

## The Composition of Liquids from Coals of Different Rank

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### INTRODUCTION

Eight coal liquids prepared from six coals (9,10) of widely differing rank were characterized by procedures previously developed for petroleum (3,4,7) and by ASTM methods. The liquids were prepared and upgraded by hydrogenation in a batch autoclave under conditions intended to minimize cracking of the hydrocarbons (especially those of cyclic and aromatic structure) and yet produce most of the liquid hydrocarbons potentially obtainable from a given coal. The extent of the hydrogenation/hydrogenolysis (henceforth referred to as hydrogenation) of the raw coal liquids was adjusted as required to decrease the nitrogen content to about 0.2 to 0.3 weight-percent with the additional intent of providing a predominantly hydrocarbon liquid for analysis. Details of the coal liquid preparations have been reported previously (9,10). This report covers the characterization of the coal liquids and possible implications in relation to refining.

Among the many reports in the literature on coal liquefaction, several indicate a relationship between the rank of the coal and the complexity of the hydrocarbon groups in the coal and in its liquefaction products. This subject was reviewed in some detail in our previous reports (9,10) and will only be summarized and updated here. Early German work indicated that the lower-rank brown coals (lignite) were more reactive, requiring less hydrogen pressure, and produced smaller polynuclear hydrocarbon units (18) than bituminous coals. Also, the asphaltene content of the coal liquids decreased with the coal rank to a minimum for sub-bituminous coals. Early work by the U.S. Bureau of Mines produced similar results (14). Low rank coals were found to be so reactive that, at a reaction temperature of 430° C, high hydrogen pressure was required to prevent repolymerization of reactive fragments to coke. More recent studies have shown that the product from the liquefaction of a Utah sub-bituminous coal with hydrogen donor solvent (6) contained less benzene insoluble material than that from Pittsburgh bituminous coal.

Although the literature suggests that the complexity of hydrocarbon groups in coal and coal liquefaction products can differ substantially with the rank of the coal, specific data are sketchy and sometimes appear to be contradictory. A recent report on the comparison of solvent-refined lignite and solvent-refined sub-bituminous and bituminous coals (2) indicates hardly any difference among the products in terms of gross combustion analysis, acid and basic titres, molecular weight, and nuclear magnetic resonance, ultraviolet and electron spin resonance spectra. Only the nitrogen content of the coal was reflected in the solvent-refined products. However, in another recent study of a lignite, a bituminous coal, and an anthracite coal, analysis of the organic compounds trapped in the coals and contained in the products of selective oxidation indicated an increased condensation of aromatic rings with increasing rank (8). Analysis was performed by gas chromatography/time-of-flight mass

spectrometry and high resolution mass spectrometry. Furthermore, an extensive, detailed study revealed compositional differences in the pyridine extracts of three coals of different rank and also in their liquefaction products produced by the Synthoil process (1). For example, larger quantities of saturates and higher percentages of lower ring number saturate compound types were observed in the two higher rank coal extracts. However, the ring distributions of the aromatic fractions from the coal extracts indicated slightly higher average ring numbers for the higher rank coals.

Similar results were obtained in a study characterizing liquid products obtained from six coals of different rank by treatment of the coals with NaOH-alcohol for one hour at 300° to 350° C (11). Characterization by elemental analysis, molecular weight determination, and proton NMR indicated that the younger (lower-rank) coals gave simpler products with primarily tetralin-type nuclei and more abundant ether linkages than the higher rank bituminous coals which yielded products with five to six rings, about two of which were naphthenic. Similarly, in Japanese studies of pyridine extracts of coals of different rank (17), analysis by proton NMR indicated more condensed aromatic rings with less substitution and shorter aliphatic chains in the higher rank coal extracts. Extracts from bituminous coals contained an average of four or five aromatic rings with side chains averaging three or four carbons in length, whereas a lignite extract had an average of one or two aromatic rings with seven- or eight-carbon, aliphatic side chains. Finally, a study using carbon-13 NMR to characterize a hard-coal-tar pitch and a brown-coal-tar pitch indicated a predominance of fused aromatic ring systems with small amounts of methyl and hydroaromatic methylene groups for the hard-coal-tar pitch (5). The results for the brown-coal-tar pitch indicated primarily long, straight-chain aliphatics estimated at 25 to 40 carbon atoms in length.

The coal liquids characterized at the Bartlesville Energy Technology Center to date have come from different projects in which the primary objective was the development of a specific process. This precluded a systematic study of the relationship of coal rank to coal liquid composition, which is the objective of this study.

#### EXPERIMENTAL PROCEDURES

The upgraded coal liquids were distilled with a metal-mesh-spinning-band still under vacuum to produce cuts at 200°, 325°, and 425° C. Asphaltenes were then precipitated from each >425° C residuum dissolved in benzene by addition of 50 volumes of normal pentane (12). Further distillations on the asphaltene-free materials, at 202° C and 4 micron pressure using a wiped-wall molecular still, produced 425°-540° C distillate cuts and residua fractions. After removal of acids and bases from the <200° C distillates by extraction methods (16), an additional chromatographic separation with silica gel provided a check for the presence of olefins. No olefins were detected; thus, the acid- and base-free distillates were analyzed by ASTM D2789-71, Standard Method of Test for Hydrocarbon Types in Low Olefinic Gasoline by Mass Spectrometry.

The higher-boiling distillates were characterized by methods adapted from the characterization of heavy ends of petroleum (3,7). A schematic of the procedure is depicted in Figure 1. Details of the procedures have been reported previously (3,7,15,16). Analyses of subfractions of aromatic concentrates separated by gel-permeation chromatography (GPC) were performed on a low-resolution CEC 21-103 C

mass spectrometer (MS) using low-ionizing energy electrons to produce predominantly molecular ions. Compound-type assignments were made by means of previously established GPC-MS correlations and high-resolution mass spectral data for selected fractions. High-resolution, 70 eV, mass spectra were obtained on an AEI MS 3074 mass spectrometer. High- and low-resolution field-ionization spectra of selected fractions, primarily polyaromatic-polar concentrates, were obtained at Oklahoma State University on a CEC Model 21-110 B mass spectrometer fitted with a razor blade source (13).

## RESULTS

Descriptions and analyses of the six coal samples used to prepare the coal liquids are summarized in Table 1. Physical and chemical property data for the eight coal liquids prepared from the six coals are summarized in Table 2. Included in Table 2 are data for repeat runs on Illinois No. 6 and Wyodak coals. The repeat run for the Illinois No. 6 coal was made because of changes in the procedure instituted during the first liquefaction run, Illinois No. 6 run 1. The changes involved a switch from CoMo to NiMo catalyst and a decrease in the maximum temperature from 425° to 400° C (9). Also, upgrading of the first Illinois crude liquid fell short of the targeted value of 0.2 to 0.3 percent nitrogen. The second Wyodak coal liquid was prepared from the same crude liquid as the first by upgrading according to the time-temperature profile used for the Illinois No. 6 run 2 liquid. It was noted previously, that the lower-rank coals reacted more readily and under milder conditions than those of higher rank for both the initial liquefaction and the upgrading of the raw coal liquids (9). Thus, the second Wyodak run was made to determine possible effects of the extent of upgrading upon liquefaction-product composition.

Distillation results for the upgraded coal liquids are also summarized in Table 2. The results showed no large differences for the liquids, except for the extensively upgraded second Wyodak liquid which had a higher percentage of low-boiling material. Asphaltene content of the six "normally" upgraded liquids ranged from about 1 to 7 percent. Inclusion of the second Wyodak and first Illinois liquids extended the ranges of asphaltene content from 0.1 to 14 percent. The asphaltene content correlated with the nitrogen content of the liquids, thus supporting the use of nitrogen content to monitor the extent of coal-liquid upgrading.

Table 3 lists the specific gravity, nitrogen content, and sulfur content of the various distillation fractions obtained from each of the eight coal liquids. Nitrogen content increased for the higher-boiling distillates as expected. All sulfur values were low as expected. Nitrogen contents of the asphaltenes from the bituminous-coal liquids were higher than those from the lower-rank-coal liquids.

Results from the dual silica-gel/alumina-gel adsorption chromatography separations of the 200°-325° C, 325°-425° C, and 425°-540° C coal-liquid distillates are summarized in Table 4. Data for the acid and base extracts of the polyaromatic-polar adsorption fractions are also included in Table 4. Detailed results from the analyses of the saturate concentrates and the fractions from the GPC separations of the aromatic concentrates are not reported here but will be published elsewhere. Compound type assignments and the quantitative results for selected fractions from the GPC separations were checked by high-resolution electron-impact MS and by high- and

low-resolution FI/MS. The results were consistent except for the indication of very small amounts of additional compound types containing S, NO, NO<sub>2</sub>, and O<sub>2</sub>.

The characterization results from all fractions for each coal liquid were combined and expressed in terms of compound types containing the same number of total rings (aromatic and naphthenic). This scheme was chosen because of the different degree of saturation of aromatic rings in the upgrading of the crude liquids from different coals, and the fact that liquefaction and upgrading reaction conditions were chosen to minimize hydrocarbon ring cracking reactions. The results are summarized in Tables 5 and 6.

## DISCUSSION

Examination of the data in Table 5 shows the coal liquids to be more alike than different with regard to distribution by ring number. Differences among the liquids from the several coals doubtless were minimized because the upgrading was adjusted to eliminate heteroatoms to about the same level in most of the liquids. The greater reactivity of the lower-rank coals and the lesser required upgrading of their crude liquids was clear from the previously reported preparations (9). Some scatter in compositional data could be expected because of the variation of details of individual preparations. However, since conditions generally were such as to avoid cracking of hydrocarbons, especially hydrocarbon ring structures, differences or similarities in basic hydrocarbon structures should be valid. One of the largest uncertainties, is the extent of hydrogenation of aromatic hydrocarbon rings originally present in the coal. Total consumption of hydrogen was approximately constant, independent of coal rank (9), so that hydrogen consumed by reaction with high-combined oxygen in lower rank coals was counterbalanced by increased saturation of aromatic rings in the coals of higher rank. With the catalyst and conditions used, many of the polynuclear aromatic structures originally present could be expected to hydrogenate partially to yield hydroaromatics in the upgraded liquids. The uncertainty in the extent of hydrogenation of aromatics is circumvented in part by counting total rings per molecule in the several distillate fractions.

Comparison of the data from the two Illinois No. 6 liquefaction and upgrading runs summarized in Table 6 indicate very little difference in the products in terms of ring number distributions especially in the lower-ring-number compounds. The largest effect in switching from CoMo to NiMo catalyst and decreasing the maximum reaction temperature from 425° to 400° C appears in the compound types containing four or more rings and in the asphaltene content. The second Illinois run with NiMo at 400° C maximum temperature produced more >4-ring compound-type material at the expense of the asphaltenes.

As seen in Table 6, the ring-number data for the two Wyodak runs show that the extent of upgrading was an important factor in final product composition. The more extensive upgrading (Wyodak run 2) produced considerably higher yields of low-ring-number compound types. This was due only in part to more complete conversion of heteroatomic-ring compounds to hydrocarbons. Beyond that, some hydrocarbon cracking must have occurred.

Comparison of the data for the Illinois run 2 and Wyodak run 2 shows significantly higher yields of low-ring-number compound types for the liquid from the lower-rank Wyodak coal. However, since the

Wyodak coal was more reactive, the extent of upgrading as seen in the nitrogen contents was much greater for the Wyodak coal even though the same upgrading temperature-time profile was followed in each case. These differences were in contrast to the data from the six coal liquids in Table 5 which were upgraded to about the same nitrogen content, and for which no clear trend in terms of yields of compounds of equal ring number as a function of coal rank was evident.

## CONCLUSIONS

On the whole, the compositional studies showed the coal liquids upgraded to about the same level of nitrogen removal were much more alike than different. There was some scatter in the compositions in terms of number of rings per molecule but no consistent trend with the rank of coal. Although the ranks of the coals used in this study covered the full range of practical interest for liquefaction, selection of additional coal samples covering an even greater range of coal rank might be of value in determining any possible dependence of coal liquid composition upon coal rank.

The extent of upgrading influenced the ring-number distribution for liquids from a given coal. More extensive upgrading produced larger amounts of low-ring-number compound types, due in part to greater removal of heteroatoms and to additional cracking of alkyl linkages between ring systems.

Finally, taking the results of this compositional study in conjunction with the results of the preparation of the coal liquids, liquids from lower-rank coals were more easily upgraded and somewhat more cracking to lower molecular weight components occurred at a given severity of upgrading.

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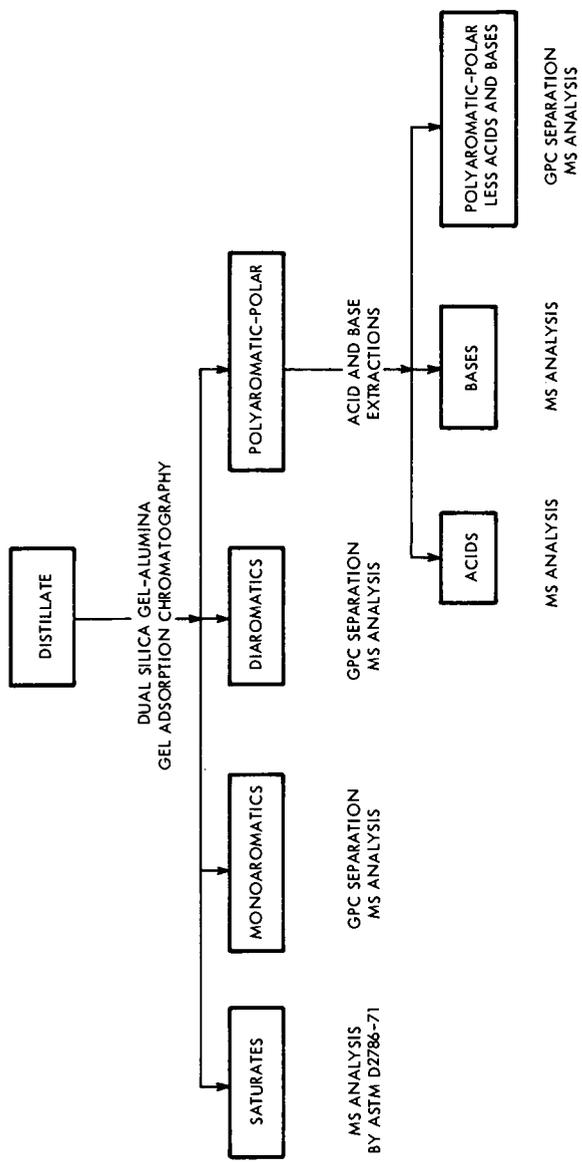


FIGURE 1. - Characterization scheme for 200-325° C, 325-425° C, and asphaltene-free 425-540° C coal liquid distillates.

TABLE I. - Analysis of coals, (weight percent)

Source Seam Rank	PA-WV Pittsburgh hvb A	Illinois No. 6 hvb B/C	W. KY -- hvb B/C	Montana (Colstrip) Rosebud subb. A	Wyoming (Wyodak) Lower subb. C	N. Dakota Beulah Std. II Lignite
Proximate analysis, (as received)						
Moisture.....	1.7	7.8	2.9	4.2	10.9	28.0
Volatile matter	35.9	34.2	38.4	34.4	39.5	31.0
Fixed carbon ..	55.1	51.4	48.8	48.5	42.7	33.3
Ash.....	7.3	6.6	9.9	12.9	6.9	7.7
Ultimate analysis, (moisture free)						
Hydrogen.....	5.1	4.9	4.9	4.3	4.8	4.5
Carbon.....	76.9	75.2	70.2	65.2	66.8	63.5
Nitrogen.....	1.5	1.6	1.4	0.6	1.0	0.9
Sulfur.....	1.6	1.5	4.3	1.8	0.5	1.3
Oxygen (diff) .	7.6	9.7	8.8	14.5	19.2	19.1
Ash.....	7.4	7.1	10.2	13.5	7.8	10.7

hvb B/C -- high volatile bituminous B or C  
subb. -- subbituminous

TABLE 2. - Properties of crude and upgraded coal liquids

Coal	Illinois No. 6		Illinois No. 6		W. Ky.	Colstrip	Wyodak		Lignite
	Pittsburgh	Run 1	Run 2	Run 2			Run 1	Run 2	
Nitrogen, wt-pct.....	0.44	1.45	1.10	1.28	0.64	0.48	0.43		
Oxygen, wt-pct.....	.59	-	1.34	2.09	1.61	1.08	1.55		
Sulfur, wt-pct.....	<.01	0.17	<0.01	0.02	<0.01	<0.01	<0.01		
				Crude Liquids					
				Upgraded Liquids					
Spec. gr. @ 60/60° F....	0.993	1.006	0.992	0.989	0.987	0.955	0.922	0.983	
SSU vis. @ 100° F.....	441	129	126	89	263	96	56	181	
SSU vis. @ 130° F.....	189	65	86	-	123	-	-	123	
Pour point, ° F.....	+5	<+5	<+5	<+5	+20	+70	+65	+45	
Carbon, wt-pct.....	89.2	90.1	88.6	88.2	88.1	88.9	88.0	89.0	
Hydrogen, wt-pct.....	10.8	9.9	10.3	10.7	10.6	11.0	11.9	10.8	
Sulfur, wt-pct.....	<0.01	0.03	0.02	0.02	<0.01	<0.01	<0.01	<0.01	
Nitrogen, wt-pct.....	.20	.444	.250	.287	.192	.095	.008	.250	
Oxygen, wt-pct.....	.28	.50	.19	.32	.34	.17	.04	.61	
Distillation, wt-pct:									
<200° C.....	10.0	12.3	11.4	16.6	11.5	13.9	18.3	12.3	
200°-325° C.....	21.7	27.3	27.9	26.1	21.5	26.7	30.3	24.0	
325°-425° C.....	20.3	20.7	22.5	22.8	21.1	21.3	19.1	20.7	
425°-540° C <sup>a</sup> .....	26.6	19.1	23.7	19.7	20.9	25.2	25.3	21.0	
>540° C <sup>b</sup> .....	16.2	6.4	7.5	9.3	18.4	11.2	6.3	15.0	
Asphaltenes, wt-pct ...	4.7	14.3	6.8	5.2	6.6	1.2	0.1	6.7	

1. Upgraded on same temperature-time program as Illinois No. 6 Run 2.

<sup>a</sup> Asphaltenes removed from >425° C residuum.

TABLE 3. - Distillation fractions of upgraded coal liquids

Coal	Pittsburgh	Illinois No. 6Run 1	Illinois No. 6Run 2	W. Ky.	Colstrip	Wyodak Run 1	Wyodak Run 2	Lignite
<200° C distillate:								
Spec. gr. ....	0.829	0.825	0.827	0.832	0.822	0.819	0.810	0.822
Sulfur, wt-pct. ....	<.01	.02	<.01	<.01	<.01	<.01	<.01	<.01
Nitrogen, wt-pct. ....	.003	.068	.001	.096	.023	.003	<.001	.007
200°-325° C:								
Spec. gr. ....	.915	.937	.926	.919	.918	.909	.897	.916
Sulfur, wt-pct. ....	<.01	.03	<.01	.02	<.01	<.01	<.01	<.01
Nitrogen, wt-pct. ....	.016	.102	.021	.062	.030	.012	.002	.104
325°-425° C:								
Spec. gr. ....	.983	1.018	1.001	.993	.982	.972	.952	.983
Sulfur, wt-pct. ....	<.01	0.01	<0.01	.02	<.01	<.01	<.01	<.01
Nitrogen, wt-pct. ....	.076	.396	.159	.200	.119	.072	.004	.304
425°-540° C <sup>1</sup> :								
Spec. gr. ....	1.045	1.079	1.061	1.049	1.032	1.026	1.001	1.032
Sulfur, wt-pct. ....	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Nitrogen, wt-pct. ....	.272	.594	.415	.415	.233	.176	.029	.412
>540° C resid <sup>1</sup> :								
Nitrogen, wt-pct. ....	.420	.709	.576	.565	.348	.275	.052	.518
Asphaltenes:								
Nitrogen, wt-pct. ....	.722	1.104	1.004	1.036	.579	.393	.194	.714
>425° C resid:								
Sulfur, wt-pct. ....	<0.01	.01	<.01	0.04	.02	<.01	<.01	<.01
Nitrogen, wt-pct. ....	.374	.829	.56	.573	.332	.219	.024	.501
Asphaltenes, wt-pct. ....	9.9	35.9	17.9	15.3	14.3	3.3	.4	15.7

<sup>1</sup> Asphaltene-free.

TABLE 4. - Fraction yields from adsorption chromatography and acid-base separations (weight percent)

Cool Liquid Fraction	Pittsburgh		Illinois No. 6 Run 1		Illinois No. 6 Run 2		Western Kentucky	
	Crude	Distillate	Crude	Distillate	Crude	Distillate	Crude	Distillate
200 - 325° C Distillate								
Saturates	11.91	54.9	7.92	29.0	11.75	42.1	11.82	45.3
Monoaromatics	7.77	35.8	15.02	55.0	13.81	49.5	12.01	46.0
Diaromatics	1.36	6.3	2.37	8.7	1.54	5.5	1.31	5.0
Polyaromatic-Polar	.36	1.7	2.00	7.3	.80	2.9	.65	2.5
Loss	.30	1.3	0.0	0.0	0.0	0.0	.31	1.2
325 - 425° C Distillate								
Saturates	5.44	26.8	2.11	10.2	3.87	17.2	4.92	21.6
Monoaromatics	10.17	50.1	6.91	33.4	9.70	43.1	9.46	41.5
Diaromatics	2.52	12.4	6.42	31.0	5.09	22.6	4.47	19.6
Polyaromatic-Polar	2.13	10.5	5.26	25.4	3.67	16.3	3.97	17.4
PAP Hydrocarbons	1.93	9.53	4.82	23.3	3.40	15.10	3.58	15.7
PAP Acids	.11	.56	.11	.51	.11	.47	.11	.47
PAP Bases	.05	.24	.14	.66	.10	.43	.09	.38
Loss	.08	.40	.19	.90	.23	1.10	.17	.80
425 - 540° C Distillate								
Saturates	2.26	8.8	.53	3.2	.80	3.6	2.17	11.8
Monoaromatics	5.76	22.4	1.30	7.9	1.80	8.1	2.36	12.8
Diaromatics	5.78	22.5	3.60	21.8	4.82	21.7	4.36	23.7
Polyaromatic-Polar	11.87	46.2	11.06	67.0	14.79	66.6	9.27	50.4
PAP Hydrocarbons	11.77	45.8	10.91	66.1	14.54	65.5	9.05	49.2
PAP Acids	.07	.29	.08	.50	.13	.59	.10	.55
PAP Bases	.05	.18	.07	.40	.10	.45	.04	.21
Loss	.01	.03	.01	.10	.01	-	.32	1.7

Cool Liquid Fraction	Calstrip		Wyodak Run 1		Wyodak Run 2		Lignite	
	Crude	Distillate	Crude	Distillate	Crude	Distillate	Crude	Distillate
200 - 325° C Distillate								
Saturates	10.75	50.0	15.06	56.4	22.73	75.0	11.71	48.8
Monoaromatics	8.92	41.5	10.12	37.9	6.24	20.6	10.13	42.2
Diaromatics	1.01	4.7	1.01	3.8	.51	1.7	1.02	4.2
Polyaromatic-Polar	.77	3.6	.51	1.9	.48	1.6	1.15	4.8
Loss	.05	.2	0.0	0.0	.34	1.1	0.0	0.0
325 - 425° C Distillate								
Saturates	5.84	27.7	6.99	32.8	11.77	61.6	5.22	25.2
Monoaromatics	8.52	40.4	8.48	39.8	4.22	22.1	7.87	38.0
Diaromatics	3.19	15.1	3.11	14.6	1.64	8.6	3.29	15.9
Polyaromatic-Polar	3.42	16.2	2.73	12.8	1.47	7.7	4.06	19.6
PAP Hydrocarbons	3.17	15.0	2.56	12.0	1.34	7.0	3.62	17.5
PAP Acids	.09	.41	.08	.37	.10	.52	.16	.76
PAP Bases	.08	.37	.03	.13	.01	.03	.18	.88
Loss	.21	1.0	.05	.25	.02	.15	.36	1.7
425 - 540° C Distillate								
Saturates	3.15	15.6	4.96	19.7	8.86	35.0	2.70	13.8
Monoaromatics	3.47	17.2	4.96	19.7	5.46	21.6	2.88	14.7
Diaromatics	4.38	21.7	4.79	19.0	3.74	14.8	3.49	17.8
Polyaromatic-Polar	9.19	45.5	10.48	41.6	7.24	28.6	10.29	52.5
PAP Hydrocarbons	8.79	43.5	10.21	40.5	6.93	27.4	10.09	51.5
PAP Acids	.12	.59	.09	.34	.14	.55	.11	.58
PAP Bases	.03	.14	.03	.11	.02	.06	.11	.54
Loss	.26	1.2	.16	.63	.15	.60	.22	1.1

TABLE 5. - Comparison of six coal liquids based on ring number, and asphaltene and residuum content (weight percent - total coal liquid basis)

No. of Rings \ Coal Liquid	Pittsburgh	Illinois No. 6 Run 2	Western Kentucky	Colstrip	Wyodak Run 1	Lignite
0	1.8	1.9	4.8	4.4	5.7	3.6
1	12.1	12.2	15.7	11.7	16.9	14.4
2	12.9	17.4	16.9	13.8	17.0	15.0
3	15.2	17.1	15.8	14.8	15.6	15.3
Sum 0 to 3	42.0	48.6	53.2	44.7	55.2	48.3
>4	32.2	31.9	27.9	27.6	29.6	24.5
Asphaltenes	4.7	6.8	5.2	6.6	1.2	6.7
>540° C Resid	16.2	7.5	9.3	18.4	11.2	15.0
Acids	.2	.2	.2	.2	.2	.3
Bases	.1	.2	.1	.1	.1	.3
Other <sup>1</sup>	4.6	4.8	4.1	2.4	2.5	4.9

<sup>1</sup> Includes unknowns, material not analyzed due to insufficient sample, and material lost in separations.

TABLE 6. - Ring number distributions for coal liquids produced from Illinois No. 6 and Wyodak coal using different processing conditions

No. of Rings \ Coal Liquid	Illinois No. 6 Run 1	Illinois No. 6 Run-2	Wyodak Run 1	Wyodak Run 2
0	2.1	1.9	5.7	6.2
1	11.4	12.2	16.9	21.6
2	16.1	17.4	17.0	20.1
3	17.3	17.1	15.6	16.9
>4	27.0	31.9	29.6	26.4
Asphaltenes	14.3	6.8	1.2	.1
>540° C Resid	6.4	7.5	11.2	6.3
Acids	.2	.2	.2	.2
Bases	.2	.2	.1	tr
Unknown and Losses	5.0	4.8	2.5	2.2