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RESEARCH AND DEVELOPMENT OF AN ADVANCED PROCESS FOR THE CONVERSION OF COAL TO SYNTHETIC GASOLINE AND OTHER DISTILLATE FUELS

Quarterly Technical Progress Report No. 6

for the Period August 1977
through October 1977

A. Schneider
E. J. Hollstein
E. J. Janoski
A. F. Talbot

SUNTECH, INC.

(A Subsidiary of Sun Company)

Research and Engineering Division

P. O. Box 1135

Marcus Hook, Pennsylvania 19061

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MASTER

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I. OBJECTIVE AND SCOPE OF WORK

The objective of this program is (1) to produce coal liquids that can be converted to high-octane gasoline and distillate motor fuels in conventional petroleum refining processes and equipment, the entire operation being economically and technically viable, and (2) to perform an engineering assessment of (1) and its economic potential in a continuous bench-scale unit employing a practical reactor design and catalyst system at a scale not exceeding 1-3 pounds of coal per hour under steady state conditions.

Specifically, the course of action is to apply very deep hydrogenation during the dissolution of coal, while minimizing cracking, to achieve hydrogen-to-carbon atomic ratios suitable for catalytic cracking, hydrocracking, etc. of the total products of deep hydrogenation or of distillate fractions thereof. It is recognized that substantial removals of nitrogen and oxygen compounds probably will not occur during the catalytic hydrogenations, and chemical removal of these non-hydrocarbons, for example by precipitation with hydrogen chloride after the hydrogenation step, but prior to the catalytic cracking of hydrocracking operations, is necessary and will be carried out.

II. SUMMARY OF PROGRESS TO DATE - (See Time-Line Chart - Figure 1)

This report describes the progress in the development of a process to convert coal to gasoline for the period August 1, 1977 through October 31, 1977. During the sixth quarterly period of effort on this contract, we accomplished the following:

1. We carried out 13 reactions in our one-liter reactor. Six of these were carried out to generate dimethyltetralins from dimethylnaphthalenes for use as special solvent. Four other reactions were carried out to generate solvent from anthracene oil. One reaction was carried out with the dimethyltetralin solvent at 425°C. The final two reactions were used to examine the activity of our powdered catalyst. The results from these reactions indicate that:

   a. demethylation of dimethyltetralines occurs at 425°C and limits its use as a solvent for the hydroliquefaction of coal. Dimethyltetralins might be used as a solvent at a reaction temperature of <400°C where demethylation may not be a problem,

   b. reproducibility of our experiments is excellent,

   c. the powdered 150 to 270 mesh version of our catalyst appears to be more active (as gauged by hydrogen absorption) than our 8 to 20 mesh version. About 20 weight percent more hydrogen is absorbed in the same period of time when the powdered catalyst is employed.

2. We continued our examination of the effect of treatment of coal hydroliquefaction products with hydrogen chloride and n-hexane. The results from these efforts indicate that:

   a. Nitrogen contents of raffinates are reduced from about 0.62 wt.% (calculated) to <0.01 wt. % and to meet our contract goal. The available data indicate that the HCl-hexane treatment removes both basic and non-basic nitrogenous material. The ratio of basic to total nitrogen in the products of our coal hydroliquefactions is generally $\geqslant$ 2:3.

   b. Depending upon the degree of hydrogenation, 12 to 14 wt.% extract is precipitated from hydroliquefaction products which were filtered and distilled to about 1000°F.

   c. About 19 to 33 wt.% material is precipitated by the HCl-hexane treatment of hydroliquefaction product which was filtered but not distilled.

   d. Similar results are obtained whether dilution with hexane is carried out before or after HCl treatment.

      Treatment by both HCl and hexane are required to produce these results. Treatment of the hydroliquefaction product which was filtered but not distilled with 100 volume % hexane alone only reduces the nitrogen level to 0.49 wt.% and it appears that this treatment does not precipitate either basic or non-basic nitrogenous material preferentially.

   e. We must attempt to hydrogenated to a greater extent to reduce the amount of coal-derived material that is precipitated.

3. The design of and a preliminary cost estimate for an ebullating bed Bench Scale Continuous Unit (BSCU) were completed. The design is for a charge rate of three pounds of coal per hour utilizing once-through hydrogen. The design closely resembling the HRI reactor, has been questioned by the Project Manager.

III. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

A. REACTIONS AND DISCUSSION

1. GENERAL

During this quarter, we carried out thirteen reactions in our one-liter reactor. Six of these were carried out to generate dimethyltetralins from dimethylnaphthalenes for use as a special solvent. Four other reactions were carried out to generate hydrogenated anthracene oil for use as solvent. One reaction was carried out with the dimethyltetralin solvent. Two other reactions were carried out utilizing the powdered form of our catalyst.

Appendix A is an up-to-date summary of all the reaction conditions and available material balances of reactions carried out in our one-liter reactor. It is intended to serve as a compendium of all of the reactions carried out in the one-liter reactor. The order of presentation in this appendix is chronological. Analyses of gaseous and liquid reaction products are presented in various tables of this report. These data are so diverse and voluminous that we will not follow our usual pattern of including summaries of these analyses in separate appendices.

Ten of the reactions completed this quarter were carried out to generate solvent for subsequent reactions. In general, these reactions were not subjected to individual detailed workup. Instead, the products of like hydrogenations were filtered, combined, and analyzed in one batch so that we could calculate the total charge analyses of subsequent reactions.

2. HYDROGENATION WITH DIMETHYLTETRAL IN SOLVENT

Six hydrogenations of dimethylnaphthalenes (DMN's) were carried out in the one-liter reactor this quarter to make dimethyltetralins (DMT's). Our hope was to use the DMT's as solvent in coal hydrogenation and to be able to separate positively the products which derive from coal from the products which derive from DMT's or DMN's.

The DMN hydrogenations were designated reactions 755: 588, 590, 592, 594, 596, and 598. Table I shows an analysis of the DMN mixture which was hydrogenated. The DMN hydrogenations were carried out at about 275°C and 2000 psig over 10 wt.% presulfided Filtrol HPC-5 CoMo catalyst. The reaction products from the six reactions were combined, filtered, and distilled into 16 overhead cuts. Cut No. 7, boiling range 247°C to 248°C was used as the solvent for Reaction 785602.

Reaction 755602 was a hydrogenation of 135.4 grams of Illinois No. 6 coal (740700) in 270.8 grams of DMT (755598, Cut No. 7) for 2 hrs. at 425°C, 2500 psig over presulfided Sun 740820-1% CoO 2% MoO_3 on 8 to 20 mesh bauxite. The reaction product has not been worked up, however, qualitative observations indicate that not much hydrogen was absorbed. Further, the relatively large amount of methane found in the gas analyses, Table II, indicates that demethylation of the DMT's (or DMN's which may have been formed) was occurring.

The net result of this indicates that if DMT is to be used as a solvent in this reaction the reaction temperature must be kept below 400°C. If time permits, we plan to carry out a reaction with DMT solvent at this temperature.

3. HYDROGENATION WITH HYDROGENATED ANTHRACENE OIL SOLVENT

Four reactions were carried out to generate hydrogenated anthracene oil solvent. The reaction products of these reactions were filtered and combined so that subsequent reactions could be carried out with a common solvent.

Reactions 755616 and 755623 were carried out to assess the activity of our powdered catalyst. By "powdered" catalyst we mean catalyst which is considerably finer than the 8 to 20 mesh catalyst which we have employed till now. In the case of Reactions 755616 and 755623 we employed catalyst Sun Code 740710 -1% CoO, 2% MoO_3 on bauxite which passed through 100 mesh and was retained on 270 mesh sieves. The available data from these reactions are summarized in Table III. Note that all of these reactions were carried out for the same length of time at the same temperature, pressure, and catalyst loading and with hydrogenated anthracene oil solvents which had about the same hydrogen to carbon atomic ratio. The products have not yet been analyzed but the hydrogen absorption was calculated accurately from data obtained from our hydrogen reservoir. These data indicate excellent reproducibility for reactions 755616 and 755623. Further, over 20 percent more hydrogen is absorbed in the same period of time when the powdered catalyst is employed. These data, together with those from the HCL treat, Table V, suggest that the powdered catalyst is much more active than a granular one. This was unexpected and may be related to the pore distributions of our bauxite base. (The next section of this report describes in detail the pore size distribution of our catalyst and that of Filtrol HPC-5). The unexpectedly high activity of the powdered catalyst may be caused by a number of factors, such as:

a. the exposure of more catalyst surface area to the very large coal molecules, or,

b. the use of a different batch of catalyst. The powdered catalyst which was used was obtained by impregnating a powdered bauxite. Perhaps better dispersion of metals could have occurred on the powdered form of the bauxite. It would be interesting to carry out an experiment with a pulverized sample of our Code 740320 - 8 to 20 mesh catalyst. We plan to carry out a comparative reaction with the HPC-5 catalyst.

4. PORE SIZE DISTRIBUTION OF CATALYSTS

Our catalyst, Sun Code 740820 - 1% CoO, 2% MoO_3 on 8 to 20 mesh bauxite, has a grossly different pore-size distribution than that of commercial Filtrol HPC-5 CoMo catalyst. Both catalysts were examined via mercury porosimetry by the American Instrument Company. The results of these examinations are reported in several ways in this report. Table IV is a listing of cumulative pore volume of the two catalysts. Figures 2A and 2B are plots of the volume of mercury penetration versus absolute pressure and equivalent pore diameter. Figure 3 is a plot of the differential change in volume of mercury penetrated by a change in pressure (dV) divided by the natural logarithm of the average pore radius (d ln r) versus the average pore radius. This plot thus shows the pore frequency as a function of pore size.

Clearly, the Sun catalyst contains much larger pores and a much broader pore-size distribution but much less pore volume. The effect of this difference in the catalysis of the hydroliquefaction of coal is not clear. As stated in the previous section, we have observed a significant increase in catalytic activity shown by the powdered version of our catalyst. A reason for this increased activity could be the unusual pore size distribution of our bauxite base.

5. HYDROGEN CHLORIDE TREATMENT OF COAL-DERIVED LIQUID PRODUCTS

a. GENERAL

To remove nitrogeneous materials from coal-derived products, we first hydrogenate to convert the nitrogen to the basic form and then treat the product with gaseous hydrogen chloride (HCl) to precipitate the basic nitrogenous material as the amine-hydrochloride. We found, however, that when we treated the hydrogenated product of coal with HCl little precipitate was formed and that the amine-hydrochloride was soluble in the bulk of the hydrogenated product. We then diluted the HCl-treated product with 100 volume % n-hexane. A precipitate occurred but this time the nitrogen content of the raffinate was 0.08 to 0.10 wt.%.

Our HCl treating procedure remains as before: carried out simply by bubbling HCl through the liquids at room temperature and pressure. Separation is effected by filtration or centrifugation. The raffinate is washed with 10 wt.% aqueous KOH and 10 wt.% aqueous KCl solutions. There are, however, some variations on the possible ways of treating a hydrogenated coal-derived product. We have devised three such schemes and have designated them as Schemes A, B, or C. Figure 4 is a schematic which summarizes the schemes. All schemes follow filtration of the hydroliquefaction product. Scheme A involves a distillation to about 1000°F followed by HCl treat and dilution with 100 volume % hexane of the 1000°F-distillate. Scheme B involves the HCl-n-C_6 treat of the whole product. Scheme C involves only the n-C_6 treat of the whole product.

b. RESULTS

Table V summarizes the work carried out thus far in the HCl-n-C_6 treatment of coal hydroliquefaction products. The data indicate that:

1. Nitrogen contents of raffinate obtained via Schemes A & B are about 0.08 to 0.10 wt.% and thus meet our contract goal

2. Both treatment with HCl and dilution with n-hexane (or its equivalent) are necessary. The order of treatment or dilution is not important. If n-hexane alone is used, the nitrogen content of the raffinate is about 0.49 wt.%

3. Depending upon the degree of hydrogenation, about 12 to 24 wt.% extract is precipitated from the hydroliquefaction products which were filtered and distilled to 1000°F. (Treated via scheme A). Calculation indicates that 37 wt.% (Reaction 755566) to 81 wt.% (Reaction 755580) of the coal-derived material is precipitated.

4. If Scheme B is considered, about 19 wt.% (Reaction 755616A) to 33 wt.% (Reaction 755546) extract is precipitated. Calculation indicates that about 58 to 100 wt.% of the coal-derived material is precipitated.

The data thus indicate that we must hydrogenate to greater extent to reduce the amount of coal which is precipitated.

Included in Table V is the percent of coal which was precipitated from the feed to the various schemes. This serves as an indicator of the depth of hydrogenation. These data support the contention that the powdered catalyst is more active than the granular catalyst (Compare Reaction 755566 to Reaction 755616A). Also, evidence is found again to show that the hydrogen content of the anthracene oil solvent affects the hydroliquefaction of coal dramatically. (Compare Reaction 755552 with Reaction 755580 - Schemes A in both cases). Finally, data are shown for reactions which were carried out in the 300 ml reactor (Reaction 740855 and 740856). These reactions were carried out in the presence of lithium and lithium-cerium-doped catalysts. Direct comparison with other reaction data is difficult, however, because of the great difference in reaction temperature and time.

B. DESIGN OF THE BENCH-SCALE CONTINUOUS UNIT (BSCU)

We have continued to design the BSCU. A preliminary cost estimate for an ebullating bed BSCU has been completed and is included in this report as Appendix B. The engineering flow diagram has been omitted because of its physical size but a preliminary copy was submitted earlier to Dr. John Shen, the Program Manager.

The design is for a charge rate of about three pounds of coal per hour and an ebullating bed unit. We have opted for a once-through hydrogen mode. On this basis, the cost estimate, including labor and equipment is $372,500. The time for completion is estimated at 12 to 16 months.

The Program Manager questions the ebullating bed design. A meeting with Dr. John Shen, the Program Manager, in September established that our design closely resembled existing processes and did not allow for continual catalyst replacement. Another problem appears to be the definition of powdered catalyst. Accordingly, he asked that we re-evaluate our design and recommendations and to present them to ERDA for review. We will complete this before December 1977.

C. WORK FORECAST

1. Continue workup and analyses of hydrogenation products.
2. Continue HCl treatment and analyses of hydrogenation products.
3. Carry out a reaction at 425°C with HPC-5 catalyst.
4. Attempt hydroliquefaction at 450°C.
5. Attempt more thorough conversion of coal to lighter materials.
6. Consider ways of separating coal ash, catalyst, and unconverted coal from each other and from liquid products.
7. Continue design of the BSCU to include evaluation of designs other than ebullating bed and evaluation of catalyst replenishment features.

IV. CONCLUSIONS

We conclude from our present work that:

1. Dimethyltetralin is unacceptable as a hydroliquefaction solvent. At 425°C DMT apparently demethylates.

2. The powdered form (150 to 270 mesh) of our catalyst appears to be significantly more active than the granular form (8 to 20 mesh).

3. Our HCl-hexane treatment yields a product with only about 0.1 wt.% nitrogen. This product meets our contract goal.

4. We apparently do not hydrogenate to a great enough extent. About 12 to 24 wt.% material precipitates with the HCL treatment of 1000°F distillate but this constitutes 37 to 81 wt.% of coal-derived material.

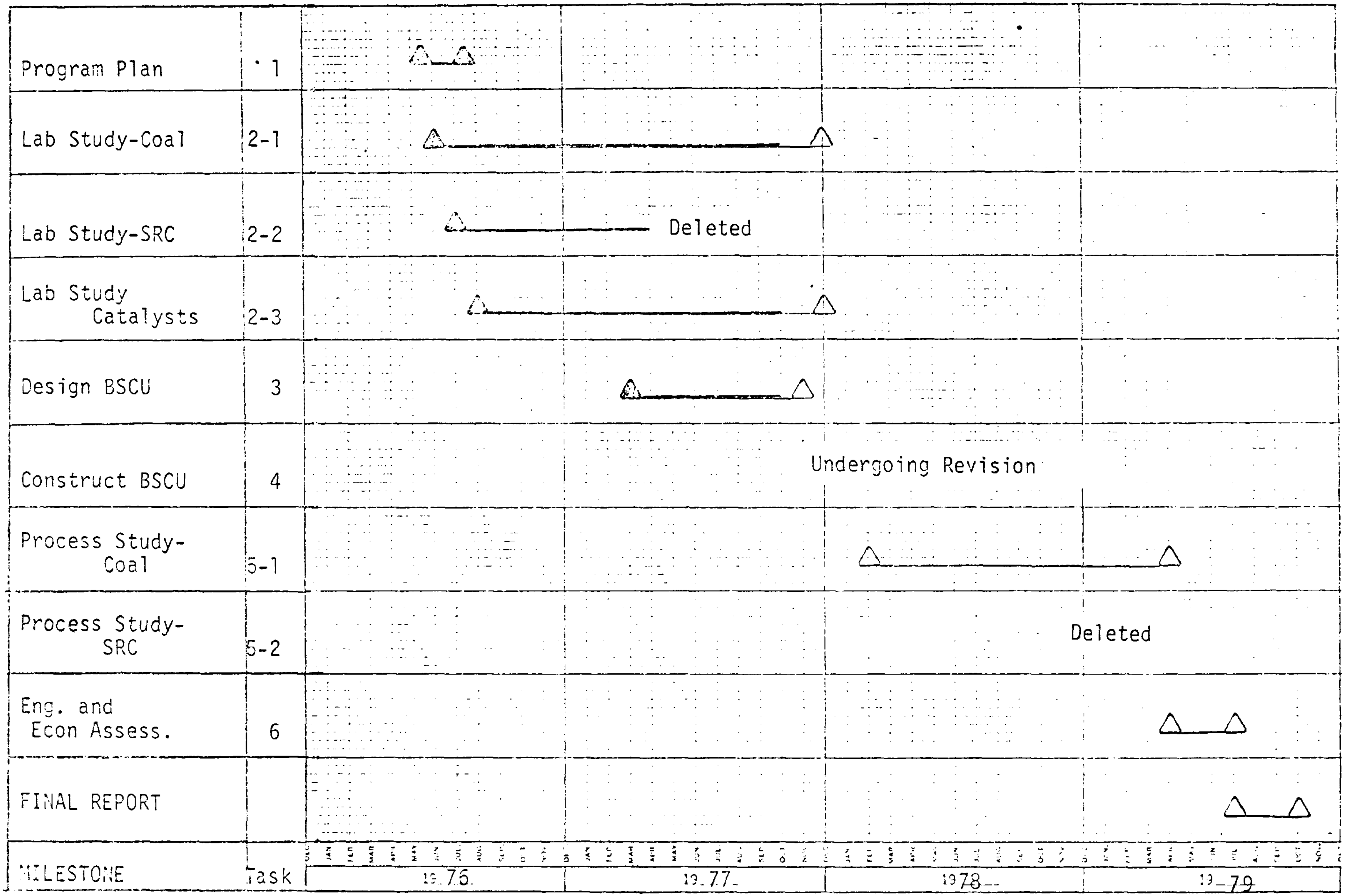
5. We have designed an ebullating bed reactor but this has evolved objections on the grounds that it resembles existing processes and does not provide for catalyst replacement.

EJH:jmr

FIGURE 1

TIME-LINE CHART(1)

QUARTERLY TECHNICAL PROGRESS REPORT NO. 6



(1) Indicates timing of tasks only. This does not indicate manpower loading.

FIGURE 2 PORE-SIZE DISTRIBUTION OF TWO CATALYSTS

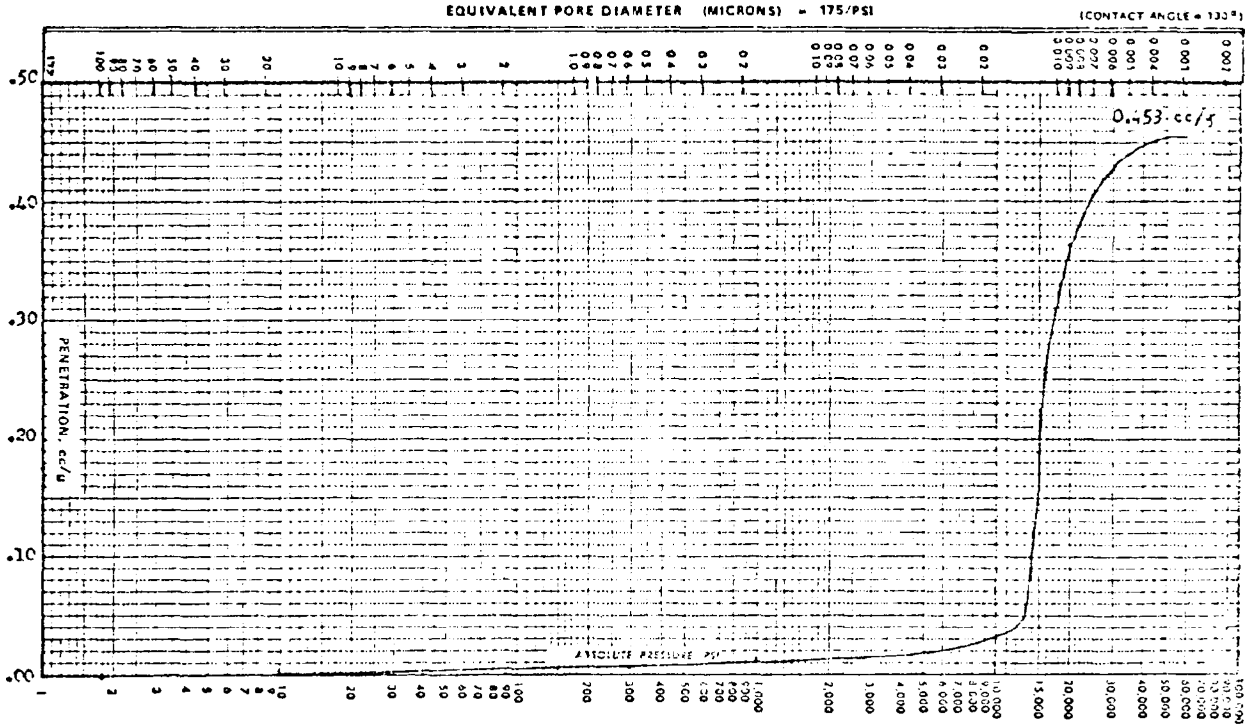
POROSITY DETERMINATION

A. Filtrol HPC-5

SAMPLE #755610-1

SAMPLE WEIGHT 0.3053 grams

DATE 9-21-77 ORDER No. R7-2626



POROSITY DETERMINATION

B. Sun 740820

SAMPLE #755610-3

SAMPLE WEIGHT 0.3059 grams

DATE 9-22-77 ORDER No. R7-2626

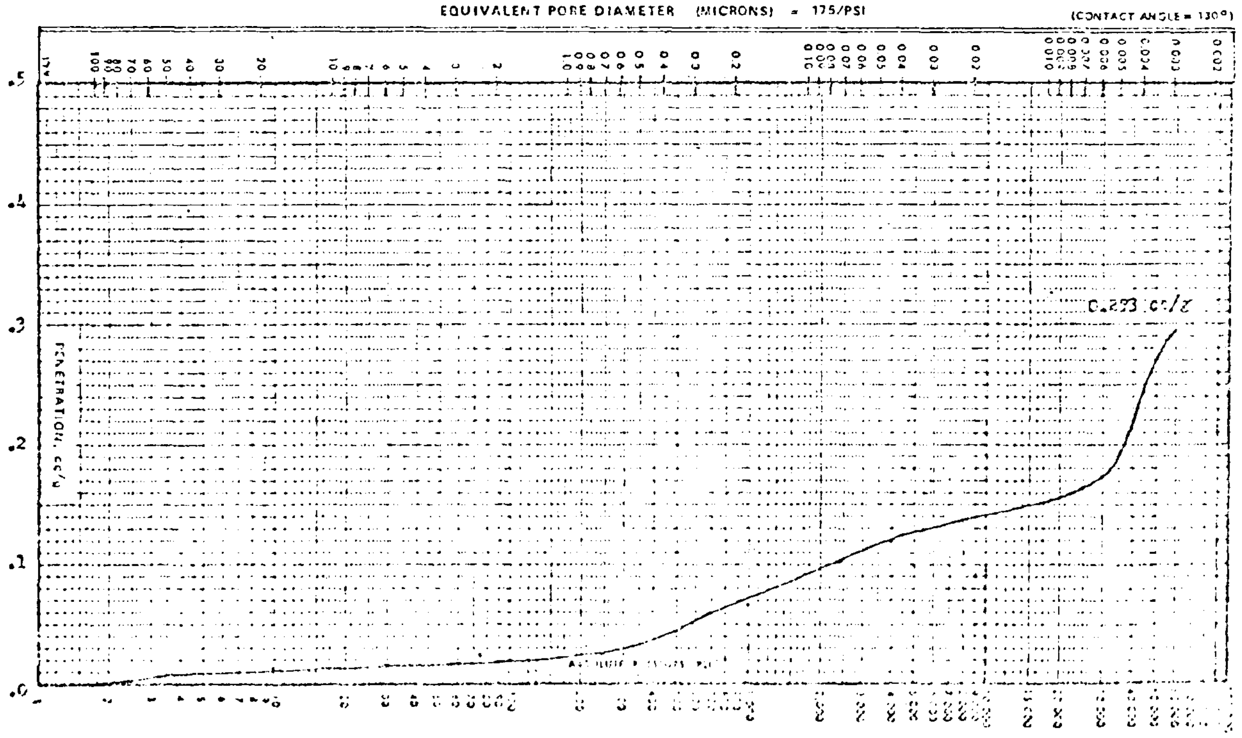


Figure 3

Catalyst Pore Frequency as a Function of Pore Size

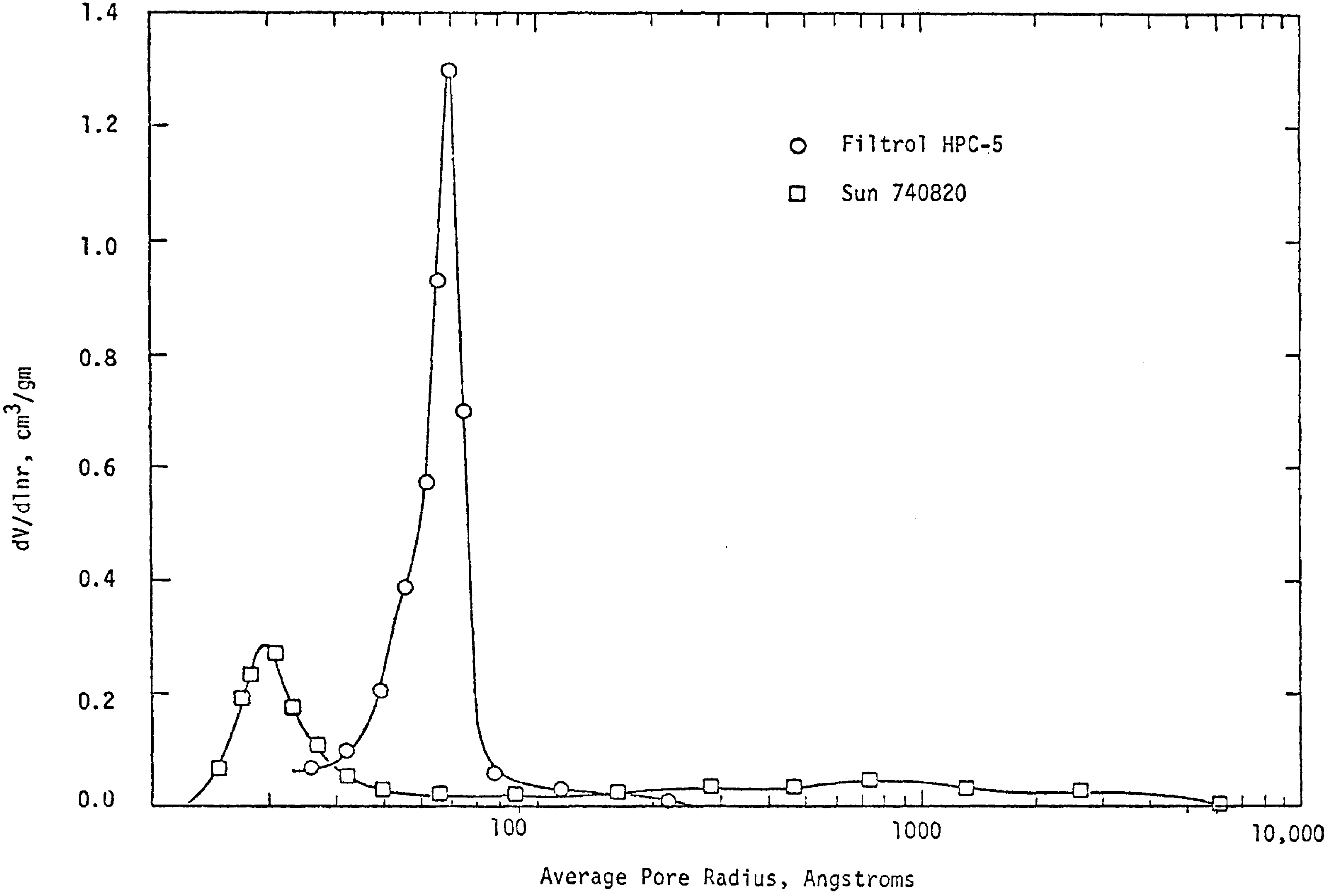


FIGURE 4

Possible Processing Schemes

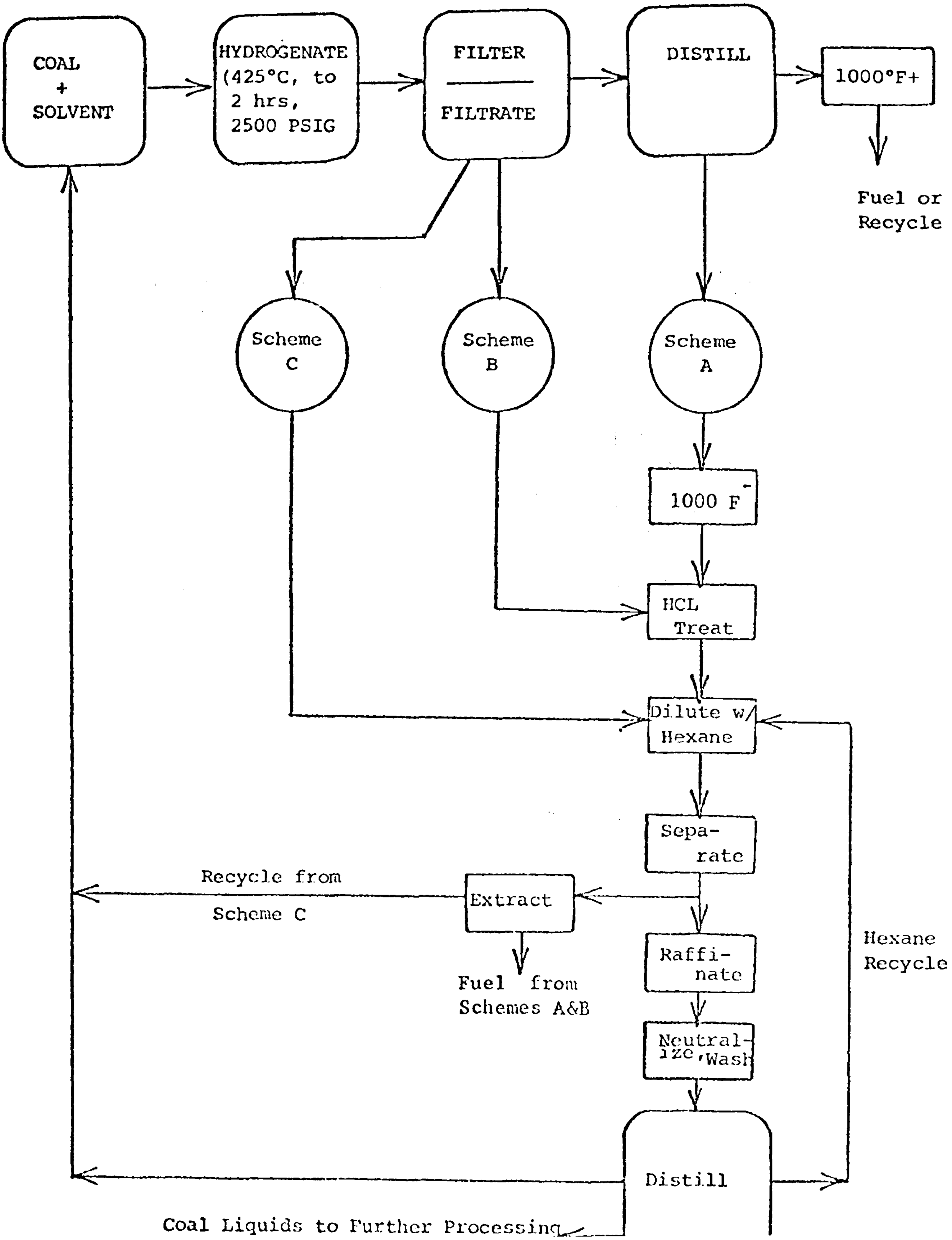


TABLE I

ANALYSIS OF DIMETHYLNAPHTHALENE MIXTURE WHICH WAS HYDROGENATED TO DIMETHYLTETRALIN FOR USE AS SOLVENT

	Wt.%
Alpha methyl naphthalene	0.4
Beta methyl naphthalene	0.2
2,6-and 2,7-dimethyl naphthalene	2.7
1,7-dimethyl naphthalene	0.6
1,3-and 1,6-dimethyl naphthalene	15.4
1,5-dimethyl naphthalene	78.9
Others, not identified	1.8
Total	100.0

Analysis via vapor phase chromatography

TABLE II

SUMMARY OF GAS ANALYSES FROM REACTION 755602

Note: Charge to one-liter stirred reactor was 270.8 gms of dimethyltetralins (Sun Code 755598, Cut. No. 7), 135.4 gms of Illinois No. 6 coal (Sun code 740700), 40.6 gms of Sun catalyst: 1% CoO, 2% CoO_3 on 8 to 20 mesh Bauxite. Reaction carried out at 425°C, 2500 psig H_2, for 2 hours. Three gas samples were taken during the reaction (hot). The fourth sample was taken after the reactor had cooled.

Gas Sample No.	1	2	3	4
Reaction time, min.	25	60	90	120
Gas, mole %, air free				
hydrogen	75.9	67.5	64.1	61.9
methane	10.2	16.8	20.7	26.5
ethane and ethene	1.6	2.1	2.5	3.0
propane	1.0	1.4	1.6	1.5
butenes	0.2	0.2	0.1	0.0
butanes	0.4	0.6	0.7	0.6
pentanes	0.3	0.4	0.4	0.0
hexenes and hexanes	0.2	0.6	0.6	0.1
carbon monoxide	0.2	0.2	0.5	0.5
carbon dioxide	1.5	1.5	1.3	0.9
hydrogen sulfide	8.5	8.7	7.5	5.0

TABLE III

SUMMARY OF REACTION DATA: COMPARISON OF SUN POWDERED AND GRANULAR CATALYST

	Reactants, gms					Reaction Conditions			Hydrogen Absorbed, gms(7)	
Reaction No.	Coal(1)	Solvent	Catalyst	H_2	Total	Time (min)	Temp (°C)	Pressure (PSIG)	Total	Per 100 gms MAF Coal
755552	133.0	268.2(2)	40.2(5)	7.3	449.1	120	425	2500	4.4	3.8
755616	134.3	268.5(3)	40.3(6)	8.2	451.3	120	425	2500	5.4	4.6
755623	134.1	268.3(4)	40.4(6)	8.6	451.4	120	425	2500	5.5	4.7

(1) Sun Code 740700 - Illinois No. 6 Coal

(2) Hydrogenated Anthracene Oil 755538-D (H/C = 1.14)

(3) Hydrogenated Anthracene Oil 755612 (H/C = 1.14)

(4) Hydrogenated Anthracene Oil 755612A (H/C = 1.17)

(5) Sun Code 740820 - 1% CoO, 2% MoO_3 on 8 to 20 mesh bauxite

(6) Sun Code 740710 - 1% CoO, 2% MoO_3 on 100 to 270 mesh bauxite

(7) Determined directly from hydrogen reservoir data

TABLE IV

Cumulative Pore Volume (ml/gm) of Selected Catalysts

Pore Diameter (Å)	Catalyst Filtrol HPC-5	 Sun 740820*
30	0.000	0.023
40	0.006	0.049
50	0.013	0.104
60	0.026	0.120
70	0.046	0.130
90	0.091	0.137
100	0.140	0.139
200	0.430	0.155
400	0.453	0.169
700	0.453	0.189
1,000	0.453	0.201
5,000	0.453	0.260
10,000	0.453	0.271

*Sun 740820 catalyst = 1% CoO, 2% MoO_3 on 8 to 20 mesh bauxite

TABLE V

SUMMARY OF DATA OBTAINED FROM HYDROGEN CHLORIDE AND HEXANE TREATMENT OF HYDROLIQUEFACTION PRODUCTS

Reaction No.	755546		755552		755566		Anthracene Oil		Hydrogenated Anthracene Oil	
Reaction Conditions (1)										
Time, min.	60		120		120		-		480	
Temp., °C	425		425		425		-		375	
Press., psig	2500		2500		2500		-		3000	
Catalyst, wt.% (9)	10		10		20		-		10 (8)	
Reaction Product Data										
Filtered Liquid Product, wt.% N	0.62		0.51		0.43		-		-	
Distilled Products										
Distillate, 1000°F										
Volume, %	93.0		95.0		94.0		-		-	
Nitrogen content, wt.%	0.42		0.44		0.35		-		-	
Bottoms, 1000°F+, vol.%	7.0		5.0		6.0		-		-	
Nitrogen Contents and Yields of Products Obtained from Treatment Schemes (2)	Nitrogen Content (wt.%)	Yield (wt.%) (3)	Nitrogen Content (wt.%)	Yield (wt.%) (3)	Nitrogen Content (wt.%)	Yield (wt.%) (3)	Nitrogen Content (wt.%)	Yield (wt.%) (3)	Nitrogen Content (wt.%)	Yield (wt.%) (3)
Scheme A Products (2)										
Raffinate	0.09	76.4	0.08	81.1	0.10	82.3	-	-	-	91.9 (3,6)
Extract	2.09	19.5	2.19	16.6	2.53	11.9	-	-	-	3.4
Loss (4)	-	4.1	-	2.3	-	5.7	-	-	-	4.7
% of Coal as extract (8)	-	65	-	57	-	37	-	-	-	-
Scheme B Products (2)										
Raffinate	0.08	64.8	-	-	-	73.3	-	-	-	-
Extract	1.50	32.2	-	-		25.6	-	-	-	-
Loss (4)	-	3.0	-	-	-	1.1	-	-	-	-
% of Coal as extract (8)	-	107	-	-	-	82	-	-	-	-
Scheme C Products (2)										
Raffinate	0.49	85.0 (5)	-	-	-	96.6 (7)	-	84.6	-	-
nC_6 insoluble	1.36	15.0	-	-	-	3.4	-	15.4	-	-
Loss (4)	-	0.0	-	-	-	0.0	-	0.0	-	-
% of Coal as extract (8)	-	44	-	-	-	3	-	-	-	-

TABLE V (Continued)

SUMMARY OF DATA OBTAINED FROM HYDROGEN CHLORIDE AND HEXANE TREATMENT OF HYDROLIQUEFACTION PRODUCTS

Reaction No.	755572	755580 (13)	740856	740855
Reaction Conditions, (1)			(16)	(16)
Time, min.	120	120	547	250
Temp., °C	425	425	400	400
Press., psig	2500	2500	2500	2500
Catalyst, wt.% (9)	5	10	10 (14)	10 (15)
Reaction Product Data				
Filtered Liquid Product, wt.% N	0.52	0.68	0.44	0.45
Distilled Products				
Distillate, 1000°F				
Volume, %	93.0	91.0	-	-
Nitrogen content wt. %1	0.45	0.55	-	-
Bottoms, 1000°F+, vol.%	7.0	9.0	-	-

Nitrogen Contents and Yields of Products Obtained from Treatment Schemes (2)	755572 Nitrogen Content (wt.%)	755572 Yield (wt.%) (3)	755580 Nitrogen Content (wt.%)	755580 Yield (wt.%) (3)	740856 Nitrogen Content (wt.%)	740856 Yield (wt. %) (3)	740855 Nitrogen Content (wt.%)	740855 Yield (wt.%) (3)
Scheme A Products (2)								
Raffinate	0.10	76.5 (12)	0.10	75.6	-	-	-	-
Extract (4)	2.30	17.0	2.80	23.7	-	-	-	-
Loss	-	6.5	-	0.7	-	-	-	-
% of coal as extract (8)	-	58	-	81	-	-	-	-
Scheme B Products (2)								
Raffinate	-	-	-	-	0.08	78.9	0.08	- -
nC6 insoluble	-	-	-	-	2.10	14.2	2.30	17.5
Loss (4)	-	-	-	-	-	6.9	-	-
% of Coal as extract (8)	-	-	-	-	-	44	-	52
Scheme C Products (2)								
Raffinate	-	-	-	-	0.40	98.9	0.43	97.8
nC6 insoluble	-	-	-	-	1.10	1.1	1.55	2.2
Loss (4)	-	-	-	-	-	0.0	-	0.0
% of coal as extract (8)	-	-	-	-	-	-	-	-

TABLE V (Continued)

SUMMARY OF DATA OBTAINED FROM HYDROGEN CHLORIDE AND HEXANE TREATMENT OF HYDROLIQUEFACTION PRODUCTS

Reaction No.	755616A(11)
Reaction Conditions(1)	
Time, min.	120
Temp., °C	425
Press., psig	2500
Catalyst, wt.%(9)	10(10)
Reaction Product Data	
Filtered Liquid Product, wt.% N	0.44
Distilled Products	
Distillate, 1000°F	
Volume, %	-
Nitrogen content, wt.%	-
Bottoms, 1000°F+, vol.%	-

Nitrogen Contents and Yields of Products Obtained from Treatment Schemes(2)	Nitrogen Content (wt.%)	Yield (wt. %) (3)
Scheme A Products(2)		
Raffinate	-	-
Extract	-	-
Loss (4)	-	-
% of Coal as extract(8)	-	-
Scheme B Products(2)		
Raffinate	0.08	79.4
Extract	2.04	19.0
Loss (4)	-	1.6
% of Coal as extract(8)	-	58
Scheme C Products(2)		
Raffinate	-	90.2
nC_6 insoluble	1.46	9.8
Loss (4)	-	0.0
% of Coal as extract(8)	-	25

FOOTNOTES FOR TABLE V

1. Reactor charge = 1.2 by weight Illinois No. 6 coal and hydrogenated anthracene oil (which had a hydrogen/carbon atomic ratio of 1.1 to 1.2, unless otherwise noted). Reactor was a 1 liter stirred autoclave.

2. See description of various schemes on Figure 1.

3. Weight % yield of each fraction based on weight of charge to treatment via Scheme A, B, or C.

4. Refers to mechanical loss plus water soluble compounds.

5. When this hexane raffinate was subsequently treated with HCl (thus the treatment was the reverse of Scheme B) the following data were obtained:

Fraction	Nitrogen Content, wt.%	Yield (wt.%)(3)
Raffinate	0.08	67.9
	1.90	29.0
Loss	--	3.1

6. No precipitate formed upon treatment with hexane.

7. Raffinate, when treated with HCl, gave 22.1 wt.% total extract (including C_6 insoluble).

8. Calculated from amount of coal which was in the product before entering the scheme.

9. Catalyst, unless otherwise noted, is Sun 1% CoO, 2% MoO_3 on 10 to 20 mesh bauxite.

10. Catalyst = 1% CoO, 2% MoO_3 on -100 + 270 mesh powdered bauxite.

11. Aliquot of the liquid-solid product was distilled to 400°F; bottoms were filtered at 350°F; then the distillate and filtrate were recombined proportionately, and treated via Schemes B and C, Figure 1.

12. Dilution with hexane before saturation with HCl yielded 1.3 wt.% precipitate which contained 2.80 wt.% nitrogen.

Footnotes for Table V (Continued)

13. Hydrogenated anthracene oil solvent used here had hydrogen/carbon atomic ratio of 0.9.

14. Catalyst here contained 0.5 wt.% Li, 1% CoO, 2% MoO_3 on 10 to 20 mesh bauxite.

15. Catalyst here contained trace Ce, 0.5 wt.% Li, 1% CoO, 2% MoO_3 on 10 to 20 mesh bauxite.

16. Reactor here was a 300 ml stirred autoclave.

APPENDIX A

SUMMARY OF REACTION CONDITIONS AND MATERIAL BALANCES FOR ONE LITER REACTOR

		Reactants, gms.					Reaction Conditions			Products, gms.							
Reaction No. (755:xxx)	Reaction Date (mo/day)	Solvent	Coal(5)	Catalyst	Hydrogen (15)	Total	Time (min.)	Temp. (°C)	Pressure (PSIG)	Gas	Wet Ice Trap	Wash Liq.	Soxhlet Liq.	Filtered Liq.	Solids	Total	Recovery Wt.%
500	1/12	507.4(1)	0.0	50.7(2)	7.8	565.9	305	375	3000	4.0	0	48.2	--	459.2	55.6	567.0	100.2
510	1/19	333.0(3)	167.0	50.0(2)	3.0	553.0	456	400	2500	13.8	0	60.8	3.3	370.2	79.1	527.2	95.3
517	2/10	508.5(1)	0.0	51.0(2)	8.0	567.5	403	375	3000	← (10) →							
522	3/2	524.0(1)	0.0	52.4(2)	8.1	584.5	370	375	3000	← (10) →							
530	3/16	537.0(1)	0.0	53.7(2)	8.2	598.9	383	375	3000	← (10) →							
532	3/18	505.5(1)	0.0	53.7(2)	7.7	566.9	440	375	3000	← (10) →							
535	4/6	360.8(4)	167.0	50.0(6)	2.1	552.1	456	400	2500	7.8	0	33.8	7.0	363.0	94.6	506.2	91.7
538	5/3	522.0(1)	0.0	52.2(2)	8.0	582.2	442	375	2500	← (10) →							
540	5/6	428.0	0.0	52.2(2)	6.5	486.7	385	375	2500	← (10) →							
546	5/17	267.2(7)	133.0	40.0(8)	3.8(9)	444.0	60	425	2500	12.6	1.4	51.0	0.3	302.1	66.4	433.8	97.0
552	5/21	268.6(7)	133.0	40.2(8)	7.3(9)	449.1	120	425	2500	15.8	6.8	35.8	10.5	303.8	65.0	437.7	97.5
559	6/3	507.0(1)	0.0	50.7(2)	15.6(9)	573.3	304	375	3000	← (10) →							
563	6/6	505.0(1)	0.0	50.5(2)	(10)	(10)	379	375	3000	← (10) →							
566	6/9	267.0(1)	133.0	80.0(8)	9.2(9)	489.2	120	425	2500	16.5	0.0	63.3	0.3	268.0	108.6	456.7	93.4
572	7/20	268.0(11)	133.0	20.1(8)	9.0(9)	430.1	120	425	2500	18.6	4.2	41.6	1.1	299.0	42.1	406.6	94.5
580	7/26	272.5(12)	135.0	40.8(8)	8.2	456.5	120	425	2500	17.6	3.8	53.5	1.2	297.4	65.6	439.1	96.2
588 to 596	7/29 to 8/8	441.5(13)	0.0	44.2(2)	(14)	(14)	253	300	2000	← (14) →							
602	8/30	270.8(16)	135.4	40.6(8)	6.1	452.9	120	425	2500	← (14) →							
612(17)	9/7 to 9/23	520.0(1)	0.0	52.0(2)	8.0	580.0	440	375	2500	← (10) →							
616	10/5	268.5(18)	134.3	40.3(19)	8.3(9)	451.4	120	425	2500	← (10) →							
623	10/27	268.3(18)	134.1	40.4(19)	8.6(9)	451.4	120	425	2500	← (10) →							

(1) Non-hydrogenated Anthracene Oil-Sun 740701
(2) Filtrol HPC-5 Co-Mo Catalyst
(3) Hydrogenated Anthracene Oil-Sun 755500
(4) Hydrogenated Anthracene Oil-Sun 755530
(5) Illinois No. 6-Sun 740700
(6) Sun 1% CoO-2% MoO_3 on 8 to 20 mesh Bauxite-740711
(7) Hydrogenated Anthracene Oil-Sun 755538
(8) Sun 1% CoO-2% MoO_3 on 8 to 20 mesh Bauxite-740820
(9) Measured via hydrogen reservoir method
(10) Not measured
(11) Hydrogenated Anthracene Oil-Sun 755559
(12) Hydrogenated Anthracene Oil-Sun 755558 (H/C=0.9)
(13) Charge here was a mixture of dimethylnaphthalenes - Sun Code 616616
(14) Data not available yet
(15) Calculated from pressure drop data
(16) Solvent = cut No. 7 of Product distilled from Reactions 755588 to 755596
(17) This was a series of 4 hydrogenations of anthracene oil to generate solvent
(18) Hydrogenated Anthracene oil - Sun 755612
(19) Sun Code 740710 1% CoO, 2% MoO_3 on bauxite 100 to 270 mesh

INTEROFFICE CORRESPONDENCE DATE: September 22, 1977

SUBJECT: COST ESTIMATE
EBULLATING BED COAL HYDROGENATION UNIT

LOCATION: ARD, Processes - Marcus Hook

FROM: J. J. Nicholas

TO: Mr. A. F. Talbot

Our contract with ERDA, E(49-18)-2306, to study conversion of coal to gasoline calls for the construction of a continuous pilot plant for the coal hydroliquefaction step. The pilot plant charge rate is to be about 3 pounds powdered coal per hour.

To procede with this commitment to ERDA, you requested a cost estimate for constructing an "Ebullating Bed Coal Hydrogenation Unit". Taking the general flow diagram and the other process information you've supplied, I estimated the construction costs for two operating schemes. Case I gives my estimated construction costs for a unit recycling process gas, presumably mostly hydrogen, through the reactor after separation from liquid and gaseous products and removal of hydrogen sulfide. Case II presents the construction costs for this same ebullating bed unit, but using once-through hdyrogen in the reactor and venting the off-gases. You letter to Dr. A. Schneider, 8/30/77, discusses the relative merits of these two operating options. Comparison of these two estimates provides cost data to help you decide which of the two operating schemes better satisfies your needs.

A summary of the estimated construction costs for these two cases is as follows:

	CASE I With Recycle Gas	CASE II With Once-Through Hydrogen
Material	$363,100	$289,150
Labor	101,100	83,350
Estimated Installed Cost	$464,200	$372,500

A breakdown of these costs for various construction tasks of the project is given in Table I. The attached drawing, RD-AA-18570, is a preliminary copy of the Engineering Flow Diagram depicting the Ebullating Bed Coal Hydrogenation Unit using recycle gas through the reactor after separation and clean-up (Case I). By eliminating that portion of the unit inside the cross-marked heavy line on this flow diagram, you are left with the unit using fresh hydrogen once-through the reactor (Case II).

Figure 1 presents our estimated work schedule for the design and construction schedule of this project, assuming funds are provided on November 1, 1977. Otherwise, simply move up the schedule to start on the date the funds become available. Optimistic times for completion are 14 months for

Case I and 12.5 months for Case II, while the more likely completion times are 18 months for Case I and 15.5 months for Case II. The largest single factor controlling the completion date will be the delivery of the high pressure equipment for this unit. Our past experience in this regard would tend to show even the longer "more likely" completion dates are overly optimistic. However, both vendors we have been talking to are eager to get the work and are promising quick service. Hopefully they will keep their promises. Another controlling factor is the number of Shop mechanics assigned to this project during our peak construction period. My optimistic calculations are based on using 5 Shop mechanics continually vs. only 3 on the most likely schedule. No overtime or weekend work was used in determining these completion dates.

Since no decision as to the location of this unit has ever been made, the costs may vary somewhat depending on the available utilities, structure, area, process vent lines, cylinder gas storage, etc.

I believe the data provided are what you requested. If I can be of further service, please call upon me.

J. J. Nicholas
J. J. NICHOLAS

JJN/ahs
attachment

cc: Mr. A. T. Finlayson
Mr. E. J. Hollstein
Mr. E. J. Janoski
Mr. H. R. Moyer
Dr. A. Schneider
Mr. I. Steinmetz
CTF

APPENDIX B

COST ESTIMATE - EBULLATING BED COAL HYDROGENATION UNIT

TABLE I

Division of Cost by Various Construction Tasks

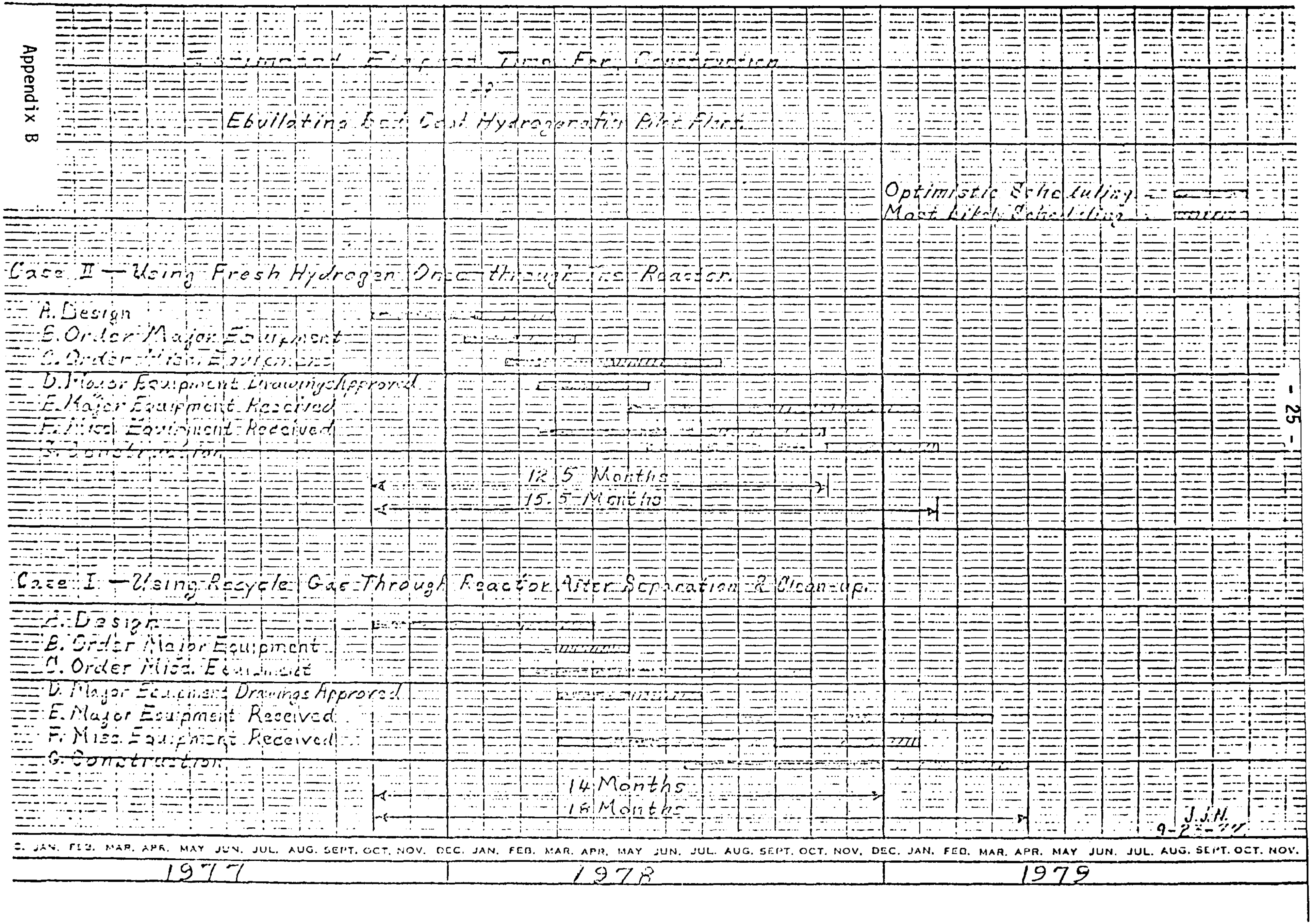
	Case I			Case II		
	Material	Labor	Total	Material	Labor	Total
Major Equipment	$152,850	$ ---	$152,850	$124,750	$ ---	$124,750
Installation & Supports	5,100	9,200	14,300	3,950	7,900	11,850
Process Piping	75,300	37,150	112,450	53,100	29,950	83,050
Service Piping	11,900	12,700	24,600	9,900	10,850	20,750
Electrical	13,800	12,700	26,500	12,000	11,050	23,050
Instrumentation	98,150	21,250	119,400	80,550	16,700	97,250
Insulation	6,000	8,100	14,100	4,900	6,900	11,800
TOTALS	$363,100	$101,100	$464,200	$289,150	$ 83,350	$372,500

	Case I	Case II
Construction Labor	23.4 Man-Months	19.3 Man-Months
Time for Completion*	14-18 Months	12.5-15.5 Months

*After approval of Funds

Appendix B

Fig. 1



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