal processes. As shown in Table 6-1, the liquids range in hydrogen content from 5.6 to 12.18 wt %; oxygen content from 0.2 to 5.6 wt %; nitrogen content from 0.02 to 2.06 wt %; and sulfur content from 0.002 to 0.96 wt %. The heating values, determined by ASTM 2382, range from 36,872 to 44,299 kJ/kg. The data from the hydrotreated SRC recycle solvent and H-Coal distillate samples demonstrate the significant heating value increase that results from this processing.

Table 6-2

CONSTANTS AND FIT FOR HEATING VALUE OF COAL LIQUIDS - EQUATION 6-4
HHV = f + g x H

			PERCENT DEVIATION	
CONSTANTS	f	g	AVERAGE	MAXIMUM
*Martel & Angello	35,722	75,967	4.55	8.85
This Work	30,599	113,568	1.63	4.51
API Data Book	Graphical Technique Based** on Gravity and Composition		3.38	5.78

*Corrected to HHV
**Samples with negative API gravity not included.

Two general forms of correlations were used. The first was similar to that employed by Dulong (2) and Gumz (3) for estimating the gross heating value (GHV) of coals based on their elemental composition,

$$HHV = aC + bH + cO + dN + eS. \qquad (6-3)$$

The method of Martel and Angello (4) used in estimating the heating value of petroleum-derived and synthetic jet fuels, is based only on their hydrogen content, i.e.,

$$HHV = f + gH. \qquad (6-4)$$

The constants and deviation of the predicted values for this latter correlation (Equation 6-4) are given in Table 6-2. As could be expected, the constants obtained from these data give a much better fit than the constants determined by

Martel and Angello for petroleum-derived jet fuels. A plot of the heating values predicted by the Martel and Angello correlation versus the measured values shows this correlation to be good for the hydrotreated samples which have hydrogen contents which more closely resemble petroleum-derived jet fuels (Figure 6-5). However, this correlation significantly overestimates the heating value of the untreated coal liquids.

The graphical correlation from the API Data Book was also evaluated (Table 6-2). Although this correlation is somewhat better than that of Martel and Angello, it gave an average deviation of 3.38%. This correlation gave consistently high values for the heating value with the smallest deviation occurring for the samples with the highest heating value.

The heating values for the liquids in Table 6-1 were correlated with their elemental content utilizing correlations requiring an increasing number of elemental analyses. Table 6-3 gives the coefficients determined in this work along with the average and maximum percent deviation obtained from the correlations. The Gumz and Dulong coefficients and deviations are also given.

The contributions to the heating value determined for the carbon are roughly equivalent in all correlations (33,830 to 34,546 kJ/kg). The hydrogen contribution is also similar except for that of Dulong. It should be expected that Dulong's hydrogen coefficient would be high since he assumed the heating value of molecular hydrogen for this value.

Although the Dulong coefficients give a poor estimation of the heating value, they are good for the higher boiling, lower heating value samples (Figure 6-5). These samples more closely resemble raw coal for which this correlation was derived.

The Gumz correlation is significantly better than that of Dulong, and nearly equivalent to the correlation determined in this work, giving only a slightly higher average deviation. The Gumz coefficients for carbon and nitrogen are slightly lower and that for hydrogen is slightly higher than obtained in this work while those for oxygen and sulfur are considerably different.

The best fit to the data in this work is obtained when five major elements in coal liquids are included in Equation 6-3 (Table 6-3). This correlation fits the data well over the entire range of heating values (Figure 6-5). The heating values obtained by this correlation have an average deviation of 0.67% from the measured

Table 6-3

CONSTANTS AND FIT FOR HEATING VALUE*OF COAL LIQUIDS - EQUATION 6-3

ELEMENT	CARBON	HYDROGEN	OXYGEN	NITROGEN	SULFUR	PERCENT DEVIATION	
COEFFICIENT	a	b	c	d	e	AVERAGE	MAXIMUM
Correlation							
Dulong	33,830	144,300	-18,040	-	9,420	2.88	5.18
Gumz	34,070	124,500	-9,848	6,285	19,130	0.71	1.87
This Work							
2 Constant	33,928	124,487	-	-	-	0.79	2.35
3 Constant	34,546	121,672	-10,317	-	-	0.70	1.98
4 Constant	33,953	126,351	-11,381	27,010	-	0.70	2.05
5 Constant	34,309	123,485	-13,719	6,521	32,862	0.67	2.14

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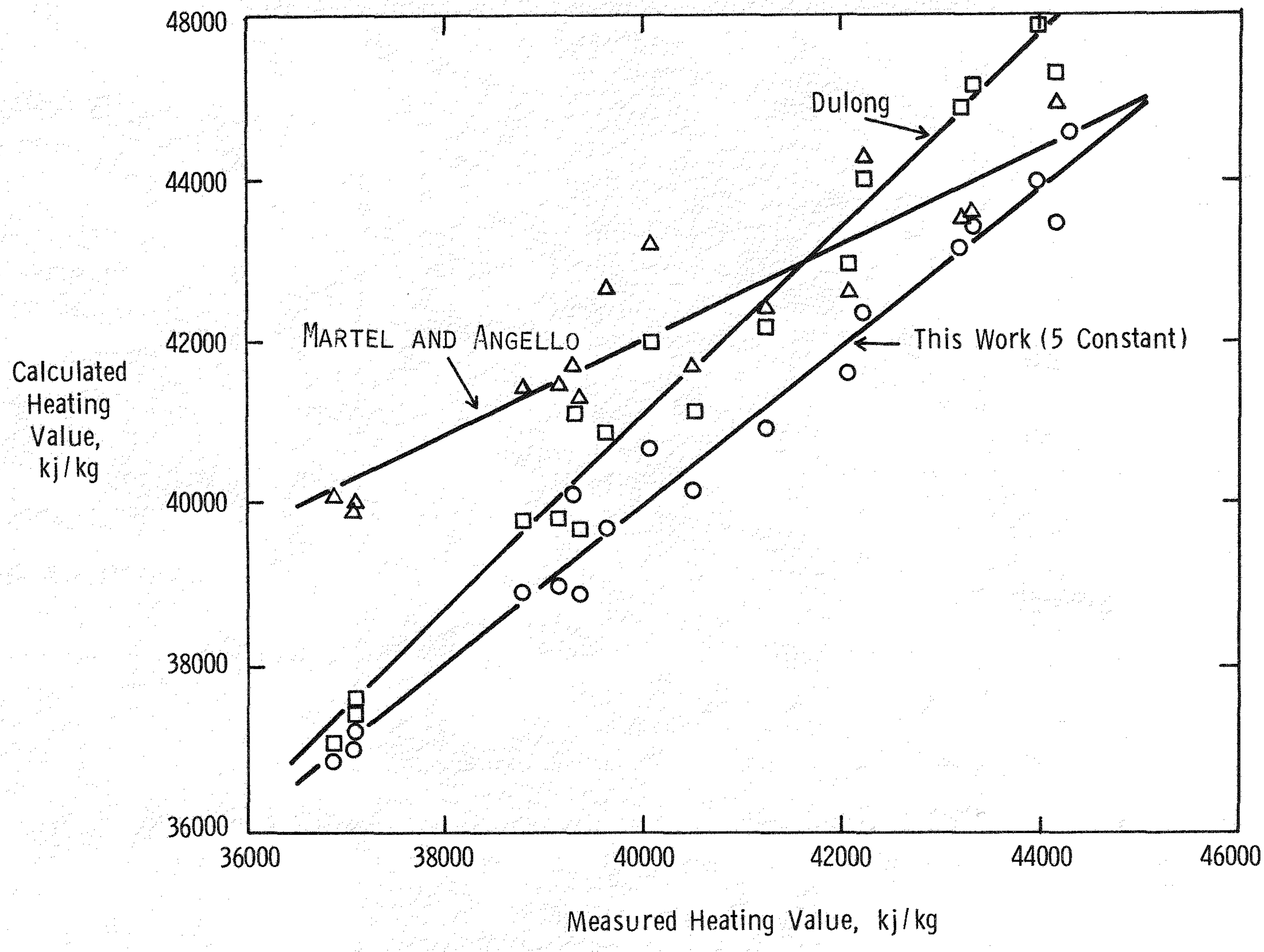
*HHV

Figure 6-5

COMPARISON OF HEATING VALUE EQUATIONS



values with a maximum deviation of 2.14%. A correlation which ignores the contribution of nitrogen and sulfur is almost as good. This is due both to the relatively low heating value of these elements and to their low concentration relative to carbon, hydrogen, and oxygen in coal liquids.

6.3 HYDROPROCESSING OF 50% W. KENTUCKY SCT SRC WITH HARSHAW 618X

The base catalyst used in the catalyst evaluation studies for the hydroprocessing of 50% W. Kentucky SCT SRC blends was Harshaw 618X. To facilitate the comparison of other catalysts with this base catalyst, data from the Harshaw 618X hydroprocessing run were used to develop an appropriate heteroatom (and CCR) removal model. Data from this study are included in Appendix B. An apparent second order rate equation was used:

$$\frac{1}{C} - \frac{1}{C_o} = k\ e^{-E/RT} / \text{LHSV} \qquad (6\text{-}5)$$

where:

C_o, C = heteroatom concentrations in feed and in product, wt %
k = second order reaction rate constant, $(\text{wt }\%)^{-1} \cdot (\text{hr}^{-1})$
E = activation energy, Btu/lb mole
T = reaction temperature, °R
LHSV = liquid hourly space velocity, hr^{-1}

The estimated reaction rate constant and activation energy for sulfur, oxygen, nitrogen, and CCR are:

	k $[(\text{wt }\%)^{-1} \cdot (\text{hr}^{-1})]$	E [(Btu/lb mole)]	C_i Range [wt %]	mean deviation [wt %]
S	2.57×10^8	43,200	0.7-0.25	0.0144
O	1.37×10^9	54,300	0.7-2.4	0.108
N	4.05×10^6	40,100	0.7-1.2	0.036
CCR	4.92×10^4	36,300	9.2-20.8	1.15

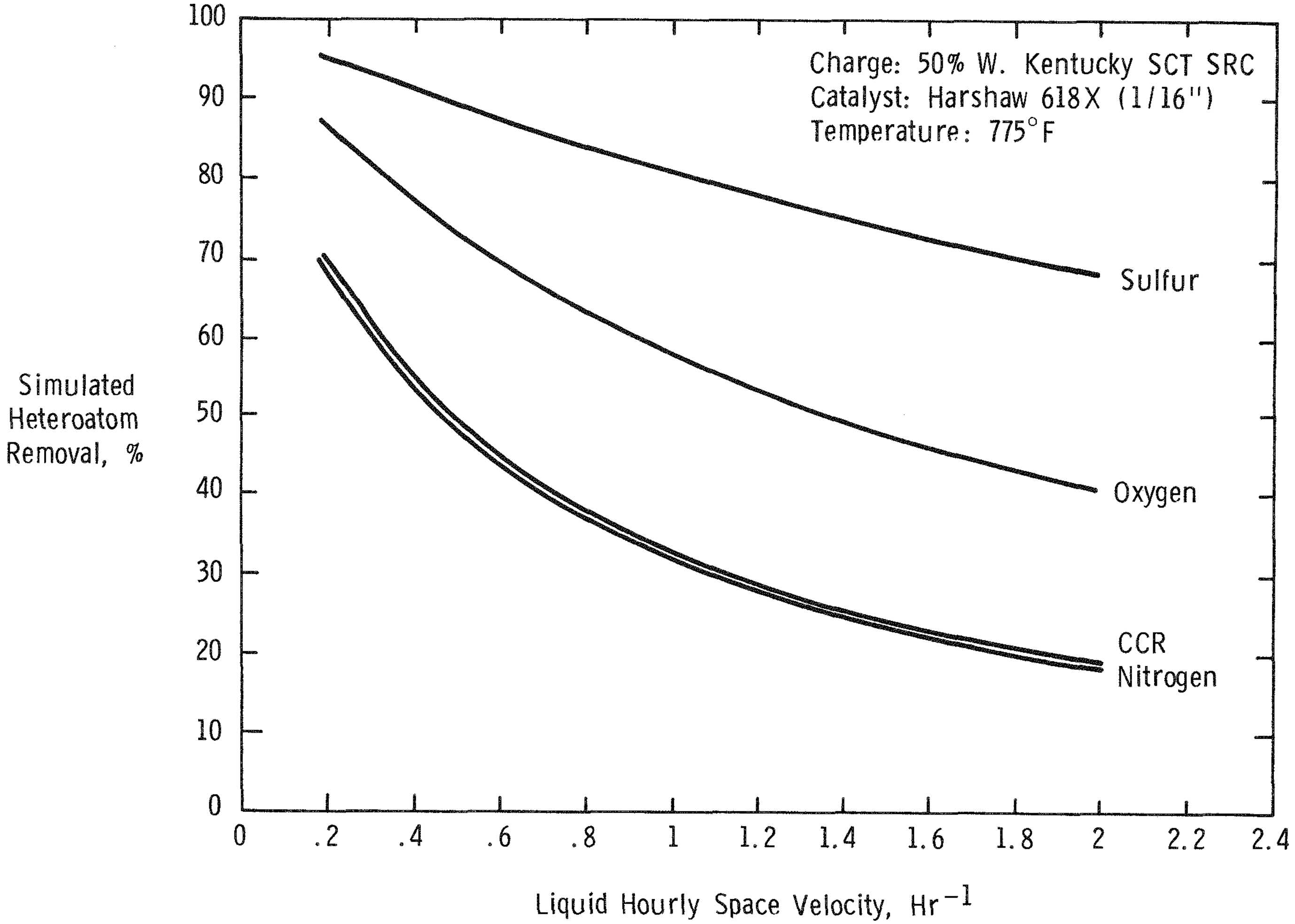
The activation energy for CCR removal is slightly low, 36,300 Btu/lb mole. Since the CCR is directly associated with the SRC, its removal serves as an indicator of SRC conversion. The low activation energy for CCR removal may reflect some diffusion resistance in the processing of the larger SRC molecules.

Using the kinetic constants determined from the data, the CCR and heteroatom removals as a function of liquid phase space velocity can be estimated. The results at 775°F are plotted in Figure 6-6. As shown, a low sulfur product (<0.5 wt %) can be achieved by processing the 50% SCT SRC at 775°F, 1.5 LHSV, and 2000 psig. At this condition the total sulfur removal is 75%.

In comparing each of the catalysts from the evaluation studies with this base catalyst, the actual operating conditions (i.e., pressure, temperature, and space velocity) were used with these kinetics to estimate the performance of the Harshaw 618X. Consequently, a direct comparison was made of the catalysts at equivalent conditions.

Figure 6-6

SIMULATION OF HETEROATOM REMOVAL: 50% W. KENTUCKY SCT SRC



Section 7

KERR-McGEE CRITICAL SOLVENT DEASHED SRC

The Kerr-McGee Critical Solvent Deashing process can be used to upgrade solvent refined coal by both fractionation and ash removal. We have compared a K-M CSD SRC with a conventionally filtered SRC in order to identify differences in composition and processability.

7.1 PROPERTIES OF KERR-McGEE CSD SRC

The elemental and trace metal analyses of the two Indiana V regular SRC samples are listed in Table 7-1. Both SRC's were deashed to less than 0.1 wt % ash content. K-M CSD SRC has a slightly higher hydrogen content and a slightly lower sulfur content than the conventionally deashed SRC. However, the Conradson Carbon Residue (CCR) of K-M CSD SRC was significantly lower (38 wt % vs. 55 wt %). This difference was consistent with the GEC analysis, shown in Table 7-2. Here, the K-M CSD SRC showed less polar and non-eluted asphaltenes. In addition, K-M CSD SRC contained fewer trace metals than **filtered SRC.** The better quality of K-M CSD SRC was expected to give a higher desulfurization (hydroprocessing) reactivity, discussed in the following sections. In contrast to the GEC analyses, the two SRC's showed a similar molecular size distribution (cf. Figure 7-1). The molecular size distributions were determined by gel permeation chromatography (GPC).

The viscosity of SRC's at 350-400°F normally is the major mechanical limitation to processing undiluted material. Viscosity measurements are shown in Table 7-3 for 70% and 90% K-M CSD SRC blends with 470°F$^+$ recycle solvent. The Kerr-McGee SRC blends have a slightly higher viscosity than a previously analyzed Monterey SRC blend (Figure 7-2). However, this difference could easily be a result of the difference in solvents used in making these blends: 400°F$^+$ for the Monterey and 475°F$^+$ for the "CSD" sample. To run smoothly in our unit, a blend should have a kinematic viscosity below 100 cs at 350-400°F. Therefore, the Kerr-McGee SRC blends were run at 70%.

Table 7-1

ELEMENTAL AND TRACE METALS COMPOSITION OF TWO INDIANA V REGULAR SRC'S

Elemental Analyses	K-M CSD SRC (78D3692)*	Filtered SRC (78D1323)
H, Wt %	6.08	5.8
S, "	0.70	0.84
N, "	1.89	2.08
O, "	3.51	3.3
Trace Metals		
Ca, ppm	25	20
Fe, "	105	NA
Pb, "	1.2	2.4
Ni, "	2.4	NA
K, "	0.01	0.01
Na, "	70	90
Ti, "	90	220
V, "	4.2	NA
Other		
CCR, Wt %	37.89	55.13
Ash, Wt %	0.06	0.09
Specific Gravity, 77°F/77°F	1.122	NA

NA = Not Available.

* Wilsonville I.D. SN-39167

Table 7-2

GEC ANALYSES OF TWO INDIANA V REGULAR SRC'S

Cut	Name	K-M CSD SRC (78D3692)*	Filtered SRC (78D1323)
1	Saturates	0.4	0.04
2-4	Aromatic Oils	10.3	4.44
5-7	Resins/Asphaltenes	41.1	33.14
8-12	Polar Asphaltenes	44.7	52.17
13	Non-Eluted Plus Loss	3.5	9.71
	Total	100.0	100.00

* Wilsonville I.D. SN-39167

Table 7-3

VISCOSITY OF KERR-McGEE CSD SRC/
RECYCLE SOLVENT BLENDS

	KINEMATIC VISCOSITY (cSt) AT	
Temperature	70% SRC	90% SRC
300°F	214.9	-
350°F	46.24	1106
400°F	20.07	230.6

Charge:

Kerr-McGee SRC:	78D-3216. (Wilsonville I.D. SN-38533)
Recycle Solvent:	475°F$^+$ recycle solvent 78D-839. (cf. Section 3.1)

Figure 7-1

SIZE DISTRIBUTION OF TWO INDIANA V REGULAR SRC'S

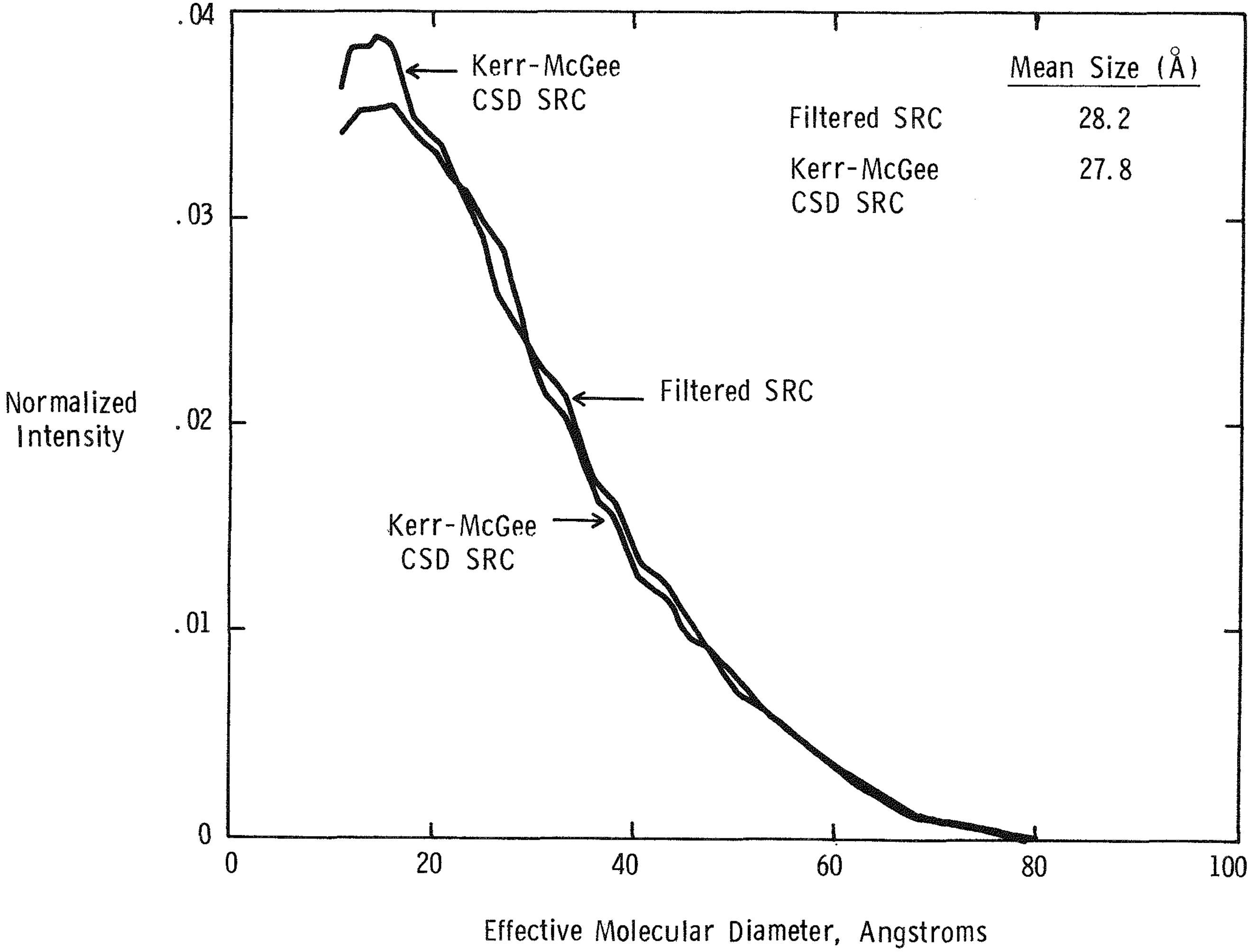
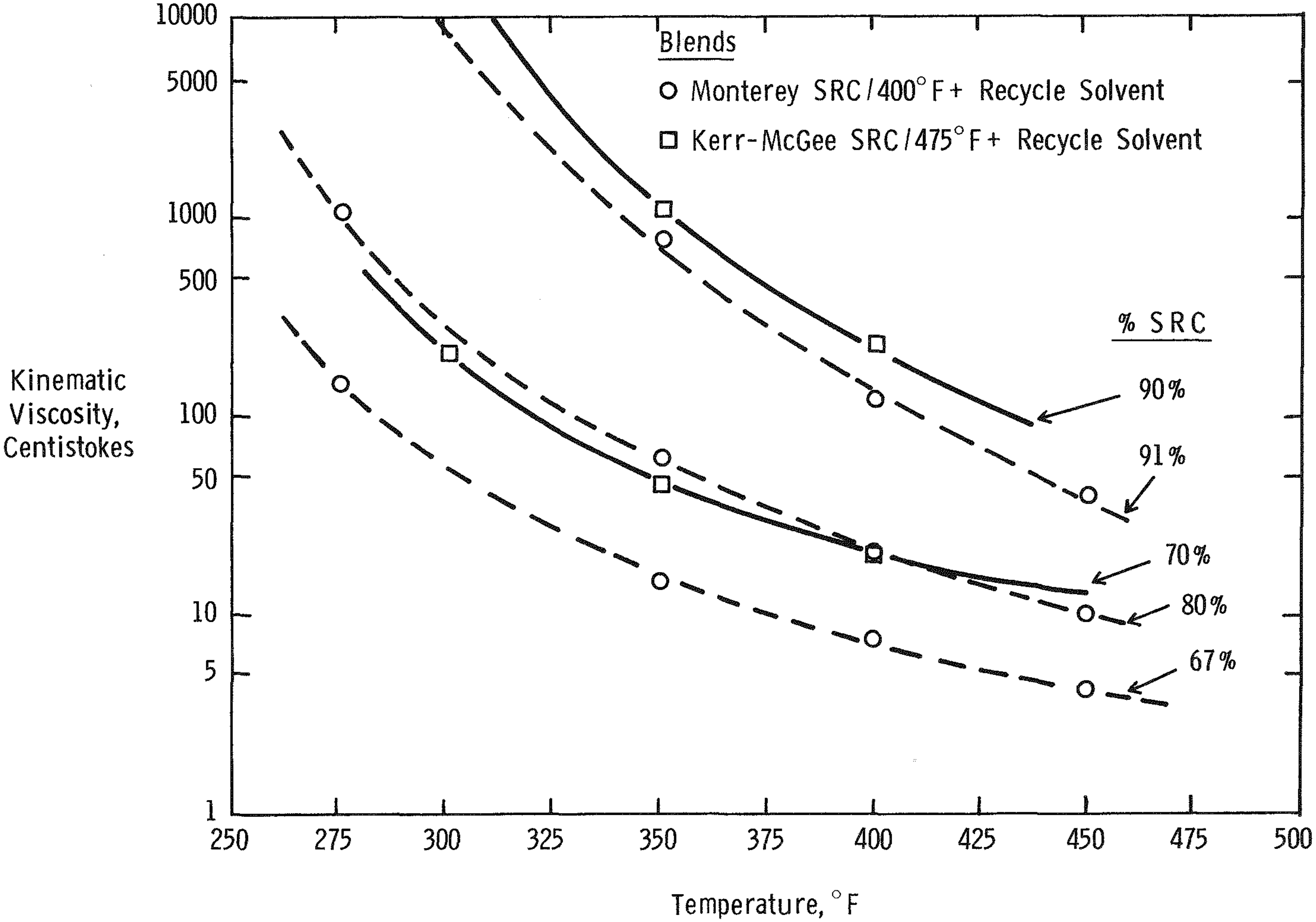


Figure 7-2

VISCOSITY OF KERR-McGEE CSD SRC BLENDS



7.2 HYDROPROCESSING OF KERR-McGEE CSD SRC

Both K-M CSD SRC and conventionally filtered Indiana V SRC were tested in our pilot unit using 70% blends over a commercial NiMo/Al_2O_3 catalyst, Harshaw 618X. The solvent used for making charge stock blends was process recycle solvent (470-720°F boiling range). The catalyst was presulfided and stabilized at 725°F with process recycle solvent prior to charging the reactor with SRC. Runs were carried out at 2000 psig and at various temperatures (725-800°F) and liquid hourly space velocities (0.4 - 2.0). These start-up operations and process conditions were similar to those used in the catalyst evaluations for upgrading short contact time SRC.

Detailed material balances for K-M CSD SRC are given in Tables 7-4 and 7-5. Similar material balances for conventionally deashed Indiana V regular SRC have been previously reported. The heteroatom removal and CCR reduction from these two SRC's are compared in Figures 7-3, 7-4, 7-5, and 7-6 for desulfurization, denitrogenation, deoxygenation, and CCR reduction, respectively. Here, the heteroatom (and CCR) removals are plotted versus reactor temperature at liquid hourly space velocities of 0.5 and 1.0. Consequently, SRC reactivities can be compared on the basis of temperature requirements for achieving specific liquid product heteroatom contents. The results show that, compared to conventionally deashed Indiana V SRC, K-M CSD SRC is:

- 40°F more reactive for sulfur removal,
- 50-75°F more reactive for nitrogen removal,
- 30-40°F more reactive for oxygen removal,
- 10-20°F more reactive for CCR reduction.

The high reactivity of the Kerr-McGee SRC can also be related in terms of space velocities required to reach a given heteroatom (or CCR) level. For desulfurization, the K-M CSD SRC can be hydroprocessed at 3-4 times the liquid hourly space velocity required for processing conventionally filtered SRC. In an integrated SRC/catalytic upgrading process, similar space velocity advantages could be realized for CCR reduction. However, much of this CCR improvement is achieved by the Kerr-McGee process itself. These space velocity advantages illustrate the impact that Critical Solvent Deashing can have on the economics of making low sulfur fuels and other higher quality products from SRC.

Although K-M CSD SRC was significantly more reactive for heteroatom removal, the hydrogen consumptions were approximately equivalent. The total hydrogen consumption

TABLE 7-4

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 70/30 KERR-MCGEE CSD SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2864 UNIT: 146

		MB-959	MB-960	MB-961	MB-962	MB-963	MB-964
OPERATING CONDITIONS							
PRESSURE, PSIG	-	2000	1960	1975	1975	1975	1975
AVG REACTOR TEMP., °F	-	724	727	780	773	778	809
LHSV VFF/HR/VCAT	-	0.43	0.86	0.91	0.43	1.71	1.67
LIQUID PRODUCT PROPERTIES							
GRAVITY, API	-9.9	-3.1	-5.2	0.8	3.4	-4.4	-2.2
HYDROGEN, WT PCT	6.56	8.08	7.59	6.51	8.71	7.29	7.54
SULFUR, WT PCT	0.660	0.132	0.210	0.089	0.063	0.179	0.127
NITROGEN, WT PCT	1.81	1.14	1.37	0.98	0.76	1.32	1.29
OXYGEN, WT PCT	3.60	1.54	2.61	5.26	0.75	2.40	1.50
CCR, WT PCT	30.36	23.46	26.12	20.07	15.46	23.92	21.09
K.V.(100 C)	-	227.30	590.60	-	13.39	158.60	43.44
HETEROATOM REMOVAL							
DESULFURIZATION, PCT WT	-	80.6	68.6	87.0	90.9	73.6	81.5
DENITROGENATION, PCT WT	-	38.8	25.3	47.8	60.0	28.9	31.7
DEOXYGENATION, PCT WT	-	58.5	28.5	86.3	80.2	34.2	60.0
CCR REMOVAL, PCT WT	-	25.0	15.1	54.0	51.5	23.2	33.4
HYDROGEN CONSUMPTION, SCF/B							
TOTAL H2 CONSUMPTION	-	1470	969	2936	2168	786	1112
H2 CONSUMED BY C1-C5	-	137	59	310	347	137	268
H2 CONSUMED BY C6+	-	987	713	2154	1332	420	514
H2 CONSUMED BY S	-	26	22	28	29	23	26
H2 CONSUMED BY N	-	116	76	143	180	87	95
H2 CONSUMED BY O	-	204	99	301	279	119	209

TABLE 7-5

FIXED BED HYDROPROCESSING OF COAL LIQUIDS
YIELDS AND DISTILLATION

CHARGE: 70/30 KERR-MCGEE CSD SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2864 UNIT: 146

	MB-959	MB-960	MB-961	MB-962	MB-963	MB-964
MATERIAL BALANCE, PCT WT	109.1	101.8	101.9	98.0	99.4	91.0
OPERATING CONDITIONS						
PRESSURE, PSIG	2000	1960	1975	1975	1975	1975
AVG REACTOR TEMP., °F	724	727	780	773	778	809
LHSV VFF/HR/VCAT	0.43	0.86	0.91	0.43	1.71	1.67
H_2 CHG, SCFB FF NLB	8706	6780	9944	8479	6923	6835
CATALYST AGE, DAYS	1.9	2.3	2.8	3.6	4.0	4.3
YIELDS (BASED ON CHARGE)						
TOT C_1-C_3, WT PCT	0.45	0.27	1.02	1.49	0.52	1.09
TOT C_4, WT PCT	0.46	0.11	0.75	0.74	0.39	0.63
TOT C_5, WT PCT	0.10	0.01	0.46	0.13	0.08	0.12
C_6+ PRODUCT, WT PCT	97.11	98.68	96.44	95.25	97.50	95.90
H_2S, WT PCT	0.57	0.48	0.61	0.64	0.52	0.57
NH_3, WT PCT	0.85	0.56	1.05	1.32	0.64	0.70
H_2O, WT PCT	2.37	1.15	3.50	3.25	1.39	2.43
DISTILLATION (D2887), °F						
IBP	-	-	269	180	-	280
5 PCT VOL	-	-	615	402	-	427
10 " "	-	-	788	471	-	470
30 " "	-	-	912	592	-	582
50 " "	-	-	996	786	-	782
70 " "	-	-	-	-	-	-
90 " "	-	-	-	-	-	-
95 " "	-	-	-	-	-	-
EP	-	-	-	-	-	-

Figure 7-3

DESULFURIZATION REACTIVITIES OF TWO INDIANA REGULAR SRC'S

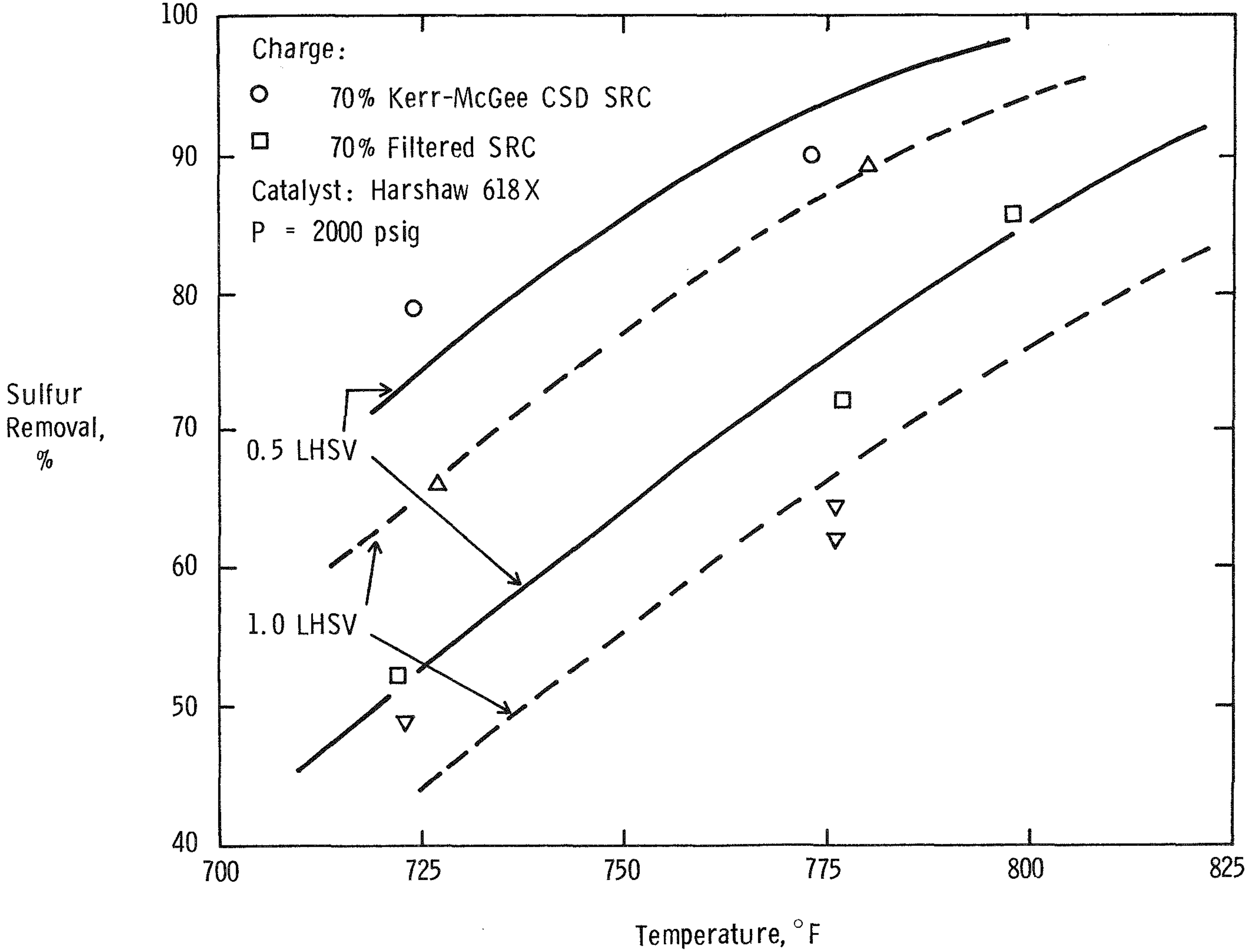


Figure 7-4

DENITROGENATION REACTIVITIES OF TWO INDIANA V REGULAR SRC'S

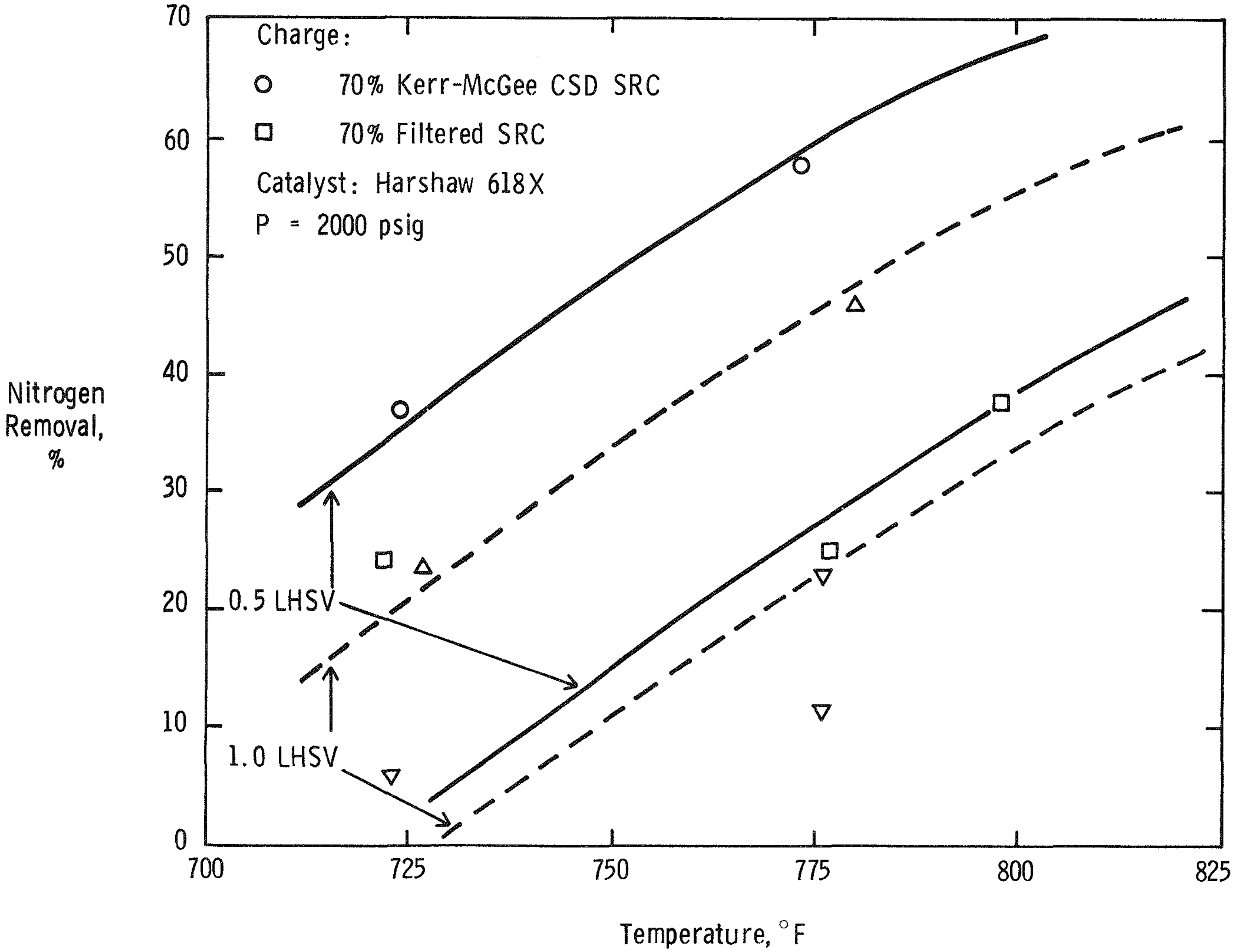


Figure 7-5

DEOXYGENATION REACTIVITIES OF TWO INDIANA V REGULAR SRC'S

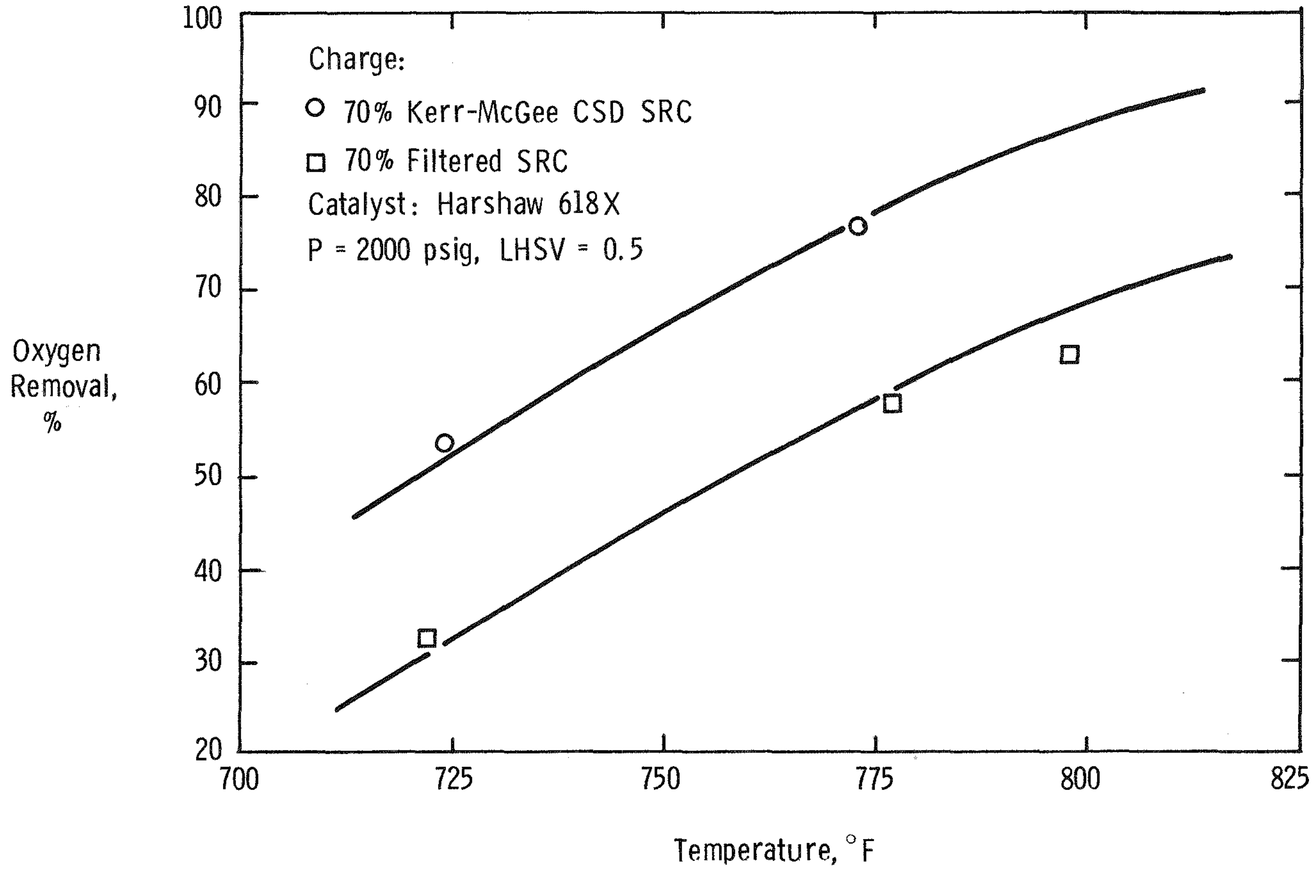
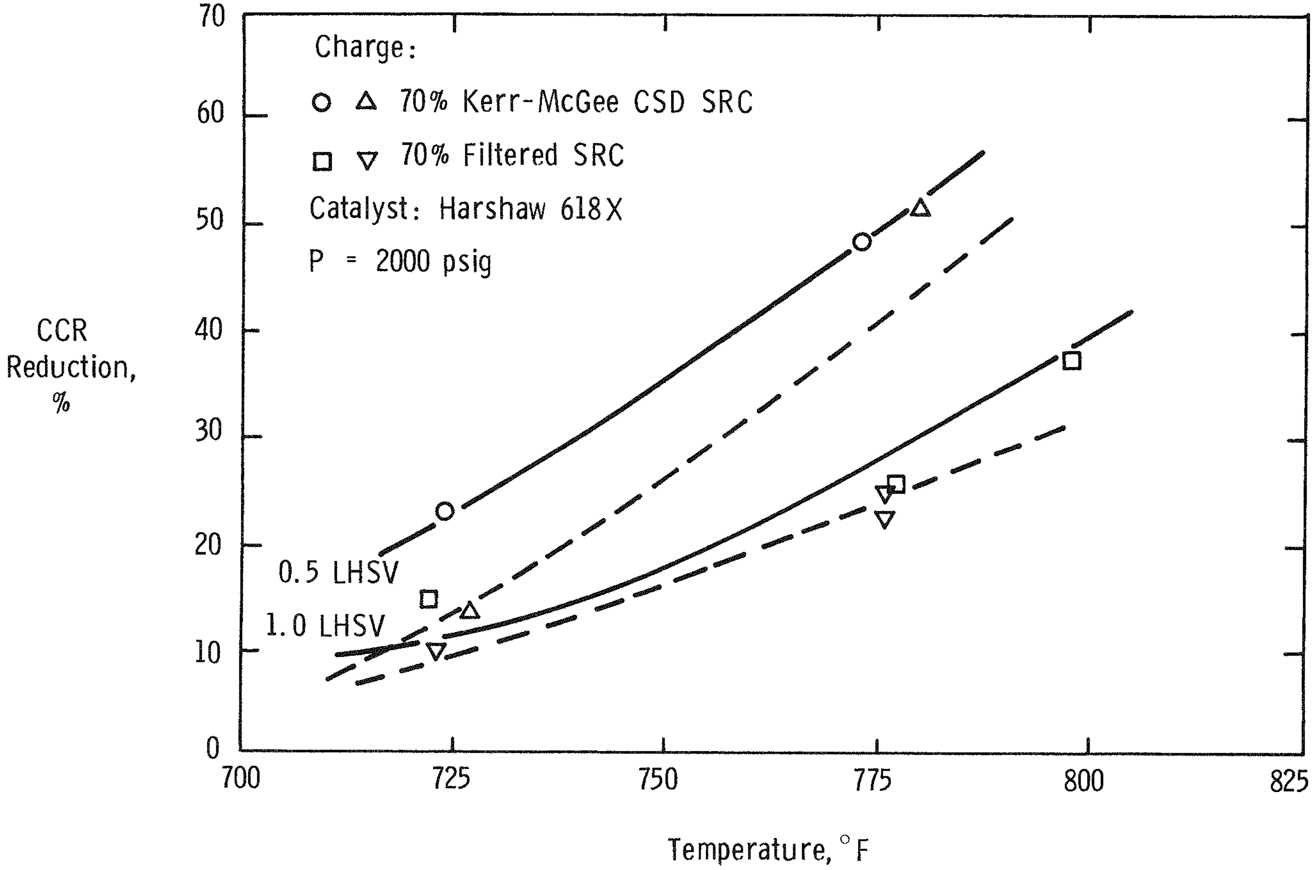


Figure 7-6

CCR REDUCTION REACTIVITIES OF TWO INDIANA V REGULAR SRC'S



versus sulfur content of the products is shown in Figure 7-7. A breakdown of the hydrogen utilization for C_1-C_5, C_6^+, and heteroatom removal is shown in Figures 7-8, 7-9, and 7-10, respectively. As shown in Figure 7-8, K-M CSD SRC consumed 50% less hydrogen for the C_1-C_5 gases than did the filtered Indiana V SRC. This reduced gas make is a result of the lower temperatures required to desulfurize the Kerr-McGee material. Since the hydrogen consumed in the gas formation constitutes only 10-20% of the total hydrogen consumption, these differences in C_1-C_5 hydrogen utilization are within the experimental error in total hydrogen consumption.

Three of the hydrotreated Kerr-McGee SRC blends were cut at 650°F; these fractions were analyzed for elemental composition, shown in Table 7-6. Two of the three samples were also separated by GEC; these materials show the normal progression to less polar compounds and eventually saturates and $650°F^-$ material with increasing hydroprocessing severity. The heteroatom (S, N, O) contents of the $650°F^-$ fractions are normally 10-50% of the corresponding contents in the $650°F^+$.

TABLE 7-6

GEC AND ELEMENTAL ANALYSES OF HYDROTREATED KERR-MCGEE CSD SRC
CHARGE: 70/30 KERR-MCGEE CSD SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2864   UNIT: 146

	CHARGE	*MB-960*	*MB-962*	*MB-964*
OPERATING CONDITIONS				
TEMPERATURE, °F	-	727	773	809
LHSV, VFF/HR/VCAT	-	0.86	0.43	1.67
PRESSURE, PSIG	-	1960	1975	1975
H2 CONSUMPTIONS, SCF/B	-	969	2168	1112
TLP PROPERTIES				
HYDROGEN, WT PCT	6.56	7.59	8.71	7.54
OXYGEN, WT PCT	3.60	2.61	0.75	1.50
NITROGEN, WT PCT	1.81	1.37	0.76	1.29
SULFUR, WT PCT	0.660	0.210	0.063	0.127
TLP YIELDS, WT PCT				
650°F-	21.8	27.6	38.1	42.8
SATURATES (CUT 1)	-	0.9	1.8	-
AROM. OILS (CUT 2-4)	-	15.4	27.9	-
RESINS/ASPH. (CUT 5-7)	-	32.6	25.1	-
POLAR ASPH. (CUT 8-13)	-	23.5	7.1	-
TOTAL	-	100.0	100.0	-
ELEMENTAL ANALYSES				
IBP-650°F				
HYDROGEN, WT PCT	8.29	9.49	10.20	9.71
OXYGEN, WT PCT	2.63	1.73	0.77	1.44
NITROGEN, WT PCT	0.602	0.528	0.224	0.552
SULFUR, WT PCT	0.370	0.049	0.020	0.006
650°F+ *BTMS*				
HYDROGEN, WT PCT	6.07	7.14	6.24	6.88
OXYGEN, WT PCT	4.82	3.04	1.65	2.46
NITROGEN, WT PCT	2.07	1.75	0.99	1.80
SULFUR, WT PCT	0.60	0.37	0.03	0.18

Figure 7-7

COMPARISON OF TWO INDIANA V REGULAR SRC'S: TOTAL HYDROGEN CONSUMPTION AS A FUNCTION OF TLP SULFUR CONTENT

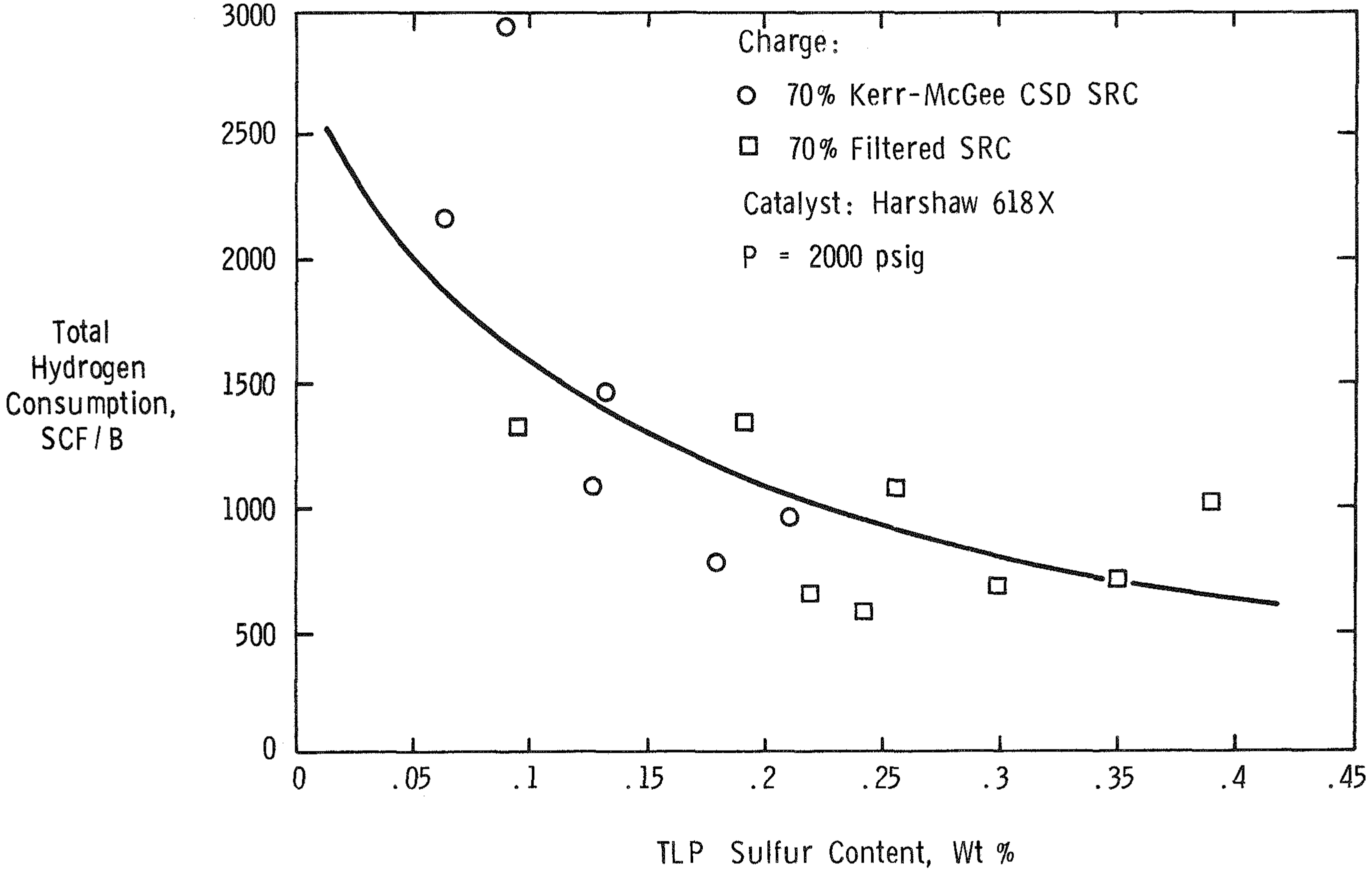


Figure 7-8

COMPARISON OF TWO INDIANA V REGULAR SRC'S:
HYDROGEN UTILIZATION IN C_1 - C_5
PRODUCTION AS A FUNCTION OF TLP SULFUR CONTENT

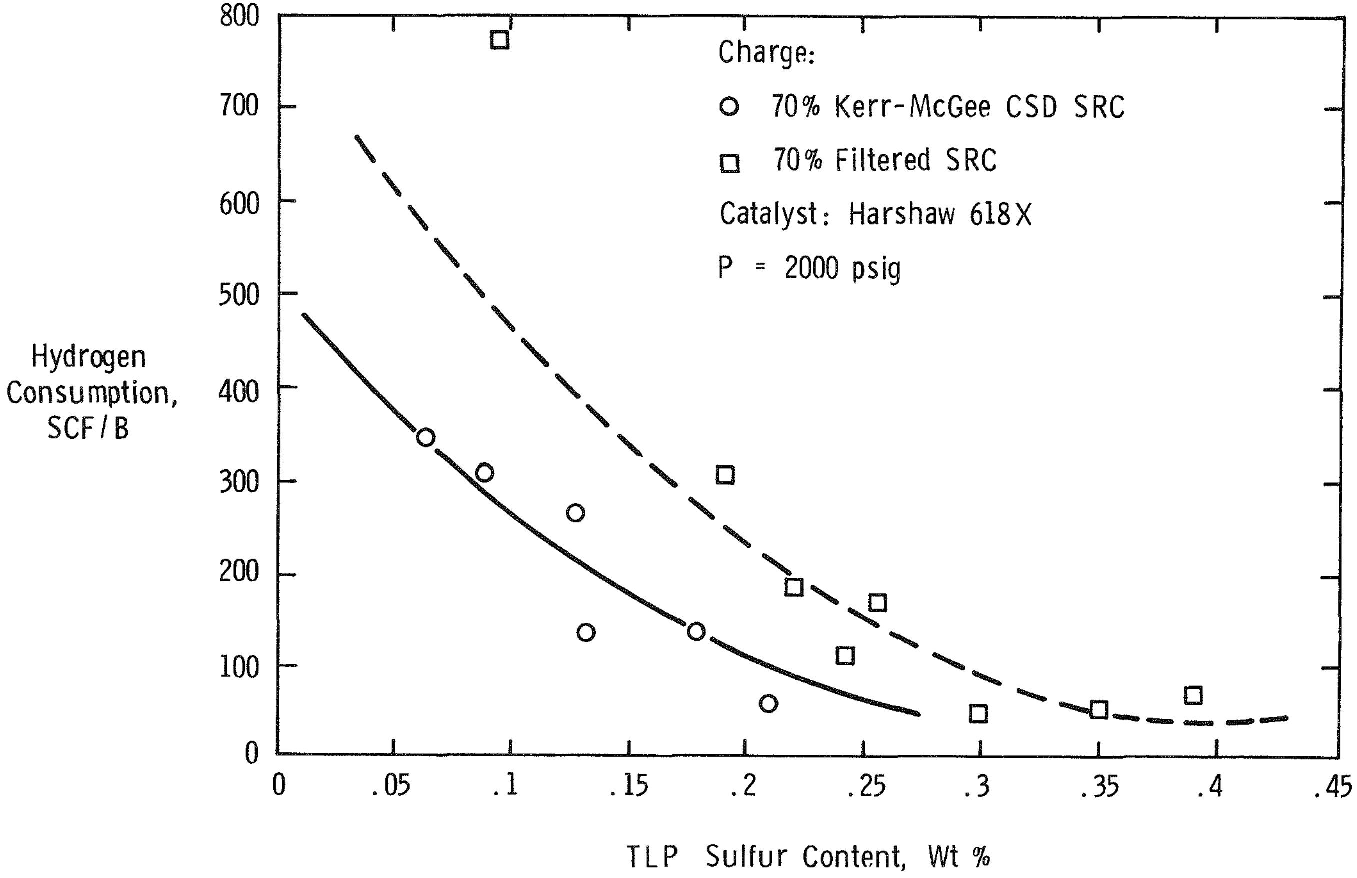


Figure 7-9

COMPARISON OF TWO INDIANA V REGULAR SRC'S: HYDROGEN UTILIZATION BY C_6^+ AS A FUNCTION OF TLP SULFUR CONTENT

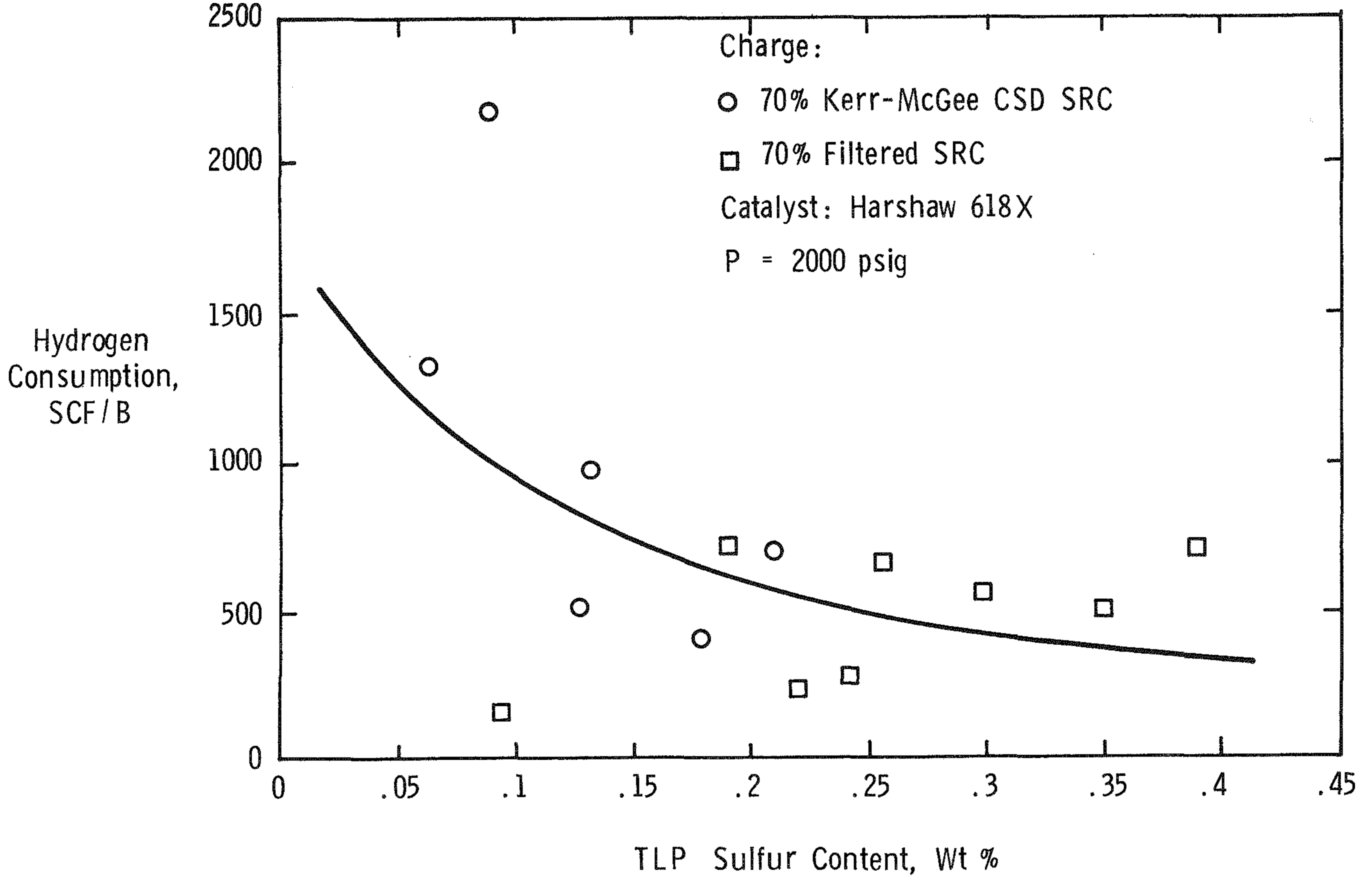
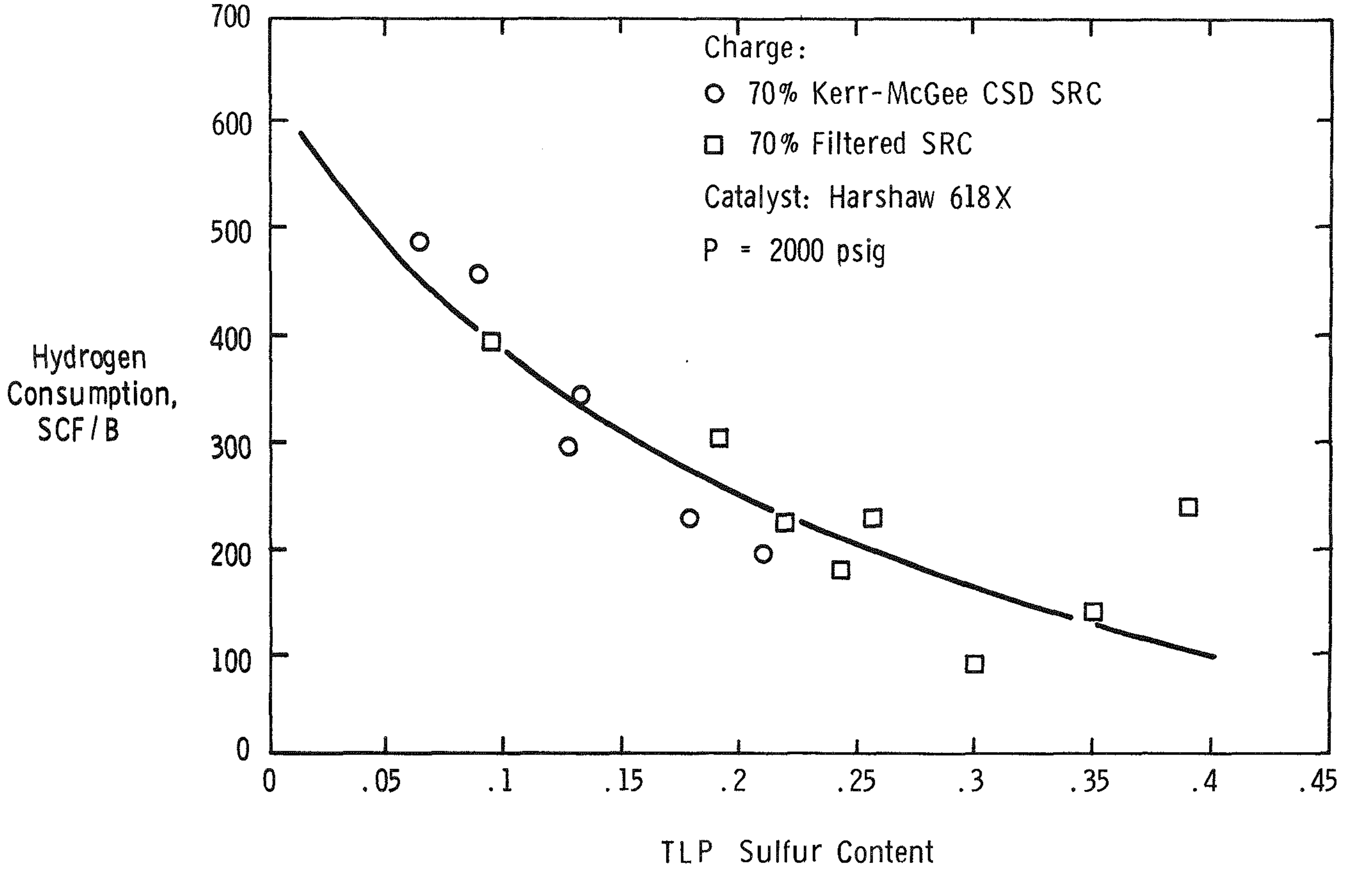


Figure 7-10

COMPARISON OF TWO INDIANA V REGULAR SRC'S;
HYDROGEN UTILIZATION FOR HETEROATOM
REMOVAL AS A FUNCTION OF TLP SULFUR CONTENT



Section 8

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Appendix A

MATERIAL BALANCES FOR HYDROPROCESSING STUDIES

Table	
A-1	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Harshaw 618X
A-2	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Harshaw 618X
A-3	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Harshaw HT-500
A-4	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: HT-500
A-5	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Mobil HCL-1
A-6	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Mobil HCL-1
A-7	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Amocat 1B
A-8	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Amocat 1B
A-9	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Cyanamid HDN-1197
A-10	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Cyanamid HDN-1197
A-11	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Solvent; Catalyst: Mobil HCL-2
A-12	Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Mobil HCL-2

Appendix A (Continued)

A-13 Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Cyanamid HDS-1443

A-14 Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Cyanamid HDS-1443

A-15 Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Mobil HCL-3

A-16 Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Mobil HCL-3

A-17 Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; TLP Properties and Hydrogen Consumption: Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Amocat 1A

A-18 Fixed Bed Hydroprocessing of Coal Liquids - Catalyst Evaluation; Yields and Distillation; Charge: 50/50 W. Kentucky SCT SRC / Recycle Solvent; Catalyst: Amocat 1A

A-19 Fixed Bed Hydroprocessing of Indiana V SRC - Low Sulfur Sample; TLP Properties and Hydrogen Consumption; Charge: 70/30 Indiana V SRC / Recycle Solvent; Catalyst: Harshaw 618X

A-20 Fixed Bed Hydroprocessing of Indiana V SRC - Low Sulfur Sample; Yields and Distillation; Charge: 70/30 Indiana V SRC / Recycle Solvent; Catalyst: Harshaw 618X

A-21 Fixed Bed Hydroprocessing of Indiana V SRC - High Sulfur Sample; TLP Properties and Hydrogen Consumption; Charge: 70/30 Indiana V SRC / Recycle Solvent Catalyst: Harshaw 618X

A-22 Fixed Bed Hydroprocessing of Indiana V SRC - High Sulfur Sample; Yields and Distillation; Charge: 70/30 Indiana V SRC / Recycle Solvent; Catalyst: Harshaw 618X

TABLE A-1

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2723 UNIT: 146

	CHARGE	MB-902	MB-903	MB-904	MB-905	MB-906	MB-907
OPERATING CONDITIONS							
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	726	725	781	768	778	802
LHSV VFF/HR/VCAT	-	0.59	0.68	0.73	1.86	0.36	0.65
LIQUID PRODUCT PROPERTIES							
GRAVITY, API	-8.7	8.6	-0.6	4.2	-2.4	4.2	5.0
HYDROGEN, WT PCT	6.41	8.61	8.04	8.50	7.45	8.88	8.65
SULFUR, WT PCT	0.660	0.110	0.191	0.087	0.246	0.077	0.076
NITROGEN, WT PCT	1.42	0.70	1.04	0.82	1.14	0.87	0.74
OXYGEN, WT PCT	4.30	2.30	2.40	1.30	3.21	1.30	1.00
CCR, WT PCT	24.58	7.95	17.61	13.38	20.78	12.36	13.43
K.V.(100 C)	-	-	112.90	-	24.32	17.99	6.71
HETEROATOM REMOVAL							
DESULFURIZATION, PCT WT	-	83.8	71.7	87.9	63.7	88.8	89.7
DENITROGENATION, PCT WT	-	52.1	28.3	47.2	21.9	41.4	53.2
DEOXYGENATION, PCT WT	-	48.0	45.3	72.3	27.3	71.1	79.1
CCR REMOVAL, PCT WT	-	68.6	29.8	50.2	17.7	51.9	50.9
HYDROGEN CONSUMPTION, SCF/B							
TOTAL H2 CONSUMPTION	-	2035	1504	2285	1069	2355	2634
H2 CONSUMED BY C1-C5	-	143	117	566	194	284	938
H2 CONSUMED BY C6+	-	1547	1112	1284	691	1654	1218
H2 CONSUMED BY S	-	26	23	28	20	28	28
H2 CONSUMED BY N	-	121	66	110	51	96	124
H2 CONSUMED BY O	-	198	187	298	113	293	326

TABLE A-1 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 *W. KENTUCKY SCT SRC / RECYCLE SOLVENT*
CATALYST: HARSHAW 618X
PROGRAM: 2723 *UNIT:* 146

	CHARGE	*MB-908*	*MB-909*	*MB-910*	*MB-911*
OPERATING CONDITIONS					
PRESSURE, PSIG	-	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	803	799	732	791
LHSV VFF/HR/VCAT	-	1.43	0.43	0.58	0.50
LIQUID PRODUCT PROPERTIES					
GRAVITY, API	-9.7	3.2	7.4	9.5	9.6
HYDROGEN, WT PCT	6.46	8.13	8.78	8.46	9.14
SULFUR, WT PCT	0.840	0.129	0.061	0.132	0.069
NITROGEN, WT PCT	1.42	0.92	0.64	0.75	0.66
OXYGEN, WT PCT	4.10	1.70	0.70	1.90	3.10
CCR, WT PCT	26.82	26.82	26.82	26.82	26.82
K.V.(100 C)	-	9.04	3.45	-	-
HETEROATOM REMOVAL					
DESULFURIZATION, PCT WT	-	85.4	93.3	84.9	92.1
DENITROGENATION, PCT WT	-	38.4	58.6	49.1	55.5
DEOXYGENATION, PCT WT	-	60.6	84.3	55.4	27.7
CCR REMOVAL, PCT WT	-	52.2	68.5	61.7	56.2
HYDROGEN CONSUMPTION, SCF/B					
TOTAL H2 CONSUMPTION	-	1740	2538	1938	2691
H2 CONSUMED BY C1-C5	-	343	684	232	563
H2 CONSUMED BY C6+	-	1033	1346	1337	1851
H2 CONSUMED BY S	-	35	38	34	37
H2 CONSUMED BY N	-	90	138	115	130
H2 CONSUMED BY O	-	240	334	219	110

TABLE A-2

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2723 UNIT: 146

	MB-902	MB-903	MB-904	MB-905	MB-906	MB-907	MB-908	MB-909	MB-910	MB-911
MATERIAL BALANCE, PCT WT	108.3	98.5	107.3	102.8	101.4	106.6	101.1	102.7	94.8	83.0
OPERATING CONDITIONS										
PRESSURE, PSIG	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	726	725	781	768	778	802	803	799	732	791
LHSV VFF/HR/VCAT	0.59	0.68	0.73	1.86	0.36	0.65	1.43	0.43	0.58	0.50
H2 CHG, SCFB FF NLB	6760	8112	6950	7262	9109	6070	7963	5810	7627	5388
CATALYST AGE, DAYS	1.7	3.1	3.6	4.0	4.6	5.4	5.9	6.8	7.5	9.1
YIELDS (BASED ON CHARGE)										
TOT C1-C3, WT PCT	0.44	0.45	1.30	0.58	1.05	3.46	1.29	2.75	0.83	2.27
TOT C4, WT PCT	0.61	0.35	1.73	0.62	0.77	1.94	0.83	1.16	0.56	0.95
TOT C5, WT PCT	0.08	0.06	1.28	0.25	0.21	1.13	0.27	0.75	0.26	0.62
C6+ PRODUCT, WT PCT	97.74	97.93	93.76	97.81	96.29	91.55	95.65	92.91	96.71	96.61
H2S, WT PCT	0.59	0.50	0.62	0.45	0.62	0.63	0.76	0.83	0.76	0.82
NH3, WT PCT	0.90	0.49	0.81	0.38	0.71	0.92	0.66	1.01	0.85	0.96
H2O, WT PCT	2.32	2.20	3.50	1.32	3.44	3.83	2.80	3.89	2.56	1.28
DISTILLATION (D2887),°F										
IBP	337	404	351	-	418	324	361	535	366	209
5 PCT VOL	431	465	454	-	465	423	446	578	486	360
10 '' ''	464	488	489	-	492	464	480	588	503	428
30 '' ''	528	579	569	-	570	554	564	621	567	529
50 '' ''	584	675	643	-	644	640	643	652	633	591
70 '' ''	688	-	818	-	834	827	837	726	776	676
90 '' ''	-	-	-	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-	-

TABLE A-3

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: HARSHAW HT-500
PROGRAM: 2755 UNIT: 146

	CHARGE	MB-912	MB-913	MB-914	MB-915
OPERATING CONDITIONS					
PRESSURE, PSIG	-	2000	2000	2000	2350
AVG REACTOR TEMP., °F	-	707	724	779	776
LHSV VFF/HR/VCAT	-	0.43	0.98	0.97	0.61
LIQUID PRODUCT PROPERTIES					
GRAVITY, API	¯8.6	1.5	¯0.4	¯0.1	¯0.5
HYDROGEN, WT PCT	6.78	8.25	7.71	7.97	8.34
SULFUR, WT PCT	0.890	0.170	0.310	0.190	0.130
NITROGEN, WT PCT	1.33	0.72	0.99	0.98	0.80
OXYGEN, WT PCT	4.10	2.08	2.43	1.81	1.40
CCR, WT PCT	23.78	16.71	18.37	14.00	16.39
K.V.(100 C)	-	-	-	-	976.60
HETEROATOM REMOVAL					
DESULFURIZATION, PCT WT	-	81.4	66.1	80.8	86.0
DENITROGENATION, PCT WT	-	47.2	27.5	33.8	42.2
DEOXYGENATION, PCT WT	-	50.5	42.3	60.3	67.2
CCR REMOVAL, PCT WT	-	31.5	24.8	47.1	33.8
HYDROGEN CONSUMPTION, SCF/B					
TOTAL H2 CONSUMPTION	-	1359	915	1796	1481
H2 CONSUMED BY C1-C5	-	64	108	961	146
H2 CONSUMED BY C6+	-	959	553	491	943
H2 CONSUMED BY S	-	35	28	34	37
H2 CONSUMED BY N	-	103	60	74	92
H2 CONSUMED BY O	-	198	166	237	264

TABLE A-4

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: HARSHAW HT-500
PROGRAM: 2755   UNIT: 146

	MB-912	MB-913	MB-914	MB-915
MATERIAL BALANCE, PCT WT	70.8	99.9	80.8	148.3
OPERATING CONDITIONS				
PRESSURE, PSIG	2000	2000	2000	2350
AVG REACTOR TEMP., °F	707	724	779	776
LHSV VFF/HR/VCAT	0.43	0.98	0.97	0.61
H2 CHG, SCFB FF NLB	5877	7509	5334	6798
CATALYST AGE, DAYS	2.3	2.9	3.3	3.6
YIELDS (BASED ON CHARGE)				
TOT C1-C3, WT PCT	0.36	0.37	3.50	0.33
TOT C4, WT PCT	0.07	0.39	2.69	0.79
TOT C5, WT PCT	0.01	0.05	0.77	0.10
C6+ PRODUCT, WT PCT	97.49	97.37	91.31	96.13
H2S, WT PCT	0.77	0.63	0.76	0.81
NH3, WT PCT	0.76	0.44	0.55	0.68
H2O, WT PCT	2.33	1.95	2.78	3.10
DISTILLATION (D2887), °F				
IBP	-	-	-	-
5 PCT VOL	-	-	-	-
10 '' ''	-	-	-	-
30 '' ''	-	-	-	-
50 '' ''	-	-	-	-
70 '' ''	-	-	-	-
90 '' ''	-	-	-	-
95 '' ''	-	-	-	-
EP	-	-	-	-

TABLE A-5

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-1
PROGRAM: 2772 UNIT: 146

	CHARGE	MB-916	MB-917	MB-918	MB-919	MB-920
OPERATING CONDITIONS						
PRESSURE, PSIG	-	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	724	727	773	777	762
LHSV VFF/HR/VCAT	-	0.52	1.04	1.06	1.70	0.59
LIQUID PRODUCT PROPERTIES						
GRAVITY, API	-7.8	0.6	-4.9	-1.8	-3.9	1.0
HYDROGEN, WT PCT	6.64	7.80	7.29	7.72	7.28	8.02
SULFUR, WT PCT	0.730	0.150	0.280	0.190	0.260	0.130
NITROGEN, WT PCT	1.37	1.14	1.25	1.22	1.29	1.16
OXYGEN, WT PCT	4.54	2.35	2.73	1.77	2.18	1.17
CCR, WT PCT	26.92	17.55	22.90	19.84	22.76	18.50
K.V.(100 C)	-	22.80	-	70.85	-	30.69
HETEROATOM REMOVAL						
DESULFURIZATION, PCT WT	-	80.4	63.6	74.9	65.5	82.7
DENITROGENATION, PCT WT	-	20.4	13.5	14.2	8.9	17.6
DEOXYGENATION, PCT WT	-	50.5	43.0	62.5	53.5	74.9
CCR REMOVAL, PCT WT	-	37.7	19.4	29.0	18.2	33.1
HYDROGEN CONSUMPTION, SCF/B						
TOTAL H2 CONSUMPTION	-	1226	878	1102	697	1589
H2 CONSUMED BY C1-C5	-	246	145	175	117	317
H2 CONSUMED BY C6+	-	689	495	600	306	881
H2 CONSUMED BY S	-	28	22	26	23	29
H2 CONSUMED BY N	-	46	30	32	20	39
H2 CONSUMED BY O	-	218	186	270	231	323

TABLE A-6

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-1
PROGRAM: 2772 UNIT: 146

	MB-916	MB-917	MB-918	MB-919	MB-920
MATERIAL BALANCE, PCT WT	102.7	110.8	91.7	94.3	95.3
OPERATING CONDITIONS					
PRESSURE, PSIG	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	724	727	773	777	762
LHSV VFF/HR/VCAT	0.52	1.04	1.06	1.70	0.59
H2 CHG, SCFB FF NLB	7939	6254	5381	7312	7665
CATALYST AGE, DAYS	1.3	6.0	2.4	2.9	3.4
YIELDS (BASED ON CHARGE)					
TOT C1-C3, WT PCT	0.93	0.32	0.88	0.61	1.14
TOT C4, WT PCT	0.75	0.08	0.23	0.14	0.93
TOT C5, WT PCT	0.12	0.69	0.01	0.01	0.06
C6+ PRODUCT, WT PCT	96.28	97.15	96.32	96.78	95.21
H2S, WT PCT	0.62	0.49	0.58	0.51	0.64
NH3, WT PCT	0.34	0.22	0.24	0.15	0.29
H2O, WT PCT	2.58	2.20	3.19	2.74	3.83
DISTILLATION (D2887), °F					
IBP	286	-	281	-	287
5 PCT VOL	433	-	451	-	440
10 '' ''	472	-	585	-	486
30 '' ''	561	-	707	-	589
50 '' ''	659	-	-	-	692
70 '' ''	-	-	-	-	-
90 '' ''	-	-	-	-	-
95 '' ''	-	-	-	-	-
EP	-	-	-	-	-

TABLE A-7

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: AMOCAT 1B
PROGRAM: 2820 UNIT: 146

	CHARGE	MB-929	MB-930	MB-931	MB-932	MB-934	MB-935	MB-936
OPERATING CONDITIONS								
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	714	715	754	775	777	801	778
LHSV VFF/HR/VCAT	-	0.49	0.83	0.85	1.58	0.44	0.34	0.45
LIQUID PRODUCT PROPERTIES								
GRAVITY, API	¯8.4	¯0.6	¯2.4	16.7	¯1.1	9.0	7.1	3.8
HYDROGEN, WT PCT	6.65	7.65	7.02	7.89	7.53	9.14	8.83	8.34
SULFUR, WT PCT	0.870	0.390	0.470	0.273	0.380	0.400	0.170	0.208
NITROGEN, WT PCT	1.30	0.97	1.11	0.94	1.08	0.64	0.70	0.90
OXYGEN, WT PCT	4.42	3.02	3.50	2.14	2.68	0.76	0.59	1.81
CCR, WT PCT	26.60	15.24	15.10	13.49	16.33	8.35	8.17	12.90
K.V.(100 C)	-	20.77	60.15	8.91	24.38	2.88	2.23	5.99
HETEROATOM REMOVAL								
DESULFURIZATION, PCT WT	-	56.0	46.8	69.7	57.3	57.2	82.6	77.4
DENITROGENATION, PCT WT	-	26.7	15.9	30.1	18.9	54.2	52.0	34.5
DEOXYGENATION, PCT WT	-	32.9	22.0	53.2	40.8	84.0	88.1	61.3
CCR REMOVAL, PCT WT	-	43.7	44.1	51.0	40.0	70.8	72.6	54.1
HYDROGEN CONSUMPTION, SCF/B								
TOTAL H2 CONSUMPTION	-	1021	415	1343	941	2961	3010	1984
H2 CONSUMED BY C1-C5	-	68	38	189	102	886	1442	565
H2 CONSUMED BY C6+	-	715	218	808	582	1536	1005	1022
H2 CONSUMED BY S	-	25	21	31	26	26	37	35
H2 CONSUMED BY N	-	62	37	70	44	125	120	80
H2 CONSUMED BY O	-	151	101	244	187	386	405	282

TABLE A-8

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION

CHARGE: 50/50 *W. KENTUCKY SCT SRC / RECYCLE SOLVENT*
CATALYST: AMOCAT 1B
PROGRAM: 2820 *UNIT*: 146

	MB-929	*MB*-930	*MB*-931	*MB*-932	*MB*-934	*MB*-935	*MB*-936
MATERIAL BALANCE, PCT WT	100.5	99.8	105.3	102.2	100.4	105.3	99.1
OPERATING CONDITIONS							
PRESSURE, PSIG	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °*F*	714	715	754	775	777	801	778
LHSV VFF/HR/VCAT	0.49	0.83	0.85	1.58	0.44	0.34	0.45
H2 CHG, SCFB FF NLB	6748	5875	9385	7591	7982	11788	6757
CATALYST AGE, DAYS	0.7	1.3	1.6	2.0	3.1	5.1	5.9
YIELDS (BASED ON CHARGE)							
*TOT C*1-*C*3, *WT PCT*	0.33	0.18	0.98	0.47	3.92	6.59	2.33
*TOT C*4, *WT PCT*	0.07	0.05	0.16	0.12	1.19	1.73	1.03
*TOT C*5, *WT PCT*	-	-	0.01	0.01	0.26	0.27	0.17
*C*6+ *PRODUCT, WT PCT*	98.26	98.50	96.70	97.68	92.64	89.08	94.56
*H*2*S, WT PCT*	0.52	0.43	0.64	0.53	0.53	0.76	0.72
*NH*3, *WT PCT*	0.42	0.25	0.48	0.30	0.86	0.82	0.55
*H*2*O, WT PCT*	1.64	1.10	2.65	2.03	4.18	4.39	3.05
DISTILLATION (*D*2887),°*F*							
IBP	320	348	295	326	180	179	255
5 *PCT VOL*	445	460	430	446	383	353	425
10 '' ''	477	479	467	484	472	420	467
30 '' ''	557	560	549	574	588	518	564
50 '' ''	652	651	615	679	751	591	669
70 '' ''	-	-	838	-	-	828	-
90 '' ''	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-

TABLE A-9

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: CYANAMID HDN-1197
PROGRAM: 2827 UNIT: 146

	CHARGE	MB-937	MB-938
OPERATING CONDITIONS			
PRESSURE, PSIG	-	2000	2000
AVG REACTOR TEMP., °F	-	722	711
LHSV VFF/HR/VCAT	-	0.44	0.76
LIQUID PRODUCT PROPERTIES			
GRAVITY, API	-8.7	7.5	3.3
HYDROGEN, WT PCT	6.73	9.16	8.43
SULFUR, WT PCT	0.670	0.066	0.148
NITROGEN, WT PCT	1.40	0.68	0.85
OXYGEN, WT PCT	4.30	3.99	2.50
CCR, WT PCT	24.42	9.46	15.26
K.V.(100 C)	-	4.49	473.70
HETEROATOM REMOVAL			
DESULFURIZATION, PCT WT	-	90.1	78.3
DENITROGENATION, PCT WT	-	51.1	40.3
DEOXYGENATION, PCT WT	-	6.7	42.8
CCR REMOVAL, PCT WT	-	60.7	38.0
HYDROGEN CONSUMPTION, SCF/B			
TOTAL H2 CONSUMPTION	-	2375	1712
H2 CONSUMED BY C1-C5	-	61	47
H2 CONSUMED BY C6+	-	2119	1335
H2 CONSUMED BY S	-	32	28
H2 CONSUMED BY N	-	132	104
H2 CONSUMED BY O	-	31	198

TABLE A-10

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: CYANAMID HDN-1197
PROGRAM: 2827 UNIT: 146

	MB-937	MB-938
MATERIAL BALANCE, PCT WT	93.1	97.6
OPERATING CONDITIONS		
PRESSURE, PSIG	2000	2000
AVG REACTOR TEMP., °F	722	711
LHSV VFF/HR/VCAT	0.44	0.76
H_2 CHG, SCFB FF NLB	6993	7608
CATALYST AGE, DAYS	1.1	2.9
YIELDS (BASED ON CHARGE)		
TOT C_1-C_3, WT PCT	0.28	0.22
TOT C_4, WT PCT	0.06	0.06
TOT C_5, WT PCT	0.01	-
C_6+ PRODUCT, WT PCT	100.60	98.42
H_2S, WT PCT	0.64	0.56
NH_3, WT PCT	0.87	0.69
H_2O, WT PCT	0.32	2.07
DISTILLATION (D2887), °F		
IBP	219	236
5 PCT VOL	366	388
10 '' ''	408	426
30 '' ''	487	498
50 '' ''	554	569
70 '' ''	677	-
90 '' ''	-	-
95 '' ''	-	-
EP	-	-

TABLE A-11

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 *W. KENTUCKY SCT SRC / RECYCLE SOLVENT*
CATALYST: *MOBIL HCL*-2
PROGRAM: 2842 *UNIT*: 146

	CHARGE	*MB*-939	*MB*-940	*MB*-941	*MB*-942	*MB*-943
OPERATING CONDITIONS						
PRESSURE, PSIG	-	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °*F*	-	725	728	774	765	775
LHSV VFF/HR/VCAT	-	0.46	0.89	0.83	0.46	1.82
LIQUID PRODUCT PROPERTIES						
GRAVITY, API	-7.9	3.3	1.8	5.6	7.1	1.7
HYDROGEN, WT PCT	6.71	7.98	7.78	8.35	9.30	7.94
SULFUR, WT PCT	0.750	0.112	0.164	0.047	0.034	0.189
NITROGEN, WT PCT	1.39	0.92	1.05	0.82	0.69	1.00
OXYGEN, WT PCT	4.40	1.69	2.38	1.27	1.17	2.37
CCR, WT PCT	23.56	13.98	14.44	12.08	11.22	16.16
K.V.(100 *C*)	-	9.64	29.30	5.44	3.31	23.90
HETEROATOM REMOVAL						
DESULFURIZATION, PCT WT	-	87.1	80.0	94.0	95.8	75.6
DENITROGENATION, PCT WT	-	42.9	31.1	43.4	53.9	30.3
DEOXYGENATION, PCT WT	-	67.2	51.1	72.6	75.5	48.3
CCR REMOVAL, PCT WT	-	74.7	44.1	50.8	55.8	33.5
HYDROGEN CONSUMPTION, SCF/B						
*TOTAL H*2 *CONSUMPTION*	-	1857	1375	1578	3872	1181
*H*2 *CONSUMED BY C*1-*C*5	-	1091	612	161	1781	120
*H*2 *CONSUMED BY C*6+	-	354	448	979	1616	761
*H*2 *CONSUMED BY S*	-	31	29	33	34	27
*H*2 *CONSUMED BY N*	-	97	70	98	122	68
*H*2 *CONSUMED BY O*	-	284	216	307	319	204

TABLE A-11 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION

CHARGE: 50/50 *W. KENTUCKY SCT SRC / RECYCLE SOLVENT*
CATALYST: MOBIL HCL-2
PROGRAM: 2842 *UNIT:* 146

	CHARGE	*MB-944*	*MB-945*	*MB-946*	*MB-947*	*MB-948*
OPERATING CONDITIONS						
PRESSURE, PSIG	-	1975	1975	1975	2000	2000
AVG REACTOR TEMP., °F	-	800	808	797	727	778
LHSV VFF/HR/VCAT	-	0.76	1.70	0.44	0.43	0.27
LIQUID PRODUCT PROPERTIES						
GRAVITY, API	-7.9	6.0	2.4	9.3	2.2	9.3
HYDROGEN, WT PCT	6.71	8.80	8.02	8.94	7.96	9.60
SULFUR, WT PCT	0.750	0.044	0.115	0.011	0.149	0.092
NITROGEN, WT PCT	1.39	0.73	0.93	0.59	1.07	0.74
OXYGEN, WT PCT	4.40	1.00	1.69	0.71	2.29	0.45
CCR, WT PCT	23.56	10.02	14.89	9.02	14.44	8.93
K.V.(100 C)	-	3.64	9.39	2.80	16.21	3.04
HETEROATOM REMOVAL						
DESULFURIZATION, PCT WT	-	94.3	85.7	98.6	80.6	88.3
DENITROGENATION, PCT WT	-	49.3	37.4	60.2	25.0	49.3
DEOXYGENATION, PCT WT	-	78.3	64.4	85.0	49.8	90.3
CCR REMOVAL, PCT WT	-	59.0	40.9	64.6	40.3	63.9
HYDROGEN CONSUMPTION, SCF/B						
TOTAL H2 CONSUMPTION	-	2002	1498	2182	1176	2443
H2 CONSUMED BY C1-C5	-	180	500	294	92	293
H2 CONSUMED BY C6+	-	1347	610	1358	789	1625
H2 CONSUMED BY S	-	34	31	35	29	31
H2 CONSUMED BY N	-	112	85	136	57	112
H2 CONSUMED BY O	-	331	272	359	210	382

TABLE A-12

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-2
PROGRAM: 2842 UNIT: 146

	MB-939	MB-940	MB-941	MB-942	MB-943	MB-944	MB-945	MB-946	MB-947	MB-948
MATERIAL BALANCE, PCT WT	109.3	102.5	98.6	99.5	107.7	94.8	97.1	97.3	97.3	105.2
OPERATING CONDITIONS										
PRESSURE, PSIG	2000	2000	2000	2000	2000	1975	1975	1975	2000	2000
AVG REACTOR TEMP., °F	725	728	774	765	775	800	808	797	727	778
LHSV VFF/HR/VCAT	0.46	0.89	0.83	0.46	1.82	0.76	1.70	0.44	0.43	0.27
H2 CHG, SCFB FF NLB	8493	6465	6031	14014	9292	9540	7341	8111	8082	5253
CATALYST AGE, DAYS	1.2	1.8	2.3	3.2	3.5	4.1	4.5	5.2	6.2	7.5
YIELDS (BASED ON CHARGE)										
TOT C1-C3, WT PCT	1.89	1.05	0.95	11.28	0.70	1.06	1.83	1.01	0.47	1.55
TOT C4, WT PCT	1.34	0.89	0.13	1.15	0.15	0.14	1.62	0.80	0.11	0.32
TOT C5, WT PCT	5.64	3.07	0.01	0.95	0.02	0.02	0.30	0.31	0.01	0.02
C6+ PRODUCT, WT PCT	88.79	93.08	95.88	99.11	97.16	96.51	93.70	94.70	97.41	95.29
H2S, WT PCT	0.69	0.64	0.75	0.76	0.60	0.75	0.68	0.79	0.64	0.70
NH3, WT PCT	0.73	0.52	0.73	0.91	0.51	0.83	0.63	1.02	0.42	0.83
H2O, WT PCT	3.36	2.55	3.63	3.78	2.41	3.91	3.22	4.25	2.49	4.52
DISTILLATION (D2887),°F										
IBP	264	308	182	213	354	249	317	261	330	-
5 PCT VOL	422	433	414	397	470	406	450	383	459	-
10 " "	467	473	468	468	486	457	481	462	484	-
30 " "	544	558	564	545	571	552	566	544	568	-
50 " "	613	638	622	606	660	614	638	615	660	-
70 " "	-	-	-	727	-	736	-	751	-	-
90 " "	-	-	-	-	-	-	-	-	-	-
95 " "	-	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-	-

TABLE A-13

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: CYANAMID HDS-1443
PROGRAM: 2851 UNIT: 146

	CHARGE	MB-949	MB-950	MB-951	MB-952	MB-953
OPERATING CONDITIONS						
PRESSURE, PSIG	-	1975	1975	1975	1975	1975
AVG REACTOR TEMP., °F	-	721	725	777	769	776
LHSV VFF/HR/VCAT	-	0.39	0.85	0.90	0.43	1.65
LIQUID PRODUCT PROPERTIES						
GRAVITY, API	-7.4	5.9	5.7	4.7	8.8	1.1
HYDROGEN, WT PCT	6.59	8.80	7.35	8.32	9.24	8.02
SULFUR, WT PCT	0.700	0.086	0.161	0.083	0.036	0.133
NITROGEN, WT PCT	1.42	0.78	1.15	0.93	0.64	1.10
OXYGEN, WT PCT	4.30	1.41	2.38	1.25	0.69	2.08
CCR, WT PCT	23.90	12.08	16.96	15.65	9.42	15.50
K.V.(100 C)	-	8.88	26.24	6.99	3.46	12.81
HETEROATOM REMOVAL						
DESULFURIZATION, PCT WT	-	88.3	77.6	88.6	95.1	81.6
DENITROGENATION, PCT WT	-	47.9	21.1	37.1	56.7	25.1
DEOXYGENATION, PCT WT	-	69.0	46.2	72.1	84.6	53.3
CCR REMOVAL, PCT WT	-	51.0	29.4	35.8	61.3	35.9
HYDROGEN CONSUMPTION, SCF/B						
TOTAL H2 CONSUMPTION	-	2093	659	1573	2331	1296
H2 CONSUMED BY C1-C5	-	423	42	181	171	190
H2 CONSUMED BY C6+	-	1249	354	982	1652	804
H2 CONSUMED BY S	-	29	26	29	31	27
H2 CONSUMED BY N	-	110	49	85	130	58
H2 CONSUMED BY O	-	282	189	295	346	218

TABLE A-13 (CONTINUED)

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: CYANAMID HDS-1443
PROGRAM: 2851 UNIT: 146

	CHARGE	MB-954	MB-955	MB-956	MB-957	MB-958
OPERATING CONDITIONS						
PRESSURE, PSIG	-	1975	1975	1975	1975	1975
AVG REACTOR TEMP., °F	-	800	800	791	723	770
LHSV VFF/HR/VCAT	-	0.90	1.75	0.48	0.53	0.32
LIQUID PRODUCT PROPERTIES						
GRAVITY, API	-7.4	1.5	2.1	8.0	10.0	-0.2
HYDROGEN, WT PCT	6.59	8.83	7.96	8.94	7.91	9.06
SULFUR, WT PCT	0.700	0.034	0.125	0.015	0.168	0.040
NITROGEN, WT PCT	1.42	0.79	1.09	0.64	1.17	0.74
OXYGEN, WT PCT	4.30	0.90	1.86	0.78	2.55	0.66
CCR, WT PCT	23.90	10.50	15.09	9.21	17.04	10.48
K.V.(100 C)	-	3.38	8.76	3.39	31.98	3.50
HETEROATOM REMOVAL						
DESULFURIZATION, PCT WT	-	95.4	83.0	98.0	76.9	94.7
DENITROGENATION, PCT WT	-	46.9	26.8	57.0	20.9	51.4
DEOXYGENATION, PCT WT	-	80.1	58.8	82.7	45.2	85.7
CCR REMOVAL, PCT WT	-	57.2	38.5	62.4	30.1	58.2
HYDROGEN CONSUMPTION, SCF/B						
TOTAL H2 CONSUMPTION	-	2044	1335	2150	1281	2456
H2 CONSUMED BY C1-C5	-	266	327	261	339	629
H2 CONSUMED BY C6+	-	1311	679	1388	683	1327
H2 CONSUMED BY S	-	32	27	32	25	31
H2 CONSUMED BY N	-	108	62	131	48	118
H2 CONSUMED BY O	-	327	240	338	185	350

TABLE A-14

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: CYANAMID HDS-1443
PROGRAM: 2851 UNIT: 146

	MB-949	MB-950	MB-951	MB-952	MB-953	MB-954	MB-955	MB-956	MB-957	MB-958
MATERIAL BALANCE, PCT WT	101.9	99.1	98.3	98.3	100.3	100.6	100.1	93.3	101.0	101.3
OPERATING CONDITIONS										
PRESSURE, PSIG	1975	1975	1975	1975	1975	1975	1975	1975	1975	1975
AVG REACTOR TEMP., °F	721	725	777	769	776	800	800	791	723	770
LHSV VFF/HR/VCAT	0.39	0.85	0.90	0.43	1.65	0.90	1.75	0.48	0.53	0.32
H2 CHG, SCFB FF NLB	9809	7849	8794	10123	8017	9064	7510	5891	9310	5272
CATALYST AGE, DAYS	1.8	2.4	2.9	3.8	4.4	4.8	5.3	6.1	7.5	8.5
YIELDS (BASED ON CHARGE)										
TOT C1-C3, WT PCT	1.77	0.26	0.95	0.92	0.76	1.37	1.23	1.42	1.24	3.07
TOT C4, WT PCT	1.12	0.01	0.22	0.22	0.47	0.42	1.13	0.23	1.07	0.94
TOT C5, WT PCT	0.15	-	0.01	0.01	0.12	0.02	0.08	0.01	0.19	0.20
C6+ PRODUCT, WT PCT	94.92	97.42	96.12	96.16	96.75	95.50	95.40	95.47	96.08	93.31
H2S, WT PCT	0.66	0.58	0.66	0.71	0.61	0.71	0.62	0.73	0.57	0.70
NH3, WT PCT	0.83	0.36	0.64	0.98	0.43	0.81	0.46	0.98	0.36	0.89
H2O, WT PCT	3.35	2.24	3.50	4.11	2.59	3.89	2.86	4.01	2.19	4.16
DISTILLATION (D2887),°F										
IBP	266	264	-	265	231	284	318	282	581	240
5 PCT VOL	395	432	-	365	349	405	441	393	655	349
10 " "	446	469	-	416	416	450	472	441	690	420
30 " "	555	567	-	474	633	548	533	542	778	633
50 " "	633	665	-	566	784	584	584	585	871	820
70 " "	-	-	-	616	894	688	647	734	-	978
90 " "	-	-	-	795	-	-	796	-	-	-
95 " "	-	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-	-

TABLE A-15

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-3
PROGRAM: 2873 UNIT: 146

	CHARGE	MB-965	MB-966	MB-967	MB-968	MB-969	MB-970	MB-971	MB-972
OPERATING CONDITIONS									
PRESSURE, PSIG	-	1970	1975	1975	1975	1975	1975	1975	1990
AVG REACTOR TEMP., °F	-	719	802	779	792	729	807	778	703
LHSV VFF/HR/VCAT	-	0.64	0.38	0.52	0.96	0.95	0.73	1.61	0.28
LIQUID PRODUCT PROPERTIES									
GRAVITY, API	-8.0	1.9	14.2	7.6	5.1	-1.0	6.0	0.7	3.3
HYDROGEN, WT PCT	6.38	8.46	9.79	9.32	8.87	8.18	8.90	7.61	8.53
SULFUR, WT PCT	0.700	0.144	0.004	0.041	0.052	0.216	0.099	0.174	0.077
NITROGEN, WT PCT	1.40	0.97	0.26	0.60	0.84	1.16	0.59	1.18	0.91
OXYGEN, WT PCT	4.45	2.02	0.17	0.75	1.37	2.66	0.57	2.48	1.45
CCR, WT PCT	24.64	14.32	4.51	9.68	11.07	18.25	9.16	15.65	14.17
K.V.(100 C)	-	-	4.55	5.60	6.38	120.30	2.99	42.33	20.19
HETEROATOM REMOVAL									
DESULFURIZATION, PCT WT	-	79.9	99.5	94.3	92.8	69.6	86.6	75.5	89.3
DENITROGENATION, PCT WT	-	38.7	84.4	62.4	47.8	26.3	64.0	-26.1	33.3
DEOXYGENATION, PCT WT	-	56.2	96.5	83.9	70.7	41.9	88.0	44.3	67.8
CCR REMOVAL, PCT WT	-	44.6	83.5	62.9	57.9	29.0	65.8	37.3	43.9
HYDROGEN CONSUMPTION, SCF/B									
TOTAL H2 CONSUMPTION	-	1871	3364	2613	2338	1627	2447	1072	1912
H2 CONSUMED BY C1-C5	-	58	635	31	232	84	322	66	59
H2 CONSUMED BY C6+	-	1444	2062	2028	1646	1271	1551	852	1467
H2 CONSUMED BY S	-	27	33	32	31	23	29	25	30
H2 CONSUMED BY N	-	99	215	159	122	67	163	-57	72
H2 CONSUMED BY O	-	244	419	364	307	182	382	185	283

TABLE A-16

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: MOBIL HCL-3
PROGRAM: 2873 UNIT: 146

	MB-965	MB-966	MB-967	MB-968	MB-969	MB-970	MB-971	MB-972
MATERIAL BALANCE, PCT WT	94.7	92.9	129.8	93.2	97.8	98.2	96.1	89.9
OPERATING CONDITIONS								
PRESSURE, PSIG	1970	1975	1975	1975	1975	1975	1975	1990
AVG REACTOR TEMP., °F	719	802	779	792	729	807	778	703
LHSV VFF/HR/VCAT	0.64	0.38	0.52	0.96	0.95	0.73	1.61	0.28
H2 CHG, SCFB FF NLB	6683	11480	7655	9355	7815	10678	5843	11667
CATALYST AGE, DAYS	1.7	2.7	3.7	4.5	4.9	5.4	5.8	6.5
YIELDS (BASED ON CHARGE)								
TOT C1-C3, WT PCT	0.28	2.79	0.11	1.17	0.54	1.56	0.33	0.21
TOT C4, WT PCT	0.09	1.42	0.11	0.33	0.03	0.65	0.10	0.21
TOT C5, WT PCT	0.01	0.09	0.01	0.01	0.02	0.03	0.01	0.01
C6+ PRODUCT, WT PCT	97.89	92.88	97.06	96.37	98.40	94.64	98.65	97.54
H2S, WT PCT	0.59	0.74	0.70	0.69	0.52	0.64	0.56	0.66
NH3, WT PCT	0.73	1.59	1.18	0.90	0.50	1.21	0.42	0.54
H2O, WT PCT	2.85	4.90	4.26	3.59	2.13	4.47	2.19	3.35
DISTILLATION (D2887),°F								
IBP	-	224	177	284	-	-	312	264
5 PCT VOL	-	353	367	376	-	-	425	426
10 " "	-	412	446	422	-	-	444	445
30 " "	-	516	549	509	-	-	534	564
50 " "	-	583	616	603	-	-	584	651
70 " "	-	703	873	912	-	-	651	-
90 " "	-	-	-	-	-	-	870	-
95 " "	-	-	-	-	-	-	982	-
EP	-	-	-	-	-	-	-	-

TABLE A-17

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: AMOCAT 1A
PROGRAM: 2887 UNIT: 146

	CHARGE	MB-979	MB-980	MB-981	MB-982	MB-983	MB-984	MB-985	MB-986
OPERATING CONDITIONS									
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	722	807	774	777	727	809	773	720
LHSV VFF/HR/VCAT	-	0.48	0.56	0.43	0.91	0.92	0.92	1.68	0.44
LIQUID PRODUCT PROPERTIES									
GRAVITY, API	-8.3	5.8	7.9	5.5	2.0	-2.6	3.3	-2.1	-1.6
HYDROGEN, WT PCT	6.61	8.71	8.58	8.30	7.73	7.11	7.55	7.20	7.46
SULFUR, WT PCT	0.750	0.103	0.029	0.063	0.145	0.310	0.120	0.271	0.211
NITROGEN, WT PCT	1.42	0.88	0.77	0.94	1.07	1.26	1.03	1.26	1.24
OXYGEN, WT PCT	4.30	1.35	1.15	1.04	1.54	2.89	1.15	3.03	2.26
CCR, WT PCT	23.70	12.47	5.99	10.64	14.56	19.02	13.31	19.83	17.37
K.V.(100 C)	-	7.83	3.24	4.80	11.17	90.23	8.16	70.93	-
HETEROATOM REMOVAL									
DESULFURIZATION, PCT WT	-	86.6	96.3	92.0	81.4	59.5	85.0	64.6	74.0
DENITROGENATION, PCT WT	-	39.7	48.7	37.1	27.6	13.0	32.0	13.2	19.2
DEOXYGENATION, PCT WT	-	69.4	74.6	77.0	65.5	34.0	74.9	30.9	51.3
CCR REMOVAL, PCT WT	-	48.8	76.1	57.3	41.0	21.4	47.4	18.2	32.2
HYDROGEN CONSUMPTION, SCF/B									
TOTAL H2 CONSUMPTION	-	1894	2045	1732	1164	512	1209	619	1202
H2 CONSUMED BY C1-C5	-	75	449	327	186	50	442	109	522
H2 CONSUMED BY C6+	-	1411	1142	970	617	272	356	329	398
H2 CONSUMED BY S	-	31	34	33	29	21	30	23	26
H2 CONSUMED BY N	-	92	113	86	64	30	74	31	45
H2 CONSUMED BY O	-	284	306	315	268	139	307	127	210

TABLE A-18

FIXED BED HYDROPROCESSING OF COAL LIQUIDS--- CATALYST EVALUATION
YIELDS AND DISTILLATION

CHARGE: 50/50 W. KENTUCKY SCT SRC / RECYCLE SOLVENT
CATALYST: AMOCAT 1A
PROGRAM: 2887 UNIT: 146

	MB-979	MB-980	MB-981	MB-982	MB-983	MB-984	MB-985	MB-986
MATERIAL BALANCE, PCT WT	90.6	95.3	98.0	101.6	101.3	99.6	99.0	103.4
OPERATING CONDITIONS								
PRESSURE, PSIG	2000	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	722	807	774	777	727	809	773	720
LHSV VFF/HR/VCAT	0.48	0.56	0.43	0.91	0.92	0.92	1.68	0.44
H2 CHG, SCFB FF NLB	8130	7650	10930	7565	7130	6482	7466	7653
CATALYST AGE, DAYS	1.0	2.0	3.0	3.6	4.1	4.6	5.1	6.0
YIELDS (BASED ON CHARGE)								
TOT C1-C3, WT PCT	0.36	2.37	1.70	0.98	0.25	2.06	0.58	1.32
TOT C4, WT PCT	0.13	0.48	0.40	0.21	0.07	0.79	0.14	2.06
TOT C5, WT PCT	0.01	0.02	0.01	0.01	-	0.12	0.01	0.65
C6+ PRODUCT, WT PCT	97.27	94.61	95.08	96.05	98.01	93.78	97.85	94.16
H2S, WT PCT	0.69	0.77	0.73	0.65	0.47	0.68	0.52	0.59
NH3, WT PCT	0.69	0.84	0.64	0.48	0.22	0.55	0.23	0.33
H2O, WT PCT	3.35	3.61	3.72	3.17	1.64	3.62	1.49	2.48
DISTILLATION (D2887), °F								
IBP	-	182	215	-	-	-	282	324
5 PCT VOL	-	404	465	-	-	-	457	469
10 '' ''	-	466	501	-	-	-	493	504
30 '' ''	-	560	583	-	-	-	636	585
50 '' ''	-	636	663	-	-	-	-	661
70 '' ''	-	899	919	-	-	-	-	855
90 '' ''	-	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-

TABLE A-19

FIXED BED HYDROPROCESSING OF INDIANA V SRC--- LOW SULFUR SAMPLE
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2698 UNIT: 146

	CHARGE	MB-891	CHARGE	MB-893	MB-894
OPERATING CONDITIONS					
PRESSURE, PSIG	-	2000	-	2000	2000
AVG REACTOR TEMP., °F	-	726	-	724	725
LHSV VFF/HR/VCAT	-	0.37	-	0.43	0.87
LIQUID PRODUCT PROPERTIES					
GRAVITY, API	¯15.7	¯4.6	¯15.0	¯8.8	¯9.6
HYDROGEN, WT PCT	6.85	7.10	5.92	6.97	6.49
SULFUR, WT PCT	0.380	0.200	0.380	0.200	0.150
NITROGEN, WT PCT	1.51	0.86	1.43	0.86	1.00
OXYGEN, WT PCT	3.71	2.10	3.60	2.30	2.70
CCR, WT PCT	45.34	30.50	42.96	37.32	38.65
K.V.(100 C)	-	-	-	9350.00	-
HETEROATOM REMOVAL					
DESULFURIZATION, PCT WT	-	49.2	-	48.5	61.2
DENITROGENATION, PCT WT	-	45.0	-	41.1	31.2
DEOXYGENATION, PCT WT	-	45.4	-	37.4	26.3
CCR REMOVAL, PCT WT	-	35.1	-	14.9	11.5
HYDROGEN CONSUMPTION, SCF/B					
TOTAL H2 CONSUMPTION	-	460	-	1125	650
H2 CONSUMED BY C1-C5	-	166	-	152	95
H2 CONSUMED BY C6+	-	¯4	-	726	370
H2 CONSUMED BY S	-	9	-	9	12
H2 CONSUMED BY N	-	118	-	102	77
H2 CONSUMED BY O	-	171	-	136	95

TABLE A-19 (CONTINUED)

FIXED BED HYDROPROCESSING OF INDIANA V SRC--- LOW SULFUR SAMPLE
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 70/30 *INDIANA V SRC / RECYCLE SOLVENT*
CATALYST: HARSHAW 618*X*
PROGRAM: 2698 *UNIT:* 146

	CHARGE	*MB*-895	*MB*-896	*MB*-897	*MB*-898	*MB*-899	*MB*-900	*MB*-901
OPERATING CONDITIONS								
PRESSURE, PSIG	-	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	785	803	720	723	799	799	775
LHSV VFF/HR/VCAT	-	1.90	0.50	0.39	0.22	1.57	0.84	0.77
LIQUID PRODUCT PROPERTIES								
GRAVITY, API	¯12.3	¯6.2	¯5.6	¯6.1	¯3.1	¯9.7	¯8.0	¯8.6
HYDROGEN, WT PCT	6.25	7.02	7.86	7.37	7.90	6.50	6.88	6.57
SULFUR, WT PCT	0.710	0.246	0.064	0.215	0.163	0.250	0.127	0.182
NITROGEN, WT PCT	1.53	1.22	0.93	1.19	0.97	1.31	0.91	1.32
OXYGEN, WT PCT	3.90	2.30	1.00	2.10	1.30	2.40	1.60	2.10
CCR, WT PCT	44.00	24.66	15.35	23.06	19.61	28.49	32.78	36.25
K.V.(100 *C*)	-	129.10	10.93	392.60	142.20	-	-	-
HETEROATOM REMOVAL								
DESULFURIZATION, PCT WT	-	66.2	91.4	70.5	77.9	65.9	83.1	75.4
DENITROGENATION, PCT WT	-	22.2	41.8	24.3	39.1	17.1	43.8	17.4
DEOXYGENATION, PCT WT	-	42.5	75.5	47.6	68.0	40.4	61.3	48.4
CCR REMOVAL, PCT WT	-	45.3	66.6	49.0	57.2	37.3	29.6	21.1
HYDROGEN CONSUMPTION, SCF/B								
TOTAL H2 CONSUMPTION	-	818	1677	1143	1700	428	988	595
H2 CONSUMED BY C1-C5	-	106	248	148	260	173	415	304
H2 CONSUMED BY C6+	-	468	999	724	1050	32	194	33
H2 CONSUMED BY S	-	23	32	25	27	23	29	26
H2 CONSUMED BY N	-	57	108	63	101	44	113	45
H2 CONSUMED BY O	-	163	290	183	262	156	236	186

TABLE A-20

FIXED BED HYDROPROCESSING OF INDIANA V SRC--- LOW SULFUR SAMPLE
YIELDS AND DISTILLATION

CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2698 UNIT: 146

	MB-891	MB-893	MB-894	MB-895	MB-896	MB-897	MB-898	MB-899	MB-900	MB-901
MATERIAL BALANCE, PCT WT	118.7	104.2	110.8	87.9	103.8	92.2	96.7	99.4	110.8	122.3
OPERATING CONDITIONS										
PRESSURE, PSIG	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	726	724	725	785	803	720	723	799	799	775
LHSV VFF/HR/VCAT	0.37	0.43	0.87	1.90	0.50	0.39	0.22	1.57	0.84	0.77
H2 CHG, SCFB FF NLB	7401	8278	6699	4445	5882	8222	6036	6988	6250	4448
CATALYST AGE, DAYS	1.5	6.7	7.3	8.1	9.0	10.0	10.6	11.1	11.6	12.1
YIELDS (BASED ON CHARGE)										
TOT C1-C3, WT PCT	0.61	0.53	0.44	0.45	1.07	0.54	1.10	0.70	1.88	1.38
TOT C4, WT PCT	0.42	0.42	0.17	0.21	0.54	0.41	0.57	0.39	0.69	0.51
TOT C5, WT PCT	0.09	0.09	0.01	0.08	0.04	0.09	0.11	0.08	0.10	0.08
C6+ PRODUCT, WT PCT	96.54	97.93	98.33	97.54	95.74	97.35	96.09	96.79	94.45	95.78
H2S, WT PCT	0.20	0.20	0.25	0.50	0.69	0.53	0.59	0.50	0.63	0.57
NH3, WT PCT	0.83	0.71	0.54	0.41	0.78	0.45	0.73	0.32	0.82	0.32
H20, WT PCT	1.89	1.52	1.06	1.87	3.31	2.09	2.98	1.78	2.69	2.13
DISTILLATION (D2887), °F										
IBP	-	-	-	-	-	-	402	-	-	-
5 PCT VOL	-	-	-	-	-	-	477	-	-	-
10 '' ''	-	-	-	-	-	-	525	-	-	-
30 '' ''	-	-	-	-	-	-	684	-	-	-
50 '' ''	-	-	-	-	-	-	901	-	-	-
70 '' ''	-	-	-	-	-	-	-	-	-	-
90 '' ''	-	-	-	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-	-	-

TABLE A-21

FIXED BED HYDROPROCESSING OF INDIANA V SRC--- HIGH SULFUR SAMPLE
TLP PROPERTIES AND HYDROGEN CONSUMPTION
CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2782 UNIT: 146

	CHARGE	MB-921	MB-922	MB-923	MB-924	CHARGE	MB-925	MB-926	MB-927	MB-928
OPERATING CONDITIONS										
PRESSURE, PSIG	-	2000	2000	2000	2000	-	2000	2000	2000	2000
AVG REACTOR TEMP., °F	-	722	723	776	768	-	780	776	777	798
LHSV VFF/HR/VCAT	-	0.41	0.81	0.91	1.51	-	1.81	1.14	0.58	0.48
LIQUID PRODUCT PROPERTIES										
GRAVITY, API	¯13.6	¯5.7	¯8.3	¯6.2	¯9.7	¯13.5	¯8.5	¯7.4	¯7.1	11.4
HYDROGEN, WT PCT	6.44	7.50	7.20	6.95	7.17	6.43	6.93	7.47	7.64	7.18
SULFUR, WT PCT	0.620	0.280	0.299	0.220	0.350	0.620	0.242	0.256	0.191	0.094
NITROGEN, WT PCT	1.78	1.32	1.68	1.40	1.70	1.73	1.28	1.59	1.38	1.16
OXYGEN, WT PCT	3.50	2.38	2.97	2.24	2.44	3.25	3.08	2.07	1.75	1.37
CCR, WT PCT	27.80	31.87	33.77	29.77	36.72	36.80	25.35	32.42	32.59	25.48
K.V.(100 C)	-	1178.00	-	-	7203.00	-	-	-	1076.00	-
HETEROATOM REMOVAL										
DESULFURIZATION, PCT WT	-	55.4	52.0	65.5	44.1	-	61.7	59.7	70.3	86.0
DENITROGENATION, PCT WT	-	26.8	6.1	23.5	5.5	-	27.4	10.3	23.1	38.0
DEOXYGENATION, PCT WT	-	33.1	15.8	37.9	31.2	-	21.1	47.2	56.0	66.9
CCR REMOVAL, PCT WT	-	16.8	11.1	23.4	3.9	-	39.5	23.1	23.7	42.8
HYDROGEN CONSUMPTION, SCF/B										
TOTAL H2 CONSUMPTION	-	1025	709	664	710	-	588	1080	1349	1331
H2 CONSUMED BY C1-C5	-	48	46	188	52	-	113	170	309	774
H2 CONSUMED BY C6+	-	763	573	252	518	-	294	681	736	163
H2 CONSUMED BY S	-	17	16	20	14	-	19	18	22	27
H2 CONSUMED BY N	-	81	18	71	17	-	81	30	68	112
H2 CONSUMED BY O	-	116	55	133	109	-	80	180	214	255

TABLE A-22

FIXED BED HYDROPROCESSING OF INDIANA V SRC--- HIGH SULFUR SAMPLE
YIELDS AND DISTILLATION

CHARGE: 70/30 INDIANA V SRC / RECYCLE SOLVENT
CATALYST: HARSHAW 618X
PROGRAM: 2782 UNIT: 146

	MB-921	MB-922	MB-923	MB-924	MB-925	MB-926	MB-927	MB-928
MATERIAL BALANCE, PCT WT	103.7	101.7	99.7	90.8	85.8	92.6	101.5	100.6
OPERATING CONDITIONS								
PRESSURE, PSIG	2000	2000	2000	2000	2000	2000	2000	2000
AVG REACTOR TEMP., °F	722	723	776	768	780	776	777	798
LHSV VFF/HR/VCAT	0.41	0.81	0.91	1.51	1.81	1.14	0.58	0.48
H2 CHG, SCFB FF NLB	8495	6118	5789	8625	2449	3659	5044	6711
CATALYST AGE, DAYS	2.5	3.0	3.4	3.7	5.3	5.4	5.7	6.5
YIELDS (BASED ON CHARGE)								
TOT C1-C3, WT PCT	0.21	0.24	0.93	0.30	0.56	0.73	1.38	3.25
TOT C4, WT PCT	0.11	0.05	0.23	0.01	0.14	0.33	0.54	1.49
TOT C5, WT PCT	0.01	-	0.01	0.01	0.01	0.05	0.07	0.26
C6+ PRODUCT, WT PCT	98.72	99.50	97.24	98.94	98.15	97.60	96.34	92.42
H2S, WT PCT	0.37	0.34	0.43	0.29	0.41	0.39	0.46	0.57
NH3, WT PCT	0.58	0.13	0.51	0.12	0.58	0.22	0.49	0.80
H2O, WT PCT	1.31	0.62	1.50	1.23	0.91	2.04	2.41	2.89
DISTILLATION (D2887), °F								
IBP	-	-	-	-	379	374	400	361
5 PCT VOL	-	-	-	-	454	478	477	465
10 '' ''	-	-	-	-	481	510	509	499
30 '' ''	-	-	-	-	570	630	628	611
50 '' ''	-	-	-	-	685	-	-	904
70 '' ''	-	-	-	-	-	-	-	-
90 '' ''	-	-	-	-	-	-	-	-
95 '' ''	-	-	-	-	-	-	-	-
EP	-	-	-	-	-	-	-	-

Appendix B

PILOT UNIT MODIFICATIONS

Modifications to our pilot unit have been made throughout EPRI Project RP361 and are summarized in previous reports (1,2). These changes were necessary to extend and improve the operability of the pilot unit in the processing of high concentration SRC blends. Additional unit modifications were made during 1978 to alleviate some recurring problems and to further improve the operability and reliability of the unit. Following these modifications, the unit has operated smoothly; no major problems have been encountered.

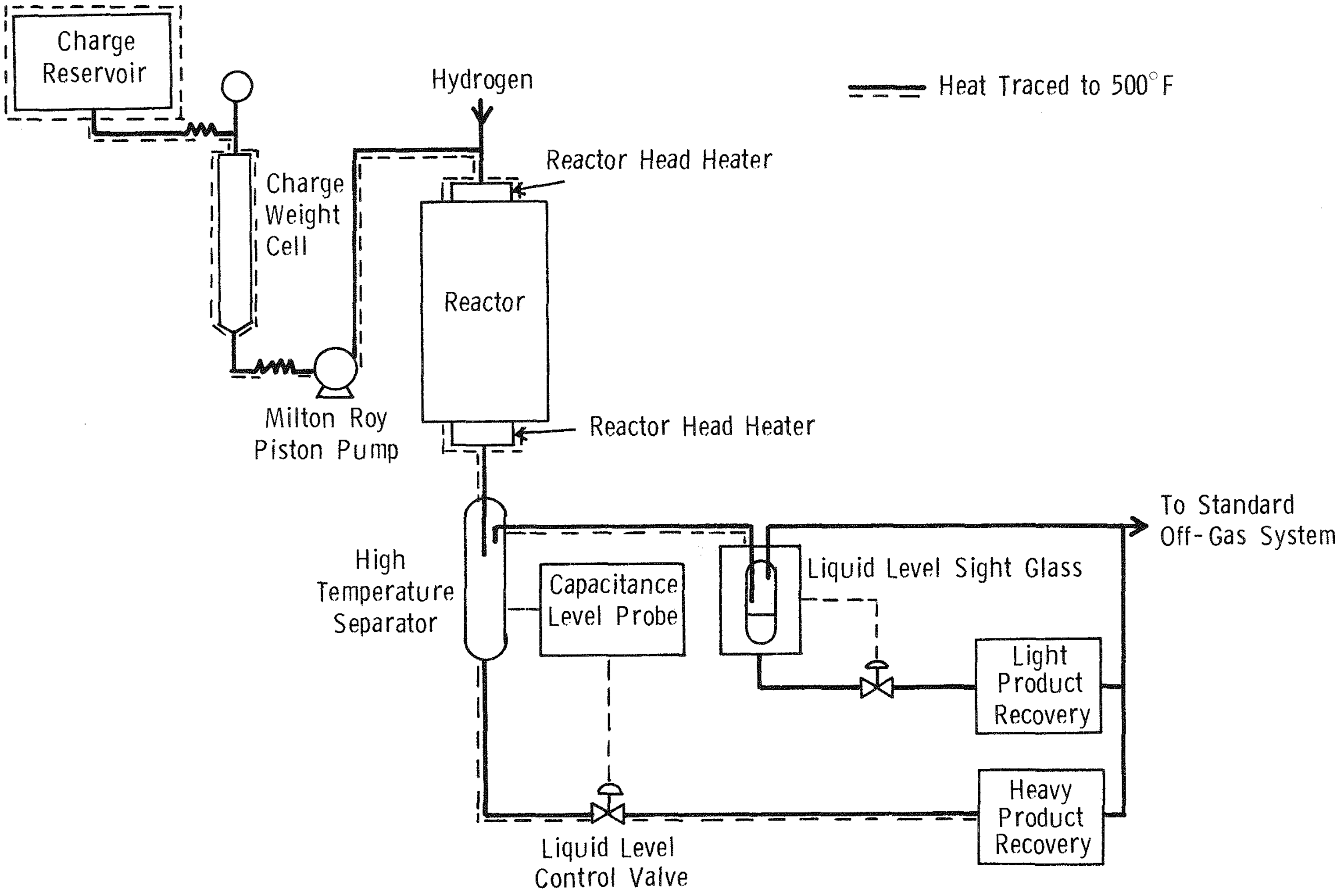
Significant changes have been made to the three major pilot unit sections - the low and high pressure charge systems and the post-reactor high pressure separation system. A schematic diagram of the present unit configuration is shown in Figure B-1.

The low pressure charge system was redesigned to eliminate the blend preparation tank with the gear pump recirculation system. This tank-pump combination was replaced with two parallel, gravity feed charge reservoirs with low speed stirrers for preparing and mixing blends. An intermediate, unstirred charge reservoir was also eliminated by the redesign. The new charge tanks each have a nitrogen purge and vent line with a condenser. The condensers have reduced solvent losses, experienced previously in the charge reservoir. To minimize charge line plugging and to allow ease of gravity feeding to both the weigh cell and charge pump, the 1/4" diameter charge lines were replaced with 3/8" diameter lines. More direct, straight-line flow paths were used, and straight-through ball valves were installed. The weigh cell was further insulated to eliminate air drafts that tended to cool and solidify the higher concentration blends in the line from the weigh cell.

Several changes were made in the high pressure charge system in order to improve pump operability and to reduce valve failure. Erratic pump rates during earlier runs were remedied by adjustment of a set screw on the pump plunger. Check valves on the pump were cleaned and replaced when they failed to seat properly. Problems

Figure B-1

DIAGRAM OF MODIFIED UNIT FOR HYDROPROCESSING COAL LIQUIDS CT-146



with vapor locking and pump seizure were reduced by careful control of the pump head temperature when switching from solvent to SRC during unit start-ups. Intermittent leaking of high pressure valves has been virtually eliminated by replacing the graphitic valve packing with new teflon-glass material. The diaphragm of a malfunctioning high pressure gauge was also replaced early in the year.

Improved operation of the high and low temperature high pressure separators has been realized as a result of two major changes. An examination of the capacitance probe in the high pressure separator was made after recurring problems with the liquid level control. The teflon sleeve on the capacitance probe was found to be blistered and had shrunk back from the end of the probe. Installation of a new probe cured the immediate problems, but "spikes" (high frequency fluctuations) were recorded in the liquid level indicator readout. Based on the hypothesis that the spikes were caused by interference from water in the product, a variable response time delay was added to the capacitance probe output signal. By damping out erroneous signals, the high pressure liquid level control system has since shown significantly improved stability. A new liquid level sight glass with a graphite-foil gasket was installed to replace the temporary all metal separator. The previously used sight glasses with graphite-organic binder gaskets repeatedly failed during the previous years' work. The graphite gaskets were found to be weakened by the coal liquids in contact with them. The graphite-foil package has held up well since they were installed.

REFERENCES

1. Bendoraitis, J. G., A. V. Cabal, R. B. Callen, M. J. Dabkowski, R. H. Heck, H. R. Ireland, C. A. Simpson, and T. R. Stein, Annual Report, EPRI Contract No. AF-444 (RP 361-2), Mobil Research and Development Corporation, October 1977.

2. Cabal, A. V., R. B. Callen, M. J. Dabkowski, R. H. Heck, S. S. Shih, C. A. Simpson, and T. R. Stein, Annual Report, EPRI Contract No. AF-873 (RP 361-2), Mobil Research and Development Corporation, December 1978.

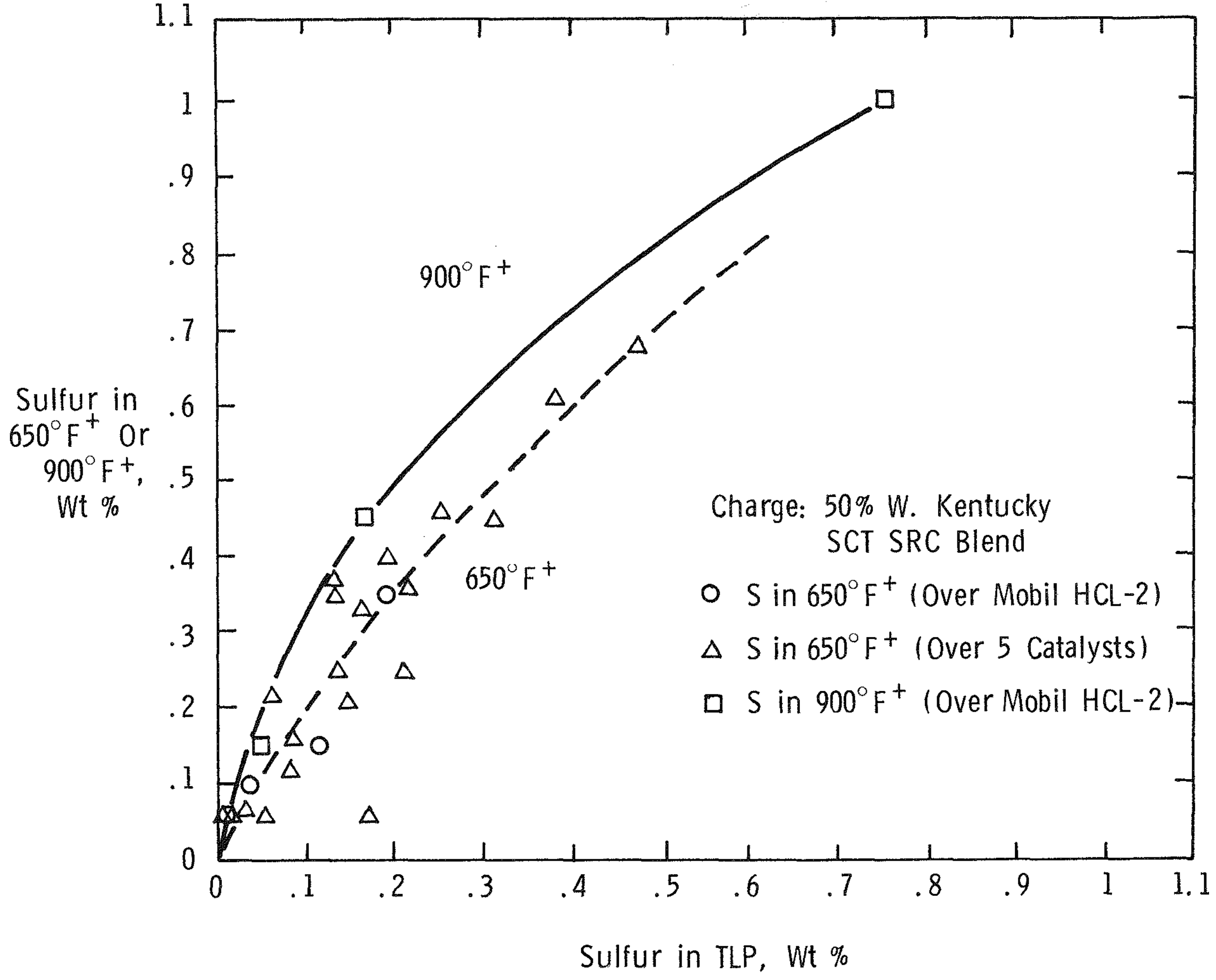
Appendix C

SULFUR DISTRIBUTION IN W. KENTUCKY SCT SRC

In the characterization of hydrotreated W. Kentucky SCT SRC blends, the sulfur distribution between the 650°F$^+$ fraction and the total liquid product (TLP) was established. This sulfur distribution is shown in Figure C-1; the 900°F$^+$ S vs. TLP S of three samples have also been included. At a sulfur concentration of 0.15 wt % in the TLP, the corresponding sulfur in the 650°F$^+$ and 900°F$^+$ are 0.3 wt % and 0.4 wt %, respectively.

Figure C-1

SULFUR DISTRIBUTION IN HYDROTREATED W. KENTUCKY SCT SRC BLEND



Appendix D

SOFTENING POINT OF HYDROPROCESSED SRC

The grindability of a solid boiler feed is an important property for mechanical handling. Feeds with low softening points have relatively poor grindability properties. In order to establish softening points of hydroprocessed SRC's, we forwarded six samples to Catalytic, Inc. for testing. The sample descriptions, sulfur contents, and softening points are shown in Table D-1. Included in Table D-1 are short contact time (W. Kentucky) and regular (Indiana V) SRC's. The products from the hydroprocessing runs were distilled to 900°F. Only the 900°F$^+$ portions were tested for softening points. Hydrotreatment lowered the softening points of the SRC's by 120-140°F.

Table D-1

SOFTENING POINTS OF SRC

Sample Descriptions	900°F$^+$ S (Wt %)	Total Liquid S (Wt %)	Softening-Melting Point (°F)*
W. Kentucky SCT SRC 76D3653	1.0	0.75	435-446
W. Kentucky SCT SRC CT-146-2842-940-900°F$^+$	0.45	.164	302-320
W. Kentucky SCT SRC CT-146-2842-941-900°F$^+$	0.15	.047	284-302
Indiana V SRC 78D1323	0.83	.75	446-464
Indiana V SRC CT-146-2893-990-900°F$^+$	0.3	.153	320-347
Indiana V SRC CT-146-2893-1002-900°F$^+$	0.5	.31	302-320

* Reported by Catalytic, Inc.