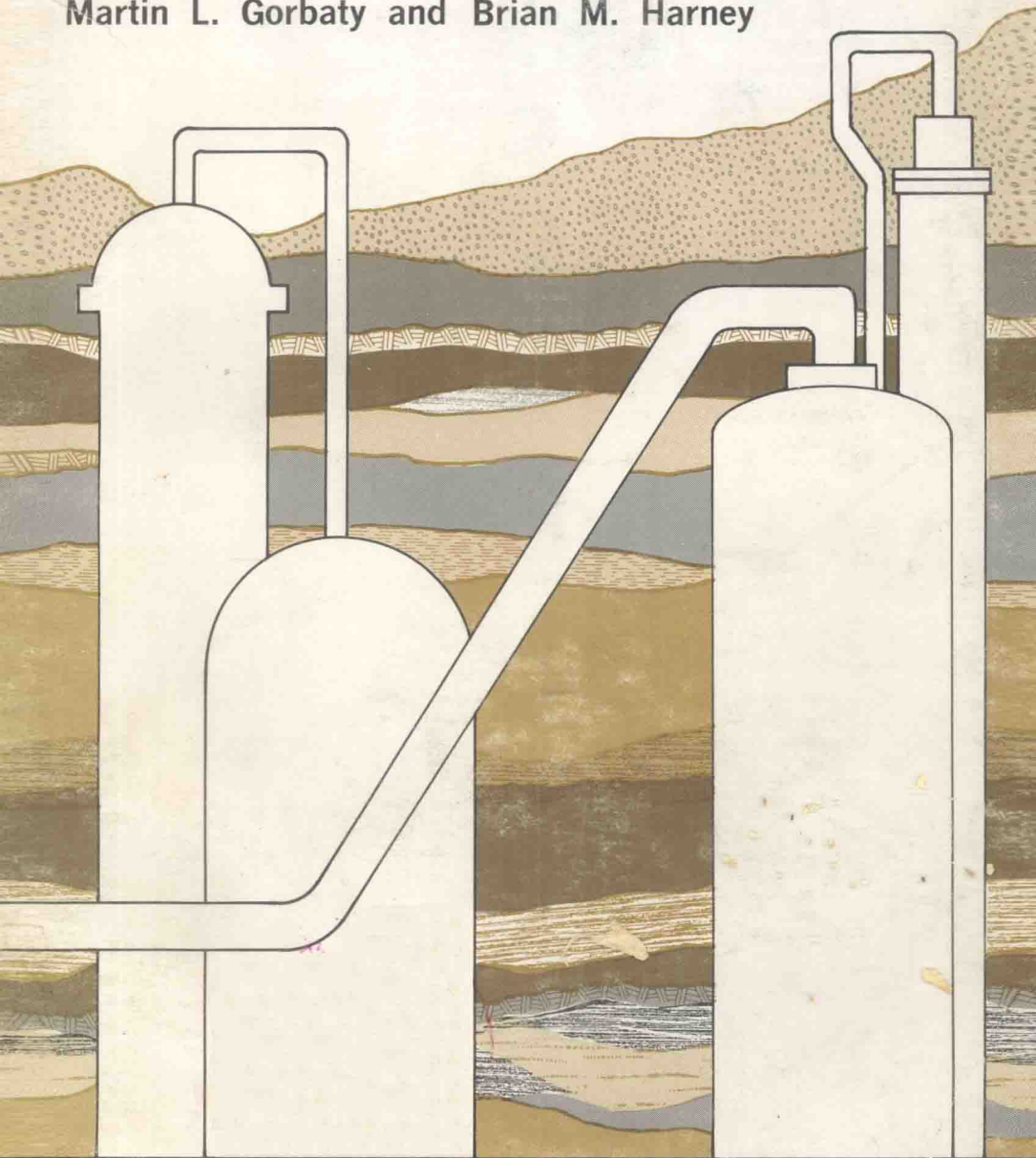# Refining of Synthetic Crudes

Martin L. Gorbaty and Brian M. Harney

Refining of Synthetic Crudes

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FOREWORD

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PREFACE

It now is recognized that increasing amounts of liquid fuels will have to be derived from sources other than petroleum, such as shale, tar sand bitumen, and coal. Generally, the quality of a fuel may be correlated with its atomic hydrogen-to-carbon (H/C) ratio. For example, methane has an atomic H/C ratio of 4 while crude petroleum and coal have ratios of about 1.7 and 0.8, respectively. Thus, the upgrading of coal to a liquid may be viewed as a hydrogen addition procedure. Also, concentrations of atoms other than carbon and hydrogen in coal-, shale-, and bitumen-derived liquids such as nitrogen, sulfur, oxygen, and trace inorganics, have to be lowered before they can be processed using current petroleum-refining technology.

The chapters in this book encapsulate a review of the state of the art in refining and upgrading of synthetic crudes, i.e., liquids derived from coal, oil shale, and tar sand bitumen. The first several chapters set the challenges in perspective by helping to define the molecular composition of liquids derived from coal. Following chapters discuss refining of shale oil and tar sand bitumen. The last chapters describe a number of different approaches directed toward the upgrading and refining of liquids derived from coal.

The chapters in this volume were presented originally at the national meeting of the American Chemical Society in August of 1977; many of them have been revised and updated. We wish to thank the contributing authors for their efforts.

It is our belief that this volume can serve as a source of information and a base from which future science and technology thrusts can emerge.

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Characterization Data for Syncrudes and Their Implication for Refining

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The character and hydrocarbon-type composition of several syncrudes have been investigated by adaptation of methods developed for heavier fractions of petroleum crude oils. The methods are reviewed briefly, and results are summarized for five coal liquids and a hydrotreated shale oil. Refining requirements for removal of heteroatoms, especially nitrogen, and conversion of polynuclear aromatics are discussed in relation to the composition of the syncrudes and the character of refined products to be expected. A preliminary report is given on the preparation of liquid samples from coals of widely different rank to permit more systematic correlation of hydrocarbon character with coal source in relation to refining.

Detailed characterization data are needed to realize the maximum potential of coal liquefaction products, shale oil, and tar sand liquids. Coal conversion processes (1, 2) now in development stages produce a wide variety of products that may or may not be directly amenable to current refining technology. The high nitrogen and aromatic contents of coal liquids have a profound effect on the selection of upgrading processes necessary to convert these materials to finished products. Nitrogen compounds, for example, are strong poisons for catalysts in both catalytic cracking and hydrocracking of heavy distillate feedstocks to produce refined liquid fuels. Aromatics, especially of the fused-ring type, lead to excessive yields of coke in either catalytic cracking or delayed-coking processes. Shale oil presents somewhat similar problems with high nitrogen but less aromatic contents.

Characterizations of synthetic liquids by different workers have been difficult to compare because of varied cut points, different methods of analysis, and incomplete reported data. More systematic data will be needed to characterize these liquids as alternative crude oils and to relate the source and liquefaction process to future requirements for production of refined liquid fuels.

The Bartlesville Energy Research Center (BERC) is working on these problems in order to supply needed characterization data, using systematic procedures (3) developed for characterizing distillable petroleum fractions. The procedures were developed through years of petroleum experience in cooperation with the American Petroleum Institute (API) and the Laramie Energy Research Center (LERC). Other reported methods of analysis (4, 5) are effective in providing similarly separated fractions, but the amount of detail obtained from these fractions by mass spectrometry and other techniques has been limited. In cases where mass spectral group-type methods (6, 7) based on high-ionizing voltage fragmentation patterns have been used for characterization, adequate qualitative and quantitative measurements needed in some applications are not possible. Mass spectrometry (MS) has been used (8) on fractions derived from minimal separation of components to provide excellent detail, but it lacks the capability to distinguish between certain hydrocarbon classes that overlap in mass spectral Z series distributions. The techniques developed and used at BERC take advantage of gradient elution chromatographic separation of distillates into saturate and aromatic concentrates. A given aromatic concentrate, e.g., the diaromatics, is further separated by molecular size by gel permeation chromatography (GPC). The GPC fractions are in turn analyzed by low-resolution, low-voltage MS and correlations established previously between the MS and GPC data. The hydrocarbon types in the given aromatic concentrate are recombined to give the composition in considerable detail. The BERC techniques have been applied with some slight modifications to a number of coal liquids (9, 10, 11, 12) available through development projects now in progress. Data so obtained are summarized and compared in this report according to aromatic and naphthene content in order to suggest to the processor the kinds of hydrocarbons he must cope with for upgrading. Preliminary data developed in a similar manner for a hydrotreated shale oil are included also.

A survey of the literature suggests that the types and complexity of hydrocarbons that can be obtained from a given coal can vary with the rank of coal. The character of the coal liquid can be affected also by liquefaction conditions, making useful comparison of liquids made in different processes, frequently at severe conditions, as well as from different coals, very difficult. In the absence of suitably comparable samples,

BERC has undertaken a systematic study which includes the preparation at mild reaction conditions of liquids from coals which vary widely in rank and quality. The liquids so obtained will be characterized and analyzed for hydrocarbon type by BERC procedures, and the data will be used to supplement the characterization studies in progress on other syncrudes. A preliminary report will be made on the progress of this work to date.

Experimental

The separation and characterization scheme used to generate appropriate compositional data has been discussed elsewhere (3, 9, 10, 11, 12) and only a brief review will be given. First, the full-boiling-range product is cut into distillates having boiling ranges approximating < 200°C, 200–370°C, 370–540°C, and 540°C + residuum. More recent modifications (to meet the expected needs of refining studies) provide distillates of more precise determination with boiling ranges of ~ < 200°C, 200–325°C, 325–425°C, 425–540°C, and 540°C + residuum. Those distillates boiling below 200°C are separated by use of high-pressure liquid chromatography (HPLC) and the fractions are analyzed by MS. The distillates boiling above 200°C are separated by liquid–solid chromatography into saturate and aromatic concentrates, and the aromatic concentrates are further separated by GPC. Acid and base removal is usually accomplished by chemical extraction of the polyaromatic–polar concentrates obtained from the liquid–solid chromatographic separation. Fractions produced in all of these separations are analyzed by MS (and GPC correlations), UV fluorescence spectrometry, and nuclear magnetic resonance (NMR) spectrometry to determine the qualitative and quantitative distributions of compound types found in the fractions. Data derived from these techniques can be so detailed as to provide quantitative carbon number distributions for identified compound types, or the detailed data can be recombined in various forms to provide some of the most accurate and reliable summary compositional data available. Summary-type data will be discussed in this report.

For the batch preparation of coal liquids, BERC is using a 2-L magnetically stirred batch autoclave supplied by Autoclave Engineers. The experimental procedures being used are adaptations of those used at the Pittsburgh Energy Research Center (PERC) and by other research groups, and they are not intended to represent development of a practical process. Rather, a liquid which represents most of the hydrocarbons contained in the source coal is sought, with a minimum of cracking of the original hydrocarbons. Hydrogenation conditions generally have been at 2,500–2,700 psig, with the temperature being increased slowly from approximately 300°–400°C over a period of several hours. Hydrogen was added periodically to maintain total pressure. For two coals processed to date, hydrogen consumption started at or below 325°C. A typical charge to the autoclave consisted of 375 g of coal slurried with 500 g of solvent (Tetralin or filtered liquid product recycled in successive runs), 40–60 g of crushed Cyanamid HDS-3A NiMo catalyst, and 1–2 g of sulfur (to help

sulfide the catalyst). The coal–solvent slurry, catalyst, and sulfur were introduced through a charging port in the autoclave cover. Product was removed through the same port by means of a dip-tube and vacuum system. Benzene was charged to the autoclave at the end of a series of runs to remove residual product and solids. When sufficient inventory of coal-derived liquid was accumulated, a final upgrading run was made with presulfided catalyst to yield a liquid product which contained less than about 0.4 wt % nitrogen.

Tetralin was used as a hydrogen-donor solvent in the initial stages of liquefaction. For the first coal processed (Illinois No. 6 bituminous), the Tetralin–naphthalene was removed by distillation after sufficient liquid inventory was accumulated to provide coal liquid as the solvent for further batches. The bottoms from the distillation were solid at ambient temperature and were given an upgrading treatment before use as a recycle solvent.

Results and Discussion

Some of the data to be discussed were reported earlier (13, 14) in somewhat different form and are included with data more recently acquired on other alternate fuels to provide comparative characteristics of all the materials examined.

Coal Liquids and Shale Oil. Some properties of five coal liquids and a hydrotreated shale oil are given in Table I, including sulfur, nitrogen, acid and base contents, and distributions by boiling range. Ring-number

Table I.   Some Physical and Chemical Properties

Liquid	Sulfur % Wt	Nitrogen % Wt	Acids % Wt
COED[b] Utah Coal	0.05	0.48	8.17
COED[c] Western Kentucky Coal	0.08	0.23	3.11
Synthoil[d] West Virginia Coal	0.42	0.79	10.40
H–Coal Fuel Oil[f] Illinois No. 6 Coal	0.21	0.44	14.69
H–Coal Syncrude[g] Illinois No. 6 Coal	0.27	0.63	13.81
Hydrotreated Shale Oil	< 0.01	0.04	—

[a] Approximate boiling ranges.
[b] Approximately 10% of this material was not analyzed. 1.50% Heteroatomic compounds determined in addition to acids and bases extracted.
[c] Approximately 8% of this material was not analyzed. 0.3% Heteroatomic compounds determined in addition to acids and bases extracted.

distributions of the major hydrocarbon classes found in each coal liquid and the hydrotreated shale oil are shown in Tables II, III, IV, and V. While these are only summary data, they represent cumulative totals derived from more detailed data and therefore should be considerably more accurate than those acquired by more gross compositional methods.

Endpoints for the H–Coal syncrude and the two Char Oil Energy Development (COED) syncrudes were determined in earlier work (9, 10, 12) to be in the range of 510°–550°C, but the H–Coal fuel oil endpoint (12) was determined to be lower at approximately 450°–460°C. In Table I, the Synthoil product shows 25.7% residuum boiling above 540°C. These differences in cut points in the liquids make comparisons of data difficult, since the bottoms of the H–Coal materials were not included and the Synthoil residuum was not analyzed and cannot be included in any comparison. However, the nitrogen contents of the coal liquids given in Table I are indicative of the degree of upgrading already done (since the nitrogen concentrations in the coals are high) and can be used as indicators of the degree of upgrading needed to turn these liquids into refined products. For instance, the nitrogen value shown for Synthoil would indicate that this material requires considerably more upgrading than would be necessary for the other liquids shown if part or all of it were to be used for transportation fuels. The larger ring-number systems shown for the Synthoil composite in Tables IV and V tend to substantiate this

for Five Coal Liquids and a Hydrotreated Shale Oil

	Distillate			
Bases % Wt	< 200°C % Wt	200–370°C % Wt	370–540°C % Wt	540°C Resid % Wt
1.30	13.3	45.4	40.3	—
1.05	21.0	54.2	24.2	—
2.75	4.4	42.6	27.3	25.7[e]
1.29	35.5	63.8		—
2.73	34.6	65.4		—
—	24.3	48.4	24.5	—

[d] Approximately 4% (plus resid) of this material was not analyzed. 2.75% Heteroatomic compounds determined in addition to acids and bases extracted.
[e] Not analyzed.
[f] Only overhead products analyzed. Material above 200°C boiled up to 460°C.
[g] Only overhead products analyzed. Material above 200°C boiled up to 510°C.

Table II.   Ring-Number Distributions[a] for Saturate Concentrates Derived from Five Coal Liquids and a Hydrotreated Shale Oil

Total Rings	COED UT % Wt	COED W. KY % Wt	Synthoil WV % Wt	H–Coal Fuel Oil IL No. 6 % Wt	H–Coal Syncrude IL No. 6 % Wt	Hydro- treated Shale Oil % Wt
0	8.73	4.26	1.87	1.73	1.42	21.4
1	2.48	4.34	2.62	2.04	1.14	13.3
2	2.15	3.89	1.76	1.90	1.05	8.5
3	2.20	3.16	1.75	1.15	0.62	5.4
4	2.25	2.06	1.15	0.60	0.45	2.7
5	3.96	0.93	0.31	—	—	2.2
6	1.30	0.44	—	—	—	—
< 200°C Sat.	7.05	14.34	1.19	14.20	15.99	22.8
Totals	30.12	33.42	10.65	21.62	20.67	76.3

[a] Determined from distillates in Table I.

observation. This suggests that less carbon–carbon bond cracking occurred in production of the Synthoil liquid, although the coal rank could have some influence. From the distributions shown in Tables III, IV, and V for the coal liquids, aromatics having four or more total rings amount to 24.1, 21.6, 27.0, 8.1, and 18.8% of the total liquid for the Utah COED, Western Kentucky COED, Synthoil, H–Coal fuel oil, and H–Coal syncrude, respectively. This suggests that the Synthoil product contains more of the higher ring systems, but the implication may not be valid since the residuum fractions of the liquids were not analyzed. The shale oil contains only 22.2% total aromatics and these contain only 1–3 rings. Most of the

Table III.   Ring-Number Distributions[a] for Monoaromatic Concentrates Derived from Five Coal Liquids and a Hydrotreated Shale Oil

Total Rings	COED UT % Wt	COED W. KY % Wt	Synthoil WV % Wt	H–Coal Fuel Oil IL No. 6 % Wt	H–Coal Syncrude IL No. 6 % Wt	Hydro- treated Shale Oil % Wt
1	1.75	2.04	0.30	1.15	0.96	1.9
2	5.10	8.22	0.95	7.85	4.07	4.4
3	4.79	9.23	4.84	5.02	2.12	3.5
4	3.12	5.67	4.85	0.61	0.35	2.1
5	1.74	2.42	1.57	0.11	0.10	1.0
6	0.56	0.88	0.37	0.07	0.04	0.3
7	0.16	0.32	0.10	—	0.02	0.2
8	0.05	0.11	0.04	—	0.02	trace
< 200°C Monoaromatics	2.79	4.75	1.21	12.18	10.85	4.0
Totals	20.06	33.64	14.23	26.99	18.53	17.4

[a] Determined from distillates in Table I.

Table IV. Ring-Number Distributions^a for Diaromatic Concentrates Derived from Five Coal Liquids and a Hydrotreated Shale Oil

Total Rings	COED UT % Wt	COED W. KY % Wt	Synthoil WV % Wt	H–Coal Fuel Oil IL No. 6 % Wt	H–Coal Syncrude IL No. 6 % Wt	Hydro-treated Shale Oil % Wt
2	4.13	2.14	3.26	6.49	4.15	1.1
3	4.32	3.83	4.62	5.13	3.40	1.1
4	2.91	3.15	3.19	1.47	1.74	0.6
5	1.81	1.94	1.56	0.43	0.79	0.3
6	0.77	1.04	1.18	0.04	0.31	0.1
7	0.34	0.48	0.99	0.03	0.22	0.1
8	0.13	0.16	0.36	trace	0.04	trace
9	—	0.04	0.16	—	0.04	trace
10	—	—	0.05	—	0.02	trace
11	—	—	0.02	—	—	trace
< 200°C Diaromatics	—	—	0.14	trace	0.06	—
Totals	14.41	12.78	15.53	13.59	10.77	3.3

^{a} Determined from distillates in Table I.

coal liquids are highly aromatic (saturates ranged from 11–33% of the liquid analyzed) and contain significant amounts of heteroatomic compound types such as acids (3.1–14.7%) and bases (1.1–2.8%). On the other hand, the hydrotreated shale oil is very high in saturates (76%). Furthermore, the saturate fractions of the coal liquids boiling above 200°C contain approximately 63–80% naphthenes, part of which would be aromatics in the original coal.

Table V. Ring-Number Distributions^a for Polyaromatic Concentrates Derived from Five Coal Liquids and a Hydrotreated Shale Oil

Total Rings	COED UT % Wt	COED W. KY % Wt	Synthoil WV % Wt	H–Coal Fuel Oil IL No. 6 % Wt	H–Coal Syncrude IL No. 6 % Wt	Hydro-treated Shale Oil % Wt
3	1.94	1.48	1.03	5.81	3.76	0.4
4	3.65	2.11	2.93	3.84	4.15	0.5
5	3.63	1.62	2.21	0.97	3.69	0.3
6	3.58	1.25	2.18	0.32	3.84	0.2
7	1.67	1.24	2.28	0.07	0.81	0.1
8	0.01	0.18	2.04	0.07	1.41	trace
9	—	0.03	1.05	0.03	0.67	trace
10	—	—	0.70	—	0.39	—
11	—	—	0.19	—	0.19	—
Totals	14.48	7.91	14.61	11.11	18.91	1.5

^{a} Determined from distillates in Table I.

The ring-number tabulations in Tables II, III, IV, and V are satisfactory for a simplified summary of composition and comparison. The typical Co–Mo or Ni–Mo catalyst commonly used for upgrading will saturate many of the multiple aromatic rings, depending upon severity and activity, but frequently not the last ring of a condensed-ring polyaromatic. Thus, the total number of rings is a measure of the complexity of the hydrocarbon structure. As noted earlier, more detailed data on the distribution of hydrocarbon types in these liquids are available when needed.

Batch Preparation of Coal Liquids at BERC. Two coal liquids had been prepared at the time this report was written, but preliminary analytical data for only one were available and these data are shown in Table VI for the coal liquid prepared from Illinois No. 6 coal. Also shown in the table are typical analytical data for the original coal as supplied by E. L. Obermiller of Conoco Coal Development Co. Mr. Obermiller also provided samples of four other coals used in this program. A sixth sample (lignite) was supplied by J. E. Schiller of the Grand Forks Energy Research Center. While the data in Table VI are only preliminary, sufficient experience has been gained with the batch preparation of small samples of liquids at BERC to suggest that the original goal can be realized to establish correlations of the complexity of the hydrocarbons derived from a given coal with its rank or quality. The three distillates prepared from the Illinois No. 6 coal appear to be quite stable; the less than 200°C distillate is completely clear of any coloration and the 200°–325°C distillate is only slightly yellow in color after about three weeks on the shelf. Asphaltenes will be removed from the 425°C residuum and

Table VI. **Preliminary Data for Coal Liquid Prepared from Illinois No. 6 Coal and Typical Properties of Original Coal**

Upgraded Coal Liquid		*Typical Analyses of Coal, % Wt*	
Specific Gravity	1.006	Moisture	7
SSU Viscosity @ 100°F	129 sec	Volatile Matter	38.15[a]
SSU Viscosity @ 130°F	65 sec	Ash	6.75[a]
Sulfur, % Wt	0.03	Sulfur	1.20[a]
Nitrogen, % Wt	0.444	Nitrogen	1
Carbon, % Wt	90.1	Carbon	69
Hydrogen, % Wt	9.88	Hydrogen	5
Distillates Prepared:			
< 200°C, % Wt	12.3		
200°–325°C, % Wt	27.3		
325°–425°C, % Wt	20.7		
> 425°C Resid, % Wt	39.7		

[a] Actual analysis.

the asphaltene-free residuum will be vacuum flashed to 540°C to provide a heavier distillate fraction. These distillates are suitable for and will be processed through our separation and characterization scheme to establish hydrocarbon types and quantities.

Applications to Refining

The first essential step in refining these synthetic crude oils is severe catalytic hydrogenation to remove most of the sulfur, nitrogen, and oxygen and to convert at least part of the heavy ends to lower-boiling distillates for further refining. The removal of nitrogen is more difficult than that of sulfur or oxygen and is, therefore, crucial to the early stages of refining. The hydrocarbon character of shale oil already resembles that of many petroleum crude oils, as indicated by its hydrogen/carbon atom ratio of approximately 1.6, except that it has been cracked thermally and typically contains 2.0% nitrogen. The crude shale oil can be subjected to a preliminary delayed coking in order to convert residuum and decrease the required severity of subsequent hydrogenation to remove nitrogen. However, this would not be suitable for highly aromatic coal liquids because the heavy ends would yield little but coke and gas. Another requirement in the initial upgrading by hydrogenation is coping with residual solids in the synthetic crude oil, which thus far has been present typically at concentrations of 0.1% or more of the synthetic crude oil from coal, or about ten times as much as in petroleum residua being charged to hydrodesulfurization processes.

However, the really limiting factor in the refining of coal liquids is attributable to their highly aromatic character as shown in Tables II, III, IV, and V in particular to the presence of substantial concentrations of condensed-ring polyaromatics such as chrysenes, pyrenes, etc. Without going into detail, each condensed aromatic ring must be saturated by hydrogenation before it can be cracked to produce lower-boiling distillate products. Thus, hydrogen consumption increases by large increments as the number of aromatic rings in the condensed-ring hydrocarbon increases, and more severe process conditions are required. Furthermore, the difficulty of such conversion increases sharply for the more highly condensed aromatics of the pyrene type, which are slow to react and tend to cover catalysts and prevent conversion of other heavy components (15). Nitrogen is incorporated in these essentially aromatic ring structures, which explains why it is more difficult to remove than sulfur or oxygen. In compounds with a large number of condensed rings, as in acridine and higher homologs, the nitrogen-containing ring is still less accessible for saturation and cracking for removal of the nitrogen. Aromatic rings are extremely resistant to the cracking, witness hydrodealkyla-

tion process which converts alkylbenzenes to benzene at temperatures of
1150°–1300°F (621°–704°C) with no loss of rings. Sulfur and oxygen,
being divalent, are excluded from such stable benzene-like rings and are
in compounds from which they are more easily removed by hydrogena-
tion. Coal liquids produced by noncatalytic liquefaction, even in the
presence of hydrogen, contain essentially the same 1.0–1.4% nitrogen as
the original. In fact, the degree to which a synthetic crude oil from coal
has been upgraded by catalytic hydrogenation can be fairly well gaged
by the nitrogen content—typically about 0.5% as illustrated in Table I
for relatively moderate hydrotreatment to 0.05% for severe hydrotreat-
ment. Removal of nitrogen from the heavier ends of synthetic crude oils is
required for hydrocracking to lighter distillates and gasoline, although
petroleum hydrocracking processes are reported to tolerate as much as
0.2–0.3% nitrogen. The two factors of (a) condensed-ring aromatic
structures which (b) contain nitrogen account largely for the reported
difficulty of conversion, even by hydrogenation and hydrocracking, of
the heavy ends of coal liquids (16).

The only detailed refining study reported (16) for a syncrude from
coal, using present-day catalytic processes, was made on a severely hydro-
treated liquid from the COED pyrolysis process. This feedstock may
not be fully representative of syncrudes which can be produced by direct
hydrogenation of the coal to give much greater percentage yields of
liquids, but the difficulties encountered in converting the heavy ends
boiling above about 750°F (399°C) to lighter distillate products, for rea-
sons which have already been discussed, suggest that such heavy ends
may be blended better into heavy fuel oil than to add excessively to the
cost of refining.

The lighter distillates and naphtha from oil shale and coal can be
refined by processes used for petroleum, except that more severe hydro-
treatment will be required to remove nitrogen and the other nonhydro-
carbon impurities that poison catalysts and cause product instabiltiy.

Once the synthetic crude oils from coal and oil shale have been
upgraded and the heavy ends converted to lighter distillates, further
refining by existing processes need not be covered in detail except to note
the essential character of the products. The paraffinic syncrude from oil
shale yields middle distillates which are excellent jet and diesel fuel
stocks. The principal requirements are removal of nitrogen to the extent
necessary for good thermal stability of the fuels and adjustment of cut
points to meet required pour or freeze points, limited by the presence of
waxy straight-chain paraffins. The heavy naphtha from shale oil can be
further hydrotreated and catalytically reformed to acceptable octane
number, but with considerable loss of volume because of the only mod-
erate content of cyclic hydrocarbons, typically 45–50%. On the other