

SHORT CONTACT TIME COAL LIQUEFACTION
1. Techniques & Product Distributions*

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INTRODUCTION

In our investigation of the nature and origin of asphaltenes in processed coals we have concentrated on the chemistry and kinetics of reactions accompanying the dissolution of coals. These investigations have been facilitated by the development of two new techniques for the study of coal liquefaction reactions and their products. First, we have designed and operated a 300-ml batch autoclave system capable of such rapid injection, sampling, and quenching, that contact times as short as 15 seconds can be achieved. Second, we have developed a rapid but detailed high pressure liquid chromatographic procedure for classifying coal liquid products into ten fractions of known chemical functionality.

In this report we will describe the design and operation of the short contact time reactor and show its capabilities, and the types of information we have obtained, by giving the details of several runs. We will then present some of our early key findings on the chemistry and kinetics of the solvent-refined coal process. The development and use of the fractionation procedure will be presented elsewhere (1).

The type of coal liquefaction method being studied is exemplified by the solvent-refined coal process in which a slurry of coal in a solvent derived from the coal in the process is passed through a reactor in which it is heated in the presence of H_2 under conditions sufficient to liquefy the coal and partially desulfurize the products. The unreacted residue is removed and the remainder distilled to produce a variety of products, including the solvent which is recycled.

Coal Conversions: Techniques

Coal Preparations - Coals obtained for study in this project have been stored in wet lump form in an inert atmosphere. All coal preparations were conducted under a subcontract with the Department of Aerospace and Mechanical Sciences, Princeton University, project titled "Mechanisms of Coal Dissolution and Asphaltene Formation," M. Summerfield, principal investigator. Prior to grinding each sample was placed in an oven at 125°C for 1 hour in a CO_2 atmosphere and allowed to cool as the oven cooled. Fragments over 0.5 cm diameter (for which it was assumed no prior oxidation had occurred) were transferred to a water-cooled rotary cutter and ground in a CO_2 atmosphere for from 0.25 to 2 min. All subsequent operations (including screening and slurry formulation) were performed in an inert atmosphere.

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Portions in various size ranges were obtained by passing the coal through a series of standard screens shaken by an electric screen vibrator for 3 hours. Agglomeration was prevented by placing rubber stoppers on each screen. Size distributions were determined from the yield of coal on each screen.

The coals used in the runs described in this paper were West Kentucky 9,14 (a high volatile B bituminous coal) and Wyodak (a sub-bituminous coal). Proximate and ultimate analyses are given in Table 1. Particles ranged in size from $\sim 1 \mu\text{m}$ to $625 \mu\text{m}$ (the majority being $45\text{--}150 \mu\text{m}$).

Conversion Apparatus and Procedure - A schematic diagram of the high pressure autoclave system is given in Figure 1. The autoclave was a conventional 300-ml stainless steel autoclave manufactured by Autoclave Engineering, Erie, Pennsylvania (#MAWP-5400) with fixed head, removable lower unit, and magnedrive stirrer; all external connections were through the head. Connections included 1/4" sample injection line, a 1/8" liquid sample withdrawal line fitted with a metal filter, and a 1/16" stainless steel shielded dual thermocouple for reading and controlling temperatures, all below the liquid level. Also attached were gas inlet and gas sampling lines above the liquid level. The stirrer drive was a Cole Palmer Constant Speed and Torque Control unit which allowed recording of motor torque during a run. The stirring blade was spiral shaped and as large as possible to produce maximum agitation; viscosity was monitored by measuring the stirrer torque. A cooling coil was mounted inside the vessel in direct contact with the contents. We have incorporated a cooling water reservoir pressured to 200 psi with N_2 to give a high coolant flow rate. The injection system consisted of a barrel with a floating piston insert having "O" ring seals.

To initiate a run a small amount of solvent was pumped up into this barrel from below, then slurry was forced in from below by means of a large metal syringe, followed by a little more solvent so that no slurry remained in the valve. With this sequence solvent was injected after the slurry to flush all the coal into the vessel. Above the piston was a reservoir of squalane (easily detected by our analytical system in case leaks occurred) which was pressured with N_2 just before the injection. All lines throughout the system were fitted with appropriate vents, rupture discs, drop-out pots, check valves, filters, pressure gauges, etc.

In a typical run, 60 g solvent was placed in the vessel, the unit was sealed, flushed twice with H_2 , pressure-tested with H_2 at the intended reaction pressure, and vented to 200 psi. Heating with an electric heater and stirring (1200 rpm) were then initiated. As the desired operating temperature was approached (typically after about 1 hr.) the injection system was sequentially loaded with 15 g solvent, 40 g 1:1 solvent coal slurry, and 5 g solvent. The time between injector loading and injection was kept as short as possible to minimize slurry settling. If the run was to be very short, the H_2 pressure was increased so that the desired pressure was reached after injection, otherwise it was adjusted after injection.

When the temperature lined out 10°C above the desired temperature, a solvent sample was taken from the vessel to establish the pre-injection solvent composition. The injector was pressured to ~ 100 psi

above the vessel and the contents forced into the vessel in about 1 sec. The temperature drop under these conditions, depending upon the temperature and the exact amounts of material in the vessel and injector, was generally 50-115°C. Recovery to the desired temperature required about .5-1 min; reaction time was assumed to start when the temperature had recovered to 10°C below the desired reaction temperature. A typical time-temperature profile is shown in Figure 2. Liquid and gas samples were occasionally taken during a run; pressure was adjusted if significant drops occurred during sampling.

To end a run, the heater was removed and simultaneously cooling water flow was started. The temperature typically dropped about 100°C within 10 sec. The temperature was adjusted to 125°C and H₂ was added to raise the pressure to ~1200 psi. The stirring speed was then reduced to ~10 RPM and the gas vented over a period of 5 min. through a dry ice trap into an 18 l evacuated reservoir. The trap then contained water, some solvent, and some light coal products. Water was determined volumetrically. The light components were distinguished from solvent by gas chromatography. The gas reservoir pressure was noted and a sample taken for mass spectroscopy.

The autoclave vessel was then cooled to room temperature and opened. The contents were removed and solids washed out with pyridine; solids adhering to surfaces were loosened with a brush. The mixture of solids and liquid was passed through a Soxhlet thimble; the thimble contents were washed with pyridine which was then combined with the liquid portion. The solids were then extracted with pyridine for 16 hrs. and that pyridine added to the other liquids. The residue was dried at 125°C in a vacuum oven and stored under Ar until further characterization was carried out.

The pyridine was removed from the combined liquids with a rotary evaporator. The resulting mixture of solvent, SRC, and light organic coal products was subjected to a vacuum distillation at 12 mm pressure. A first cut was taken at 130°C (true boiling point 260°C). If the solvent was our synthetic solvent, this was in the first cut; the boiling point of 2-methylnaphthalene is 240°C. A second cut was taken at 200°C (true boiling point 343°C, 650°F). The residue was the SRC. Within each distillate, product was distinguished from solvent by gas chromatography. The second cut was usually so small that it had to be removed from the condenser with THF.

The injector system was then flushed with THF to remove uninjected coal which was dried and weighed to complete the mass balance.

Temperature and pressure were recorded throughout the autoclave run on a strip chart.

All slurries for conversions were about 1:1 coal-to-solvent by weight and were made up at least 24 hrs. in advance of a run to insure equilibration.

We have recently converted to a new injection system using the same barrel but no piston. Instead, the slurry was driven directly into the autoclave with H₂. Injections were found to be easier and more complete with this method. The slurry was simply poured into the injector, a solvent layer was added, pressure was applied, and the injection made. Washing was as described above.

The products of a run were separated by the work-up into the following fractions: H_2S , H_2O , CO , CO_2 , CH_4 , C_2-C_5 (fully-identified by mass spec), $C_6-257^\circ F$ (the contents of the trap, corrected for H_2O and solvent), $6-257-650^\circ F$ (the overhead from the distillation, corrected for solvent), SRC (pyridine-soluble, $650^\circ F^+$), and residue (pyridine-insoluble). Elemental analyses of the SRC and residue were determined by Galbraith Laboratories, Knoxville, Tennessee.

Synthetic Recycle Solvent - In the choice of a reaction solvent, three factors were considered. First, the coal products must be distinguishable from the solvent. Second, the solvent must have, as nearly as possible, properties similar to true recycle solvents in terms of chemical functionality, H-transfer properties, and ability to solubilize products. Third, the solvent must provide a means by which the extent of H-transfer and the occurrence of thermal or catalyzed side reactions (such as cracking and isomerization) could be evaluated.

The following synthetic solvent composition was chosen: ~2% 4-picoline; ~17% p-cresol; ~43% tetralin; ~38% 2-methylnaphthalene. This mixture has the proper amounts of basic nitrogen, phenol, and hydroaromatics. It is more aromatic and lower boiling than a real recycle solvent.

A paper is in preparation describing in detail the reactions of this solvent mixture under coal liquefaction conditions, with and without coal or coal products present. Much has been learned about the chemistry of the SRC process from this system. A brief discussion will be presented here to explain the hydrogen consumption data given in the next section.

By following the solvent composition, we can measure the thermal background reactions by observing the isomerization of tetralin to methylindane. This is not affected by the presence of coal. The hydrogen-transfer rates are affected by coal, possibly by catalysis by coal minerals. This is measured by observing the formation of methyltetralin from methylnaphthalene (by transfer from tetralin to form naphthalene). Correction can be made for the reaction in the absence of coal, either by calculations based on methylindane formation or by comparison to blank runs with solvent alone. Hydrogen consumption from the solvent by coal or its products is determined by observing the formation of naphthalene and correcting for the hydrogen only transferred to make methyltetralin, and the naphthalene produced through thermal reactions. The solvent: coal ratios are high, and temperatures and H-consumption low. Our results are therefore not affected by solvent hydrogen depletion or an approach to thermodynamic equilibria.

Coal Conversions: Product Distributions

Three key factors in coal liquefaction processes for which data and understanding are particularly important are: 1. the rate of conversion to soluble products and the maximum yield obtainable, 2. sulfur removal, and 3. the hydrogen consumption required to achieve a high yield of low sulfur product. [Our results on the chemical nature of the SRC's will be presented elsewhere (1).]

In order to derive the kinetics of the conversion of coal to soluble

form it is necessary to develop understanding of reactions which occur very rapidly during the initial transformations of the coal. Most commercial operations or pilot studies now being operated can provide data only of products that result from extended reactions of coal and some process optimizations may not be observable under these conditions. It has been reported (2) that coal can be dissolved rather rapidly at temperatures $\geq 800^\circ\text{F}$. The shortest times that were defined were 2-5 min. It was also noted that the yield of soluble product could actually decline on extended reaction at high temperature due to char formation.

The emphasis of the work described in this report was on developing understanding of the chemical nature of the initially-soluble coal products and mechanisms by which they are formed and interconverted. Our initial efforts have focused on conversion of coal under mild conditions ($\sim 800^\circ\text{F}$), where the rate of formation of char is rather low. This will provide the background information needed to study coal conversion at higher temperatures (charring conditions) which is one of the objectives of future work.

A series of conversions of West Kentucky and Wyodak coals was carried out at 800°F , 1000-1300 psig H_2 in our synthetic solvent. The balances of these runs are shown in Table 2. Product composition for these runs is summarized below.

<u>Kentucky Coal</u>					
Run No.	Ext	10.00	9.00	7.00	12.00
Time (mins.)	.00	.50	1.30	40.00	417.00
% Sol.	28.00	50.00	78.20	92.52	96.10
SRC Yield	28.00	46.80	76.10	80.11	61.05
% O SRC	9.47	6.68	7.25	5.15	2.93
% S SRC	2.17	1.51	1.40	1.31	.63
H_2 Consp.	.00	.13	.34	.89	1.59

<u>Wyodak Coal</u>			
Run No.	Ext	19.00	31.00
Time (mins.)	.00	1.30	137.50
% Sol.	11.50	45.97	91.52
SRC Yield	11.50	38.53	70.03
% O SRC	12.20	11.75	5.08
% S SRC	.70	.53	.37
H_2 Consp.	.00	.41	2.46

It can be seen from the above data that conversion of the coal to $>90\%$ soluble form occurs very rapidly (we estimate ~ 3 min for West Kentucky and ~ 20 min for Wyodak).

In order to achieve $>90\%$ solubility, the oxygen content of the West Kentucky SRC had only to be reduced to about 6 wt. %. The oxygen content systematically became lower with increasing contact time and there appear to be two forms of oxygen which are kinetically distinct. One form ($\sim 40\%$ of all the oxygen) is very readily lost. The other form requires more vigorous treatment to eliminate it. This is shown in Figure 3, where the log of the % oxygen remaining in West Kentucky coal products is plotted against time (this assumes first-order dependence).

The organic sulfur content of SRC appears to be linearly related to the oxygen content of SRC. This is shown in Figure 4. Again, 40% of the organic sulfur is easily removed, but the rest is more difficult. Thus, merely dissolving the coal is not sufficient to lower the sulfur content to acceptable levels, at least not for West Kentucky 9,14.

The nitrogen content of all SRC's was not substantially affected by solvent refining of the coal. Thus, catalytic processing may be needed to lower nitrogen content, if this is required for end use.

The time-yield behavior for Wyodak and West Kentucky coals appears to be different. There may, however, be a common feature in that the maximum yield of SRC appears to be co-incident with the minimum time required for >90% conversion to soluble form. This is illustrated in Figures 5 and 6. It can be seen that the yield of SRC from West Kentucky 9,14 coal continually drops after 5-10 min. of reaction. This loss in yield is not due to char formation, however, but is the result of converting SRC to solvent and lighter products.

As can be seen in Figure 6, one significant kinetic parameter that can be used as an alternative to time is the percent O converted to CO₂ and H₂O. This parameter is not meaningful when comparing two different coals, however, as the amount and chemical type of oxygen can vary widely.

The hydrogen consumption observed for a given coal generally increases with increasing degree of solubilization. However, the yield of SRC vs. hydrogen consumption goes through a maximum. This is shown in Figure 7. Solvent yields become appreciable only with high hydrogen consumption and methane formation appears to be a major factor in high hydrogen consumption at high conversion levels.

Only limited data are presently available on the effect of temperature on the reaction kinetics or selectivity. This aspect of the work is presently being pursued. One comparison was made of the conversion of West Kentucky 9,14 coal at 800°F and 850°F at 30 sec contact time. The product balances are shown in Table 3.

It can be seen that even at this short contact time, major changes in the product are observed on increasing the temperature by ~50°F. The over-all conversion increased by 20% and the yield of SRC was increased by about 10%. Increased solvent range material yields were also observed. Because the oxygen conversion was still below 40%, no additional hydrogen consumption was noted.

A brief series of runs was made to examine the relative importance of H₂ and of solvent H-donor capacity in the early stages of liquefaction. The results are presented in Table 4. It is apparent that in the absence of H₂ gas, and H-donor solvent, heating the coal in pyridine to 800°F under N₂ pressure for either 1.3 or 60 min did not increase the pyridine solubility (Soxhlet extraction) above that observed for untreated coal. From run 18 it can be seen that the use of H₂ without a H-transfer agent resulted in a small increase in solubility. In Run 16 the use of a H-donor solvent without H₂ gave a substantial increase, approximately doubling the conversion over

extraction alone. For comparison, use of H₂ and H-donor gave 2.6 times the conversion.

These results suggest that in the early stages of liquefaction the availability of a H-transfer agent is the most important factor, although H₂ gas does have an effect. The possible importance of mass transfer of liquid solvent and product molecules to and from the reactive surface is clearly indicated. Complete liquefaction of Kentucky 9,14 coal in the absence of H₂ appears feasible in the presence of a good H-donor.

Initial Products in Solution

Information has been obtained on two additional facets of coal liquefaction: 1. the nature of the first solubilized species, and 2. the possibility of mass transport limitations on escape of the initial products from the particles.

To investigate these areas, we needed a procedure for distinguishing between coal that has been dissolved and is reacting but still remains within particles, and species that have escaped to the bulk solvent. Accordingly, we have attached a filter, below the liquid level inside the autoclave, to the liquid sampling line. We can, therefore, withdraw solution samples during a run, and these samples can contain only that portion of SRC product that is outside the coal particles. (This is also useful in scoping conversion vs. time to establish appropriate quench times for other runs.) The SRC content of these samples was determined by high pressure liquid chromatography. We used purified SRC's from previous high and low conversion runs to calibrate the system. A highly-polar solvent, pyridine, and a non-activated, trimethylsilylated silica, were used to ensure complete elution of SRC. Corrections were made for the synthetic solvent, which gives a very low response to our moving hot wire detector.

The results of run AC-31 (Wyodak coal, 800°F) are reported in Table 5. (The observations of molecular weight distribution changes as determined on these samples by gel permeation chromatography will be discussed below.)

We find that at least in the first four minutes of run AC-31 there is less SRC observed in solution than we know had been produced. In run AC-19, 1.3 min long, 38.5% of the coal was converted to SRC; after ~3 min in AC-31 only 2/3rds of this amount was observed in solution. It appears therefore that in the initial stages of the reaction a significant portion of the product remains within the coal particles. It must be pointed out that the SRC in run AC-19 was obtained after exhaustive pyridine extraction. The above conclusion is valid unless we make the unexpected finding that the synthetic solvent at 800°C is a poorer solvent for SRC than pyridine at typical extraction conditions (<100°C).

There is therefore a strong indication that even in a fast-stirred reactor with a good H-transfer solvent, there may be reactions occurring inside coal particles that could be different from those occurring outside. The results of initial studies indicate that SRC contains a bimodal molecular weight distribution. There is a high

molecular weight component (>2000 MW), or very strongly associated complexes of smaller very functional molecules, which in the early stages represent up to 40% of the product appearing in solution. The relative concentration of this material then rapidly declines, producing material in the 300-900 molecular weight range. This is shown in Figure 8. For the conversion of Wyodak coal in which the contents of the autoclave were sampled with time and analyzed by GPC, the results are shown in Table 5. These show that the absolute yield of this high molecular weight material (weight % of the coal fed) decreases throughout the run. Liquid chromatographic study of the high molecular weight material shows that it is very polar and highly functional. Furthermore, we find that the THF soluble portion of the pyridine extract of West Kentucky coal shows none of this high molecular weight material; the THF insoluble portion of the extract contains a significant amount of high molecular weight material. In addition, GPC examination of SRC's from our autoclave runs with Kentucky coal show high molecular weight material in low conversion runs, but very little in high conversion runs. The key point is that on pyridine extraction or at very low conversion, the coals show significant amounts of high molecular weight materials which are consumed rapidly on further conversion (times greater than ~3 min).

CONCLUSIONS AND WORK IN PROGRESS

From the data presented here we conclude that coal dissolution is very fast and requires very little hydrogen consumption. The presence of H₂ gas in the early stages of conversion is not critical, but a good H-donor solvent must be present. Sulfur and oxygen are removed in a kinetically-parallel fashion. About 40% of each may be removed readily and rapidly with little or no H-consumption; thereafter, considerably more hydrogen is consumed than the stoichiometry requires for the production of H₂S and H₂O. The initial products of coal dissolution contain significant amounts of high molecular weight material which is rapidly converted to low molecular weight products. The highest SRC yield is obtained early in the reaction process; improvement of SRC quality is accompanied by a decrease in yield and a large increase in hydrogen consumption.

Our present work includes more detailed chemical analysis of the SRC, extension of our studies to more coals and a wider range of conditions, and an investigation of possible mass transport limitations.

REFERENCES

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- (2) M. Monta and K. Hirose, Nenryo Kyokai-Shi 53 (564) 263-71 (1974).

ACKNOWLEDGEMENTS

We wish to acknowledge helpful discussions with M. Farcasiu and the experimental assistance of B. O. Heady.

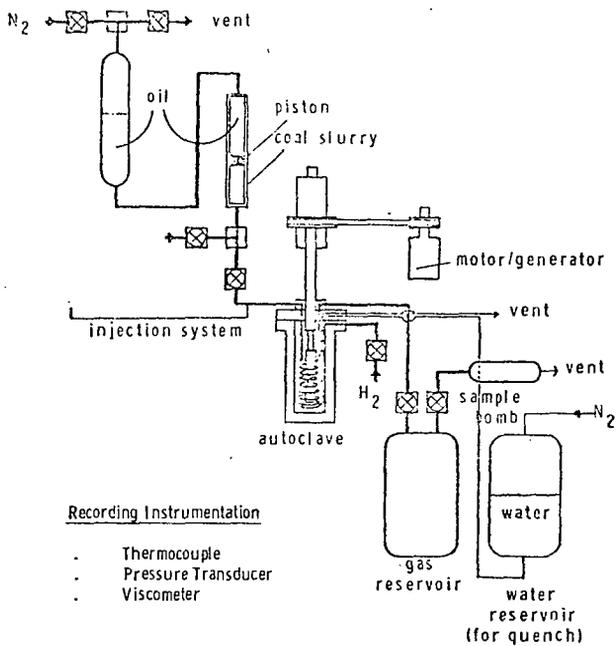


Figure 1

Figure 2

TYPICAL TIME-TEMPERATURE PROFILE

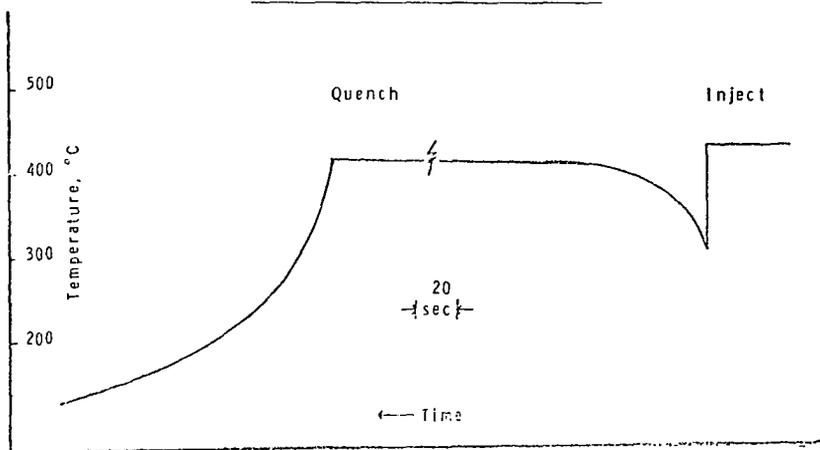


FIGURE 3

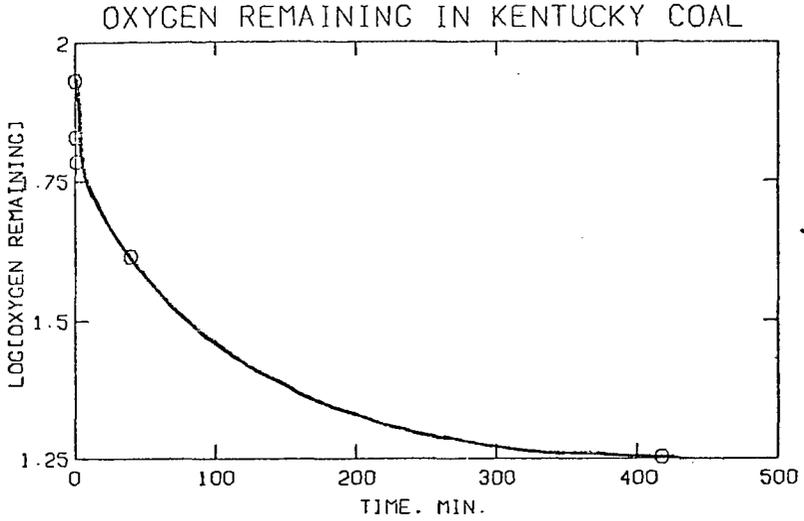


FIGURE 4

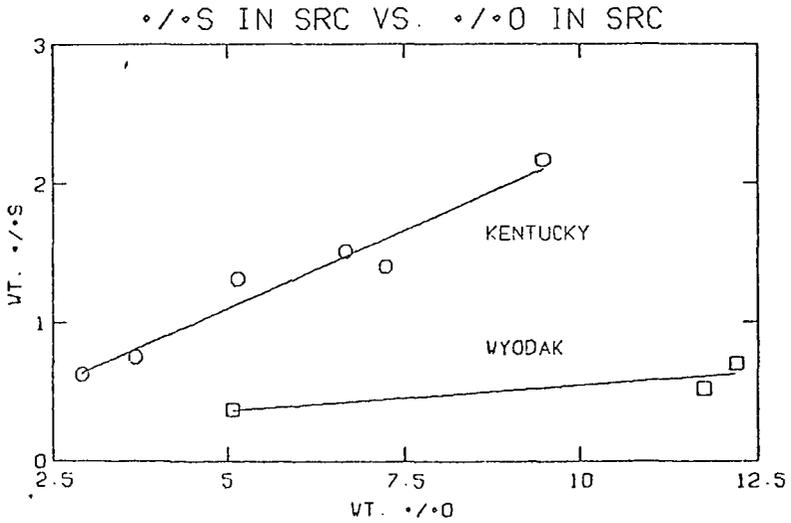


FIGURE 5

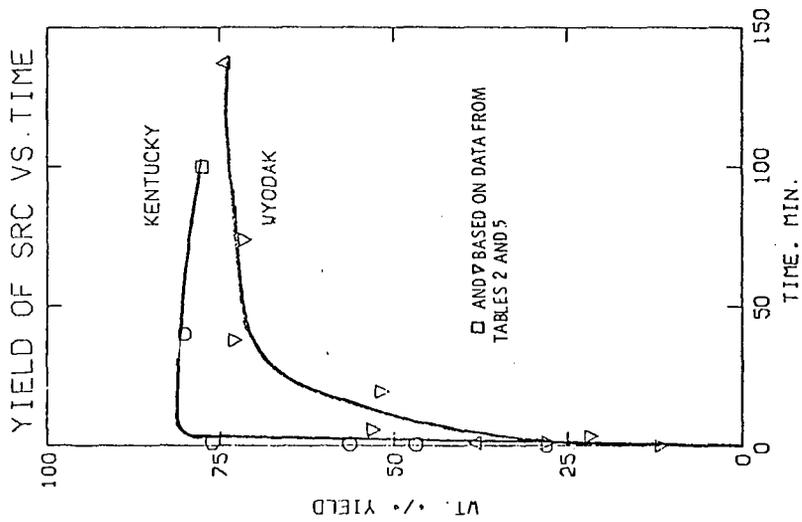


FIGURE 8

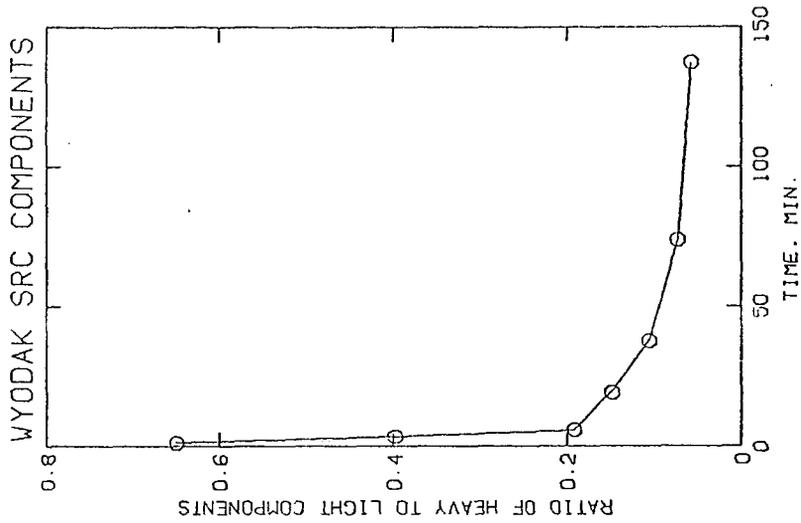


FIGURE 6

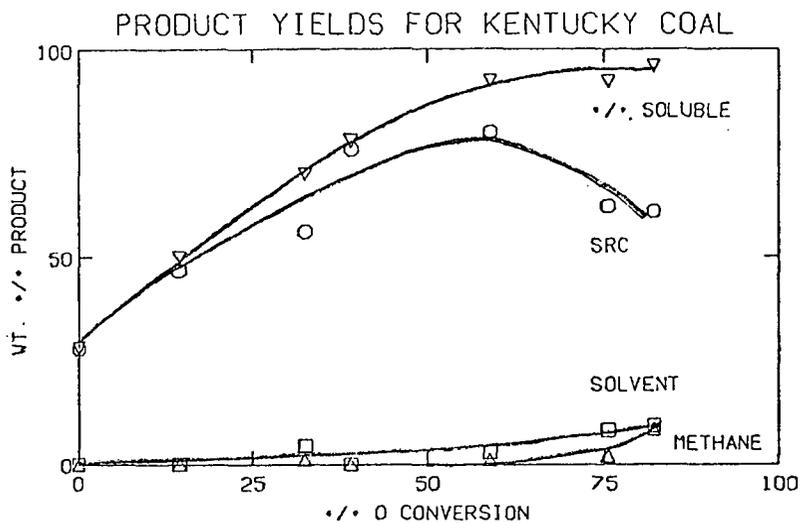


FIGURE 7

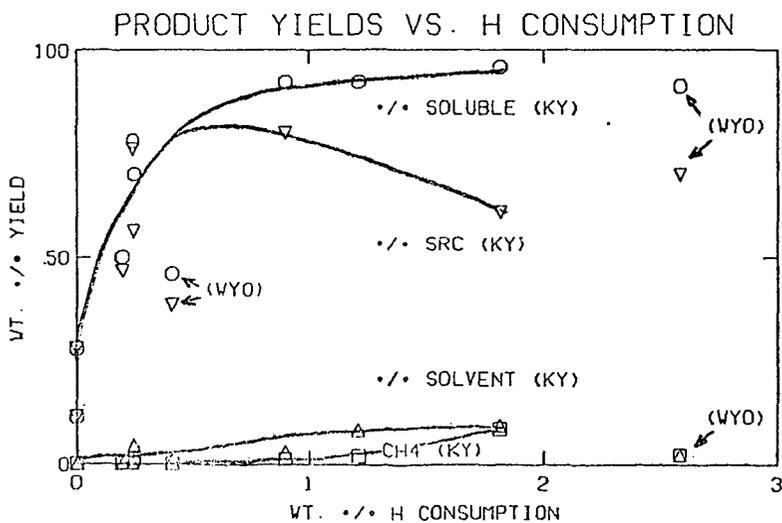


Table 1

Analyses of Coals of the Project

Name of Coal		Wyodak	W. Kentucky
State		Wyoming	Kentucky
County		Campbell	Hopkins
Seam		Anderson	9,14
Name of Mine		Bell Ayre	Colonial
% Moisture (as rec.)		22.03	6.05
% Ash (as rec.)		3.63	7.83
% Volatile Matter		47.44	36.61
% Fixed Carbon		47.90	55.06
BTU (as rec.)		9599.	12291.
BTU		12311.	13082.
Free Swelling Index		.5	4
% C		71.82	73.06
% H ₂		5.20	5.00
% O ₂		17.12	9.17
% N		.90	1.47
% S (total)		.30	2.97
% S (pyritic)		.06	1.19
% S (organic)		.23	1.36
% S (sulfate)		.01	.42
% Cl		0.0	.00
% Ash		4.66	8.33

* All analyses, are given on a dry weight basis unless otherwise stated.

† by difference

Table 2

Run Balances for Coal Conversions at 800°F

Run Number	10.00	9.00	7.00	12.00	19.00	31.00
Solvent	Synth	Synth	Synth	Synth	Synth	Synth
Coal	WKy 14	WKy 14	WKy 14	WKy 14	Wyodak	Wyodak
Temperature, °F	800.00	800.00	798.00	800.00	806.00	800.00
Pressure, psig H ₂	1348.00	1300.00	1160.00	1030.00	1415.00	1072.00
Duration (feed), min.	.50	1.30	40.00	417.00	1.30	137.50
MAF Conversion, wt. %	50.00	78.20	92.52	96.10	45.97	91.52
Solvent/Coal	4.65	6.66	4.56	9.06	8.48	6.17
H ₂ S	.24	.20	.67	.00	.01	.00
Water	4.39	3.46	4.87	5.16	4.21	4.59
CO	.10	.00	.16	.31	.28	1.28
CO ₂	.49	.54	.83	1.65	1.61	6.04
C ₁	.17	.18	1.18	8.57	.17	2.50
C ₂ -C ₅	2.51	.36	1.60	8.47	.21	4.53
(C ₆ -257°F)	.34	.14	.22	1.14	.14	.14
(257-650°F)	.00	.00	2.89	9.40	.77	2.38
SRC	46.78	76.12	80.11	61.05	38.53	70.03
MAF Residue	49.76	21.80	7.46	3.93	54.03	8.48
Balance	104.78	102.80	100.00	100.00	99.96	100.00
Ash in Residue	19.30	31.20	53.08	52.50	8.20	37.21
Duration (Solv), Min.	17.00	15.00	42.00	429.00	32.00	149.50
4-Picoline	1.90	1.90	1.87	3.20	2.01	1.28
p-Cresol	16.10	16.10	16.11	15.80	16.21	13.07
Methyl Indane	.97	.92	1.88	6.50	1.37	2.59
Tetralin	41.00	41.22	33.41	25.30	40.02	25.22
Naphthalene	2.24	2.10	8.64	14.50	2.32	18.13
Methyl Tetralin	.09	.28	1.44	4.70	.30	2.37
2-Methyl Naphthalene	37.67	37.48	36.62	29.60	37.76	37.36
H ₂ Consumption	.13	.34	.89	1.59	.41	2.46
FOOTNOTE:	5.00		11.00	8.00		10.00

- 5) Gas analysis questionable
- 8) Piston pressure resisted syringe; balance forced to total recovered products.
- 10) Balance forced; samples removed during run.
- 11) Water content based on oxygen balance.

Table 3

The Effect of Temperature on Liquefaction of W. Kentucky Coal

Run Number	10.00	14.00
Solvent	Synth	Synth
Coal	WKy 14	WKy 14
Temperature, °F	800.00	847.00
Pressure, psig, H ₂	1348.00	1012.00
Duration (feed), min.	.50	.50
MAF Conversion, wt. %	50.00	70.07
H ₂ S	.24	.10
Water	4.39	3.65
CO	.10	.21
CO ₂	.49	.83
C ₁	.17	1.25
C ₂ -C ₅	2.51	2.71
(C ₆ -257°F)	.34	.63
(257-650°F)	.00	4.38
SRC	46.78	56.31
Residue	49.76	28.93
Balance	104.78	100.00
Ash in Residue	19.30	16.93
Duration (Solv), Min.	17.00	15.00
4-Picoline	1.90	2.14
p-Cresol	16.10	16.98
Methyl Indane	.97	1.93
Tetralin	41.00	39.55
Naphthalene	2.24	1.82
Methyl Tetralin	.09	.26
2-Methyl Naphthalene	37.67	37.31
FOOTNOTE:	5.00	7.00

- 5) Gas analysis questionable
 7) Mat bal. low; bal forced to total recovered prods; gas analysis poor

Table 5

Wyodak SRC in Solution During Run AC-31

<u>Time, Min.</u>	<u>Wt. Percent of Original Coal</u>		<u>Total</u>
	<u>Low Molecular Weight</u>	<u>High Molecular Weight</u>	
1.20	16.86	10.97	27.80
3.60	15.41	6.13	21.53
6.00	44.32	8.48	52.80
19.50	44.99	6.70	51.69
38.00	65.77	6.87	72.64
74.00	66.45	4.79	71.24
137.50	70.54	4.03	74.57

Table 4

Criticality of Solvent and Hydrogen

Run Number	16.00	9.00	17.00	12.00	7.00	3.00
Solvent	Synth	Synth	Pyrd	Pyrd	Synth	Pyrd
Coal	WKy 14	WKy 14	WKy 14	WKy 14	WKy 14	WKy 14
Temperature, °F	800.00	800.00	800.00	800.00	798.00	796.00
Pressure, psig H ₂	.00	1300.00	.00	809.00	1160.00	.00
Duration (feed), min.	1.28	1.30	1.28	1.18	40.00	60.00
MAF Conversion, wt. %	65.36	78.20	30.95	40.13	92.52	29.78
Solvent/Coal	5.04	6.66	5.17	5.10	4.56	5.00
H ₂ S	1.00	.20	.00	.00	.67	.00
Water	5.28	3.46	4.75	4.59	4.87	.00
CO	.00	.00	.00	.05	.16	.00
CO ₂	1.90	.54	.00	.05	.83	1.40
C ₁	.53	.18	.00	.00	1.18	1.32
C ₂ -C ₅	2.43	.36	.00	.05	1.60	2.01
(C ₆ -257°F)	.16	.14	.11	.00	.22	.00
(257-650°F)	.58	.00	.05	.48	2.89	12.11
SRC	58.58	76.12	26.00	28.56	80.11	19.67
MAF Residue	37.35	21.80	69.05	59.87	7.46	63.49
Balance	107.81	102.80	100.00	93.65	100.00	100.00
Ash in Residue	20.63	31.20	10.97	12.30	53.08	12.02
Duration (Solv), Min.	21.00	15.00	18.00	22.00	42.00	60.00
4-Picoline	1.78	1.90	.00	.00	1.87	.00
p-Cresol	15.43	16.10	.13	.02	16.11	.00
Methyl Indane	1.79	.92	.00	.00	1.88	.00
Tetralin	38.95	41.22	.37	.25	33.41	.00
Naphthalene	2.86	2.10	.02	.00	8.64	.00
Methyl Tetralin	.12	.28	.00	.00	1.44	.00
2-Methyl Naphthalene	39.08	37.48	.27	.18	36.62	.00
H ₂ Consumption	.29	.34	.00	.00	.89	.00
FOOTNOTE:			9.00	9.00	11.00	2.00

- 2) Balance forced; feed not injected but present during heat-up.
 9) H₂O assumed to force balance 17; same H₂O amount assumed for 18.
 11) H₂ content based on oxygen balance.